# E-ISBN: 978-605-68537-6-0 TICMET'19 October 10-12, 2019

**The International Conference** of Materials and Engineering Technology

(Uluslararası Malzeme ve Mühendislik Teknolojileri Konferansı)











حامعة

حمدين خليفة

HAMAD BIN KHALIFA UNIVERSITY

enterprise europe network \*\*\*

Business Support on Your Doorstep

YÜCETEKNİK

Movero Congress ter

Goziontepuniversity

GOZIONTEPITURKEY









# INTERNATIONAL ORGANISATION **FOR MIGRATION (IOM)**



The International Conference of Materials and Engineering Technology

ME

Established in 1951, IOM is the leading inter-governmental organization in the field of migration and works closely with governmental, intergovernmental and non-governmental partners.

With 173 member states, a further 8 states holding observer status and offices in over 100 countries, IOM is dedicated to promoting humane and orderly migration for the benefit of all. It does so by providing services and advice to governments and migrants.

IOM works to help ensure the orderly and humane management of migration, to promote international cooperation on migration issues, to assist in the search for practical solutions to migration problems and to provide humanitarian assistance to migrants in need, including refugees and internally displaced people.

The IOM Constitution recognizes the link between migration and economic, social and cultural development, as well as to the right of freedom of movement. IOM works in the four broad areas of migration management:

- Migration and development
- Facilitating migration
- Regulating migration
- Forced migration.

IOM activities that cut across these areas include the promotion of international migration law, policy debate and guidance, protection of migrants' rights, migration health and the gender dimension of migration.

# **IOM IN TURKEY**

IOM Turkey, the UN Migration Agency first opened its offices in Turkey in 1991 following the aftermath of the Gulf War. IOM's partnership with the Republic of Turkey was formalized in November 2004 when Turkey was

granted member status to IOM. The partnership between IOM and Turkey continued since then, including support drafting the Law of Foreigners and International Protection, as well as establishing the Ministry of Interior's Directorate General for Migration Management in 2013. Now in its 27th year of operations in Turkey, the mission addresses the full scope of migration issues, supporting and developing government capacity to manage migration.

Initially focusing its attention on resettlement for Iraqi refugees in the 1990s, IOM Turkey later expanded in response to the devastating earthquake of 2011 in Van to include emergency response programmes. The mission's emergency response programmes have continued to grow rapidly since 2012 and 2015 with the start of the Syrian crisis and Mediterranean crisis.

Alongside IOM's role in addressing the needs of migrants during crises, the mission works in close collaboration with the Government of Turkey to address the longer-term impact of migration, including migrant assistance programmes, labour integration and migration management, immigration and border management and research and data collection on migrant movement.

Our resettlement programme has also grown to play an important role in the process of resettling refugees abroad through cultural orientation, medical checks and flight arrangements to third countries.

With over 25 years of operational experience in Turkey, the Mission is now one of the largest globally and has more than 900 staff working across the country, with sub-offices in Istanbul, Gaziantep, Hatay, Sanliurfa and Izmir and head office in Ankara.

# **CONFERENCE OBJECTIVE**

CME

The International Conference of Materials and Engineering Technology

The International Conference of Materials and Engineering Technology (TICMET'19) will be organized by Gaziantep University and together with other collaborating universities in Gaziantep, TURKEY on 10-12 October, 2019.

Gaziantep, a city Southeast Anatolia is the 6th largest city in Turkey. The city has a rich cultural heritage and it has been incorporated into the creative cities network by UNESCO in the field of gastronomy.

The main objective of TICMET'19 is to present the latest research and results of scientists related to Computer Sciences, Electrical&Electronics, Energy Technologies, Material Sciences, Manufacturing Technologies, Mechatronics and Biomedical Technologies. This conference provides opportunities for the different areas delegates to exchange new ideas and application experiences face to face, to establish business or research relations and to find global partners for future collaboration. We hope that the conferences results constituted significant contribution to the knowledge in these up to date scientific field. The organizing committee of conference is pleased to invite prospective authors to submit their original manuscripts to TICMET'19.

All abstracts will be reviewed and evaluated by the referees in the relevant field on the basis of technical and / or research content / depth, accuracy, relevance to the conference, contributions and readability.

Selected papers presented in the conference will be published one of the following journals:

- The International Jornal of Materials and Engineering Technologies (TIJMET) (Dergipark)
- El-Cezeri Journal of Science and Engineering (TR Dizin)

# **Honorary Chair**

Prof. Dr. Ali Gur, Gaziantep University - TR Prof. Dr. Edibe Sozen, Hasan Kalyoncu University – TR

## Chair

Assoc. Prof. Dr. Necip Fazil Yilmaz, Gaziantep University – TR

# Organizing Committee

Assoc. Prof. Dr. Necip Fazil Yilmaz, Gaziantep University - TR Assoc. Prof. Dr. Halil Ibrahim Kurt, Gaziantep University - TR Prof. Dr. Adem Kurt, Gazi University - TR Prof. Dr. Ali Riza Yildiz, Uludag University - TR Prof. Dr. Metin Bedir, Gaziantep University - TR Prof. Dr. Murat Oduncuoglu, Yildiz Technical University - TR Prof. Dr. Yusuf Arayici, Hasan Kalyoncu University - TR Lect. Aykut Bilici, Gaziantep University - TR



# UWE

# **Executive Committee**

Assoc. Prof. Dr. Emrah Ozahi, Gaziantep University - TR Assoc. Prof. Dr. Zuleyha Degirmenci, Gaziantep University - TR Assist. Prof. Dr. Abdulcabbar Yavuz, Gaziantep University - TR Assist. Prof. Dr. Esin Sarioglu, Gaziantep University - TR Assist. Prof. Dr. Hakan Candar, Gaziantep University - TR Dr. Erhan Durur, Gaziantep University - TR Lect. Aykut Bilici, Gaziantep University - TR Lect. Musa Yilmaz, Gaziantep University - TR Research Assist Mahmut Furkan Kalkan, Gaziantep University - TR Mr. Mucahit Dogan, Gaziantep University – TR

CMET'

The International Conference of Materials and Engineering Technology

# Scientific Committee (National Committee)

Prof. Dr. Abdulkadir Eksi, Cukurova University - TR Prof. Dr. Ismail Saritas, Selcuk University - TR Prof. Dr. Mehmet Cengiz Kayacan, Süleyman Demirel University - TR Prof. Dr. Mustafa Bakkal, Istanbul Technical University - TR Prof. Dr. Mustafa Cigdem, Yildiz Technical University - TR Prof. Dr. Oguzhan Yilmaz, Gazi University - TR Prof. Dr. Omer Eyercioglu, Gaziantep University - TR Prof. Dr. Rahmi Unal, Gazi University - TR Prof. Dr. Serdar Salman, National Defense University - TR Prof. Dr. Suleyman Can Kurnaz, Sakarya University - TR Prof. Dr. Ugur Cem Hasar, Gaziantep University - TR Prof. Dr. Yahya Bozkurt, Marmara University - TR Assist. Prof. Dr. Ali Yasar, Selcuk University - TR Assist. Prof. Dr. Aziz Baris Basyigit, Kirikkale University - TR Assist. Prof. Dr. Ilker Ali Ozkan, Selcuk University - TR Assist. Prof. Dr. Murat Koklu, Selcuk University - TR Dr. Engin Ergul, Dokuz Eylul University – TR



UWE

CMET'

The International Conference of Materials and Engineering Technology

Prof. Dr. Andrey Tsarkov, Moscow State Technical University - RU Prof. Dr. Bekir Sami Yilbas, King Fahd University of Petroleum and Minerals - SA Prof. Dr. Fabienne Delaunois, University Of Mons - BE Prof. Dr. Hazman Seli, University Of Sains Malaysia - MY Prof. Dr. Heiba Zein, Ain Shams University - EG Prof. Dr. Marwan K Khraisheh, Hamad Bin Khalifa University - QA Prof. Dr. Mohamed Elsayed Yahia, International University of Sarajevo - BA Prof. Dr. Muammer Koç, Hamad Bin Khalifa University - QA Prof. Dr. Ramazan Asmatulu, Wichita State University - US Prof. Dr. Senay Simsek, North Dakota State University - US Prof. Dr. Tai Cheng Chen, Institute Of Nuclear Energy Research Iner - TW Prof. Dr. Vincent Ji, University Of Paris Sud - FR Prof. Dr. Yousef Haik, Hamad Bin Khalifa University Hbku - QA Prof. Dr. Yusuf Ozturk, San Diego State University - US Prof. Dr. Zakaria Boumerzoug, University Of Biskra - DZ Assoc. Prof. Dr. Ahmad Azmin Mohamad, University Of Sains Malaysia - MY Assoc. Prof. Dr. Mehmet Emin Aydin, UWE Bristol - GB Assoc. Prof. Dr. Mikhail E Semenov, Tomsk Polytechnic University - RU Assist. Prof. Dr. Faten Adel Ismael Chaqmaqchee, Koya University - IQ Assist. Prof. Dr. Nurettin Sezer, Hamad Bin Khalifa University Hbku - QA Assist. Prof. Dr. Sami G Al Ghamdi, Hamad Bin Khalifa University Hbku - QA Assist. Prof. Dr. Shoukat Alim Khan, Hamad Bin Khalifa University Hbku - QA Assist. Prof. Dr. Yusuf Bicer, Hamad Bin Khalifa University Hbku - QA Dr. Abdessalem Bouferrouk, University Of The West Of England - GB Dr. Fahmi Fariq Muhammad, Koya University - IQ Dr. Mazhar Malik, University Of The West Of England - GB Dr. Mirza Nadeem Baig, Pakistan Welding Institute - PK Dr. Mohammad Ghaffar Faraj, Koya University - IQ Dr. Peter Kay, University Of The West Of England - GB Dr. Sabir Ghauri, University Of The West Of England - GB Dr. Shahroz Sakhawat, Pakistan Welding Institute – PK

# **INVITED SPEAKERS**

#### MUAMMER KOC

دمدين خليفة

HAMAD BIN KHALIFA

UNIVERSITY

Prior to his appointment as a founding professor of sustainability at HBKU in 2014, Professor Koc held professor, director, chair and dean positions at different universities in Turkey and the USA between 2000-2014. He has a PhD degree in Industrial and Systems Engineering from the Ohio State University (1999) and an Executive MBA degree from the University of Sheffield, UK (2014). He has published 130+ publications in various international journals and conferences; edited three books; organized, chaired, and co-chaired various international conferences, workshops and seminars on design, manufacturing and product development. In addition to his academic and educational activities, he provides consulting services to industry, government and educational institutes for strategic transformation, business optimization, organizational efficiency, lean operations, restructuring and reengineering initiatives. He has taught courses across a range of subjects, including product/process/business innovation and development; medical design and production; energy and efficiency; computer-aided engineering, design and manufacturing; modern manufacturing technologies; manufacturing system design; material forming plasticity; and the mechanical behavior of materials.

CMET'

The International Conference of Materials and Engineering Technology

#### MARWAN KHRAISHEH

Prior to joining the Masdar Institute faculty, Dr. Khraisheh was the Secat - J. Morris Professor and the Director of Undergraduate Studies in the Mechanical Engineering Department at the University of Kentucky. His research interests include materials processing and sustainable manufacturing. He currently focuses on developing innovative concepts and techniques for processing and fabrication of advanced materials including lightweight alloys. Dr. Khraisheh is a recipient of a number of significant awards including the prestigious US National Science Foundation CAREER Award, the 2004 Society of Manufacturing Engineers (SME) Eugene Merchant Outstanding Young Manufacturing Engineer Award, the 2003 North American Manufacturing Research Institute (NAMRI) Outstanding Paper Award and the 2005 Henry Mason Lutes Award for Excellence in Engineering Education. He serves on the Editorial Board of Journal of Materials Processing Technology (Elsevier) and is a member of a number of international technical committees from ASME, ASM and TMS. Dr. Khraisheh has edited a proceeding book for ASME and has more than 110 publications including refereed journal papers, refereed conference papers and presentations.





Prof. Dr. Muammer KOC

Professor and Coordinator

Hamad Bin Khalifa University (HBKU)

UWE

Bristol



#### UWE of the دمدين خليفة Bristol HAMAD BIN KHALIFA UNIVERSITY

# **INVITED SPEAKERS**

# Invited Speaker Prof. Dr. Zakaria BOUMERZOUG University Mohamed Khider

of Biskra

# ZAKARIA BOUMERZOUG

Zakaria Boumerzoug is a professor of metallic materials at Biskra University. He joined Biskra University in 1992. He obtained his PhD from Constantine University in 1998. He published fifty articles and he participated to more than thirty international conferences as speaker and invited speaker. He was as a member and also as a guest editor in some international conferences. He has supervised 17 PhD students. He has reviewed many articles of some scientific journals. He is a chief of an international project between Biskra University and UMONS University in Belgium. He has a short international teaching experience at Cadiz University, Spain, May 2017( ERASMUS Program). He has organised more than 10 scientific meeting in Algeria.

CMET'

The International Conference of Materials and Engineering Technology



# MEHMET AYDIN

Mehmet AYDIN joined CSCT department at the end of January 2015 as Senior Lecturer in Computer Science. Prior to this post, Mehmet AYDIN have worked in academic and research positions for various universities including University of Bedfordshire, London South Bank University and University of Aberdeen. Mehmet AYDIN editorial board member of a number of international peer-reviewed journals, and have been serving as committee member of various international conferences. Mehmet AYDIN also member of EPSRC Review College and fellow of Higher Education Academy. Mehmet AYDIN's research interests include parallel and distributed metaheuristics, wired/wireless network planning and optimization, combinatorial optimization, evolutionary computation and intelligent agents and multi agent systems.



Bristol HAMAD BIN KHALIFA

# **INVITED SPEAKERS**

# **YOUSEF HAIK**

íц

UNIVERSITY

Yousef Haik is an internationally recognized scholar in the fields of engineering and nanotechnology. He has made notable contributions to the creation of novel nanomaterial that have seen applications in magnetic hyperthermia, onsite diagnostics, antimicrobials and solar cells. He is an inventor of numerous patents, many of which have been licensed. His publication record includes over 250 peer-reviewed articles, textbooks and conference proceedings. He is honored with a number of prestigious awards, including among many, the HH Sheikh Khalifa Award for Distinguished Research Professor, Arab Thoughts Foundation and the FUIW-FUMI Research Award. He is a fellow of the American Society of Mechanical Engineers (ASME) and a member of more than a dozen international societies. He serves as the Editor in Chief and on the editorial board for more than a dozen international journals. His academic career has included appointments in engineering (Mechanical, Biomedical and Nanoengineering) and science (Medicinal Chemistry, Physics and Nanoscience) programs. He is an ABET evaluator. His administrative appointments include Associate Vice President, Dean, Director for Research Centers, Department Chair and Graduate Programs Director. He has led the creation of a number of degree programs.

CMET

The International Conference of Materials and Engineering Technology







Aslankaya Döküm yakın zamanda, kendi alanında uzman bir ekip ile Gaziantep 5. Organize Sanayi Bölgesi'nde kurulmuştur. Bölgede ürün kalitesi ve üretim teknolojisi olarak öncü bir marka olma konusunda emin adımlarla ilerleyen firmamız, kaliteyi kendine ilke edinmiştir. 5000 m2 kapalı alanda üretim faaliyetlerimizi gerçekleştiriyoruz. Şirket olarak teknolojik gelişmeleri yakından takip ederek Güneydoğu Anadolu bölgesinin en gelişmiş dökümhane laboratuvarı ve son teknoloji test ve analiz cihazlarıyla döküm sektöründe hizmet vermekteyiz.

Bulunduğu bölgede ilkleri gerçekleştirme becerisine sahip yetenekli insan gücü ve bilgi birikimine sahip olan Aslankaya Döküm, özel dökümhanelere kıyasla ürettiği parça ve yedeklerle bu bilgi birikimi ve becerisini ortaya koymuş ve kendini ispatlamıştır.

Firmamız çevreye duyarlı, kaliteli ve rekabetçi hizmet anlayışıyla üretimin her aşamasında müşteri memnuniyetini ilke edinmiştir. İnşaat ve Maden Endüstrisi, Çimento Endüstrisi, Makine Endüstrisi, Enerji Sektörü başta olmak üzere birçok sektöre ve alana en üst kalite ile üretim yapmaktayız.





Anadolu Mühendislik 1988 yılında demir & çelik fabrikalarına refrakter malzeme üretimi için Gaziantep'te kurulmuştur. Kısa sürede geniş bir müşteri portföyü edinen firmamız, ilerleyen yıllar içerisinde ithalat ve ihracata ağırlık vermiş, Avrupa, Asya, Afrika ve Ortadoğu ülkeleri gibi çok geniş bir coğrafya ile ticari ilişkiler kurmuş çok çeşitli refrakter ürünleri ithal ve ihraç etmektedir.

# Firmamızın üretmiş olduğu ürünler:

- ✓ Pota sürgü plakaları ve Reftakterleri
- 🗸 Tandiş Zirkonya Nozulları
- ✓ Şekilli Refrakterler
- ✓ Çelik Könvertörler için Curuf kesme dartı
- 🖌 Pota nozul kumu
- ✓ Refrakter harçları (Alumina, Manyezit)
- ✓ DC-AEF için iletken sıcak tamir harcı
- ✓ Pota sürgü sistemleri, Tandiş mekanizmaları ve yedekleri
- ✓ Tandiş akış kontrol sistemi
- ✓ Sürgü Plakası ve Nozul sacları





Gazi Haksan 1994 yılında Kadir FIRAT tarafından CNC dik işlem makine,CNC kayar otomat,CNC c eksen sürücülü makineleri ile yedek parça imalatına başlamıştır.

2000 yılında metal enjeksiyon, plastik enjeksiyon kendi kalıplarını üreterek, asansör ve çelik para kasaları yedek parça imalatı ile Gaziantep sanayisine katkıda bulunarak Türkiye'nin bir çok yerinde çeşitli sektörler ile çalışarak hizmet vermektedir. 1000 metrekare arazi üzerindeki fabrikada Türkiye'nin birçok bölgesindeki şehirlere uzanan hizmet ağı ile ülke ekonomisine katkıda bulunmaya devam etmiştir.



**YÜCE TEKNİK**; Firmamız 2007 Yılında kurulmuş, kuruluşundan Bu Yana Edindiği Ticari Tecrübesini Yenilikçi Yapısı Ve Zengin Ürün Yelpazesiyle Birleştirerek, Metal Sektöründe Faaliyet Gösteren Firmalara Tedarikçi Konumuyla Perakende ve Toptan satış olarak Hizmet Vermektedir.

Teknik hırdavat ve kalıp Elemanları ürünleri üzerine kurulan firmamız 2017 yılından itibaren kendi markası (HERO, WINDBREAKER,YTH) adı altında ithalat ve ihracat yapmaya başlamıştır.

Yüce Teknik, metal sektöründe müşteri odaklılık prensibi üzerinde çalışan, sektörünü iyi tanıyan, paydaşlarının beklentilerini bilen ve yeni beklentiler tanımlayıp, bunların en üst düzeyde tatminini hedefleyen, yaratıcı insan gücüne sahip ve bu doğrultuda tedarik sürecinde en iyi hizmeti veren, güvenilir, Teknik Hırdavat ve Kalıp elemanlar değer zinciri alanlarında verimli ve kârlı hizmet sunmayı hedefleyen bir Teknik Hırdavat şirketidir.





**Günmak Makine**; 1975 yılında ilkokul 3. Sınıftan terk Sıddık KAYABAŞ tarafından 75 m2 bir alanda temelleri atılan, zaman içerisinde kendini bilgi ve tecrübe olarak geliştirmenin yanında, alan ve ekipman olarak ta büyüme sağlayan şu anda 4. Organize sanayi bölgesinde 25.000 m2 bir kurulu alanda, makine ve helezon üretimi yapmaktadır. Ürün yelpazesi oldukça geniş olan GÜNMAK MAKİNE, başlıca üretimi; Anahtar teslimi Bulgur tesisleri, kırmızı mercimek tesisleri, bakliyat çeşitlerinin(arpa, yulaf, bakla, mısır, kinoa, sorgum, bezelye, nohut, pirinç vb.) her türlü makinaları(temizleme, kabuk soyma, taş ayırma, yıkama, eleme ve sınıflandırma vb.) mısır çerezi üretim makinaları, Antep fıstığı kurutma ve taşıma sistemleridir. Ayrıca plastik geri dönüşüm tesisleri, beton santralleri , asfalt plantleri, yağ fabrikaları, bakliyat üretim tesisleri, maden ocakları gibi bir çok iş kolu için ise değişik çap ve modellerde helezon üretimi yapmaktadır. Bugün itibarı ile 2 ülkeye ihracat geçekleştirmekte, ihracat ağını genişletmek için yoğun dış ticaret faaliyetlerinde bulunmaktadır.

Sürekli ve köklü değişim fırtınaları her alanda tüm sektörleri vuruyor; işletmeler, bireyler, meslekler ve kariyerler savruluyor. Başlarını kuma gömüp fırtınaların dinmesini beklemek, eskiden öğrenilenler, geleneksel yaklaşımlar yeni durumla başa çıkmaya yetmiyor. Öğrenilmiş savunma mekanizmaları, durumu kötüleştirmekten başka bir işe yaramıyor. Firmalar yenilik için ARGE merkezleri kurarak değişim rüzgarına karşı en önemli adımları atıyorlar.

Günmak değişimin gerekliliğini, yenilikçiliğin olmadığı firmaların yarınlara yelken açamayacağının bilincindedir. Bu kapsamda sürekli kendi ile yarışan yeni ürünler geliştiren ve üretenlerin başında gelmektedir. 1990 lı yıllardan beri Üniversite Sanayi işbirliği ile AR-GE bilincini başlatan, personelini bu bilinç ile yetiştiren firma, Tübitak'tan 4 projede 7 Adet AR-GE yaparak gıda dalında en fazla proje yapan firma olmuştur. 2017 yılında yapmış olduğu Bakliyat, hububat pişirme ve kurutma ve Antep fıstığı kurutma ünitesi ile, üretim kapasitesinde ve enerji tasarrufunda çok büyük ilerleme kaydederek patent başvurusu yapmış ve Türk Patent Kurumundan belgeleri almaya hak kazanmıştır. Yöresel bir ürün olan hububat ve Antep fıstığının hijyenik şartlarda el değmeden üretilebilmesi maksadı ile geliştirilen bu üniteler Avrupa gıda normlarına ve CE standartlarına uygun yüksek kapasiteli, istenilen nem oranında kurutma imkanı sağlamaktadır. Düşün enerji tüketimi, uygun kurutma oranları ile müşterilerine , geniş rekabet ortamında büyük avantaj sağlamıştır. GÜNMAK bundan sonra da aynı düşünceler ile uluslararası firma olma yolunda, Güzel ülkemizin istihdamına ihracatına ve gelişimine katkı sağlamak üzere tüm enerjisi ile yoluna devam etmektedir.





# CONTENTS

AL2024 KOMPOZİTLERİN AŞINMA KARAKTERİSTİĞİ Engin ergül, halil ibrahim kurt, can çivi, gökhan eyici	2
AA 8006 ALÜMİNYUM ALAŞIMI MUTFAK FOLYO ÜRETİMİNİN HADDELEM SÜRECİNDE OLUŞAN HATALAR ve ANALİZİ HÜSEYİN UZUN, ÖZGE AYAZ	<b>1E</b> 6
MEMRISTOR BASED LOGIC OPERATIONS IN CROSSBAR SYSTEMS UFUK ASIL, ABDULLAH SECGIN	15
CAM ELYAF TAKVİYELİ PA66 KOMPOZİTTEN BAĞLANTI DESTEK PARÇASININ ENJEKSİYONLA ÜRETİMİNE KALIP SICAKLIĞININ ETKİSİ HÜSEYİN UZUN, MÜGE FİLİZ	25
<b>POLİETİLEN FİLM YÜZEYİNİN, REAKTİF GAZ ORTAMINDA FONKSİYONELLEŞTİRİLMESİ</b> celal söylemez, ilker türkmen	32
DETERMINATION OF WEIBULL COEFFICIENTS FOR HATAY REGION BY USING THE MOVING LEAST SQUARES APPROACH YUSUF ALPER KAPLAN, AYŞE GÜL KAPLAN	37
CRITICAL SIZE OF A SLAB REACTOR WITH NEUTRON TRASNPORT THEO OF THE TRIPLET ANISOTROPIC SCATTERING HALIDE KÖKLÜ, OKAN ÖZER	<b>PRY</b> 47
<b>GRAFİT KATKILI POLİPROPİLEN KOMPOZİTLERİN MEKANİK VE TRİBOLOJİK ÖZELLİKLERİNİN İNCELENMESİ</b> salih hakan yetgin, murat çolak	57
SICAKLIK DEĞİŞİMİNİN POLİÜRETAN ÇARPIŞMA KUTULARININ DARBE PERFORMANSINA ETKİSİNİN DENEYSEL OLARAK İNCELENMESİ Murat reis	66





EXTRACTION OF INTERFACIAL PROPERTIES FOR DIFFERENT TYPES OF FRC WITH AIDS OF MODERN TECHNIQUES AMJAD KHABAZ	72
FABRICATION, CHARACTERIZATION, AND PERFORMANCE OF MIXED- MATRIX, WATER-TREATMENT MEMBRANES MODIFIED WITH 2-D NANOMATERIALS AHMED ABDALA, OMNYA ABDALLA, ZAFAR KHAN GHOURI	82
MATERIAL CHARACTERISTICS OF POROUS COPPER FOAMS FOR HEAT TRANSFER PERFORMANCE ASSESSMENT OZKAN KIREC, MELDA OZDINC CARPINLIOGLU	90
SURFACE FINISHING OF AEROSPACE MATERIALS osman soydan, kürşad göv, ömer eyercioğlu	102
MICRO HOLE DRILLING OF AEROSPACE MATERIALS AND REGRESSION ANALYSIS AYKUT AKBULUT, KURSAD GOV	112
<b>USING NANO FIBERS FOR ACOUSTIC INSULATION IN AEROSPACE</b> <b>PARTS</b> BİRKAN BAĞLAMA, KÜRŞAD GÖV, EYÜP YETER	125
CONTROLLING THE STRUCTURE AND PERFORMANCE OF GRAPHENE OXIDE MEMBRANES ISHA ABBASI, NAFIA TANSEEM, ZAFAR GHOURI, KHALED ELSAYED, AND AHMED ABDALA	133
KINEMATIC ANALYSIS OF 3 DOF FLIGHT SIMULATOR EDIP OZTURK, KURSAD GOV	140
ELECTROSPINNING OF PAN/ZEOLITE COMPOSITE NANOFIBERS Funda cengiz çallıoğlu, hülya kesici güler	152
DETERMINATION OF THE CRITICAL POINT ON SNIPER BARREL IN TERMS OF THE STRESS AND DEFORMATION CONCEPTS MEHMET HANIFI DOĞRU	160





INVESTIGATION OF THE BULLET IMPACT ENERGY PERFORMANCE ACCORDING TO VARIABLE TIP GEOMETRY	
MURAT SUBAŞI, MEHMET HANİFİ DOĞRU, EYÜP YETER, NECİP FAZIL YILMAZ	168
INVESTIGATION OF FRICTION PROPERTIES BETWEEN PLASMA SPRAY CERAMIC COATED DISC-PAD INTERACTIONS	179
VOČUNI UK FONKSIVONFI. METODUNUN NADIR TOPRAK METAI	170
HEKZABORÜRLERDE KULLANIMI; HESAPLAMALI MALZEME BİLİMİ	
ÇALIŞIVIASI CENGİZ BOZADA, MİKAİL ASLAN	184
PRINCIPLES AND FINITE ELEMENT SIMULATION OF MULTI-POINT FORMING TECHNOLOGY FOR SHEET METAL	
MAHMUT TANDOGAN, OMER EYERCIOGLU, MUSTAFA DULGER	191
ABRASIVE FLOW MACHINING OF FIBER EXTRUSION SPINNERET HOLES ADEM AKSOY, OMER EYERCIOGLU, KURSAD GOV	200
NADIR TORAK METAL HEKZABORURLERIN KATALIZOR OLARAK	
KULLANILMASI CENGİZ BOZADA, MİKAİL ASLAN	208
THE EFFECTS OF PUNCH SPEED ON THE FORGING LOAD OF HOT	
PRECISION BEVEL GEAR FORGING MEHMET ALADAG, OMER EYERCIOGLU, GULAGA TAS	214
SPIRAL KAYNAKLI ÇELIK BORULARIN KOROZYONA KARŞI FBE KAPLAMALARIN TEST VE KARAKTERİZASYONU	
MAHMUT GEL, GOKHAN ÇIL, EKREM ALTUNCU	225
COMPARISON OF OPEN SOURCE AND HARDWARE FLIGHT CONTROLLER ONUR ACAR, İBRAHİM GÖV, M. HANİFİ DOĞRU	<b>RS</b> 234
INVESTIGATION OF THE EFFECT OF CARBONATION CAKE ON	
RHEOLOGICAL PROPERTIES OF BITUMINOUS BINDERS Ahmet münir özdemir, erkut yalçın, hasan arslanoğlu, mehmet yılmaz,	
BAHA VURAL KÖK	247





# **GREY WOLF OPTIMIZATION BASED PTS SCHEME FOR PAPR REDUCTION IN OFDM SYSTEMS** YÜKSEL TOKUR BOZKURT 256 THE EFFECTS OF FLUX TYPE ON MECHANICAL AND MICROSTRUCTURAL **PROPERTIES OF S235 STRUCTURAL STEEL BY SUBMERGED ARC WELDING** AZİZ BARIŞ BAŞYİĞİT, BÜŞRA SOLAK 263 **EFFECT OF GLASS FIBER HYBRIDIZATION ON LOW VELOCITY IMPACT** BEHAVIORS OF BASALT FIBER REINFORCED COMPOSITE LAMINATES ÖZKAN ÖZBEK, ÖMER YAVUZ BOZKURT, AHMET ERKLİĞ 270 MECHANICAL PROPERTIES OF GRAPHENE NANO-PLATES **REINFORCED LDPE COMPOSITES** AHMET ERKLİĞ, TUĞÇE GÜL POLAT 277 AL CU MG MGO MWCNT KOMPOZİTLERİN FARKLI YÜKLER ALTINDA ASINMA ÖZELLİKLERİNİN İNCELENMESİ HALİL İBRAHİM KURT, ENGİN ERGÜL, MURAT ODUNCUĞLU, CAN ÇİVİ 288 MCRALY İÇERİĞİNE SAHİP TERMAL SPREY KAPLAMALARIN AŞINMA DAYANIMI AMAÇLI OLARAK GAZ TÜRBİN MOTORLARINDA KULLANILMASI MEHMET KILIC, DERVİS ÖZKAN, ABDULLAH CAHİT KARAOĞLANLI 292 AL 7075 PVC ISITICI YUZEYİNE AKIMSIZ NI-B-PTFE İLE NANOKOMPOZİT **KAPLAMA UYGULAMASI** MUSTAFA BAKKAL, ALI TANER KUZU, MAHMUT YILMAZ, ERAY ÇELİK, FİKRET ERTÜRK, BERKAY YUCETURK, İREM DURULMUŞ 299 PVA – TİO2 NANOKOMPOZİTİNİN ÜRETİMİ, KARAKTERİZASYONU VE ANTIBAKTERIYEL ÖZELLIKLERININ INCELEMESI DERYA TEKİN, DERYA BİRHAN, TANER TEKİN 306 r-GO /TiO2 NANOKOMPOZİTİNİN ÜRETİMİ, KARAKTERİZASYONU VE SERTLİK ÖZELLİKLERİNİN İNCELENMESİ DERYA TEKİN, DERYA BİRHAN, TANER TEKİN 311 INVESTIGATION OF FRICTION AND WEAR PERFORMANCE OF PINE CONE **POWDER ADDITIVE BRAKE PADS**

ADEM KURT, H.İBRAHİM TOFAN



DAVUT LACİN, A.Z. AROGUZ

# VÜCUT SICAKLIĞI UYARISI VEREBİLEN AKILLI SPOR TEKSTİL ÜRÜNÜ **TASARIMI**

HALİL İBRAHİM ÇELİK, DİLEK ALICI, ESİLE GÜLSEVGİ, ZEYNEP SENGÜL, ELİF GÜLTEKİN

Were were were were were were were were	Technolog
A REVIEW STUDY ON SMART TEXTILE INNOVATIONS AND DEVELOPM HALIL İBRAHIM ÇELİK, ABDULKADİR AĞA, ELİF GÜLTEKİN, HATİCE KÜBRA KAYNAK	<b>ENTS</b> 410
LOW VELOCITY IMPACT BEHAVIORS OF THERMOPLASTICS COMPOSI WITH DELAMINATONS	TES
OĞUZHAN DURDU, OKAN ÖZDEMİR	420
ÇOK YÖNLÜ TEKERLERE SAHİP BİR ÇATALLI YÜKLEYİCİNİN HAREKE KONTROLÜNÜN DENEYSEL İNCELENMESİ	T
MAHMUT CIMEN, METE KALYONCU	427
INVESTIGATION OF YARN PROPERTIES PRODUCED FROM 100% RECYC POLYESTER (r-PET) BY DIFFERENT SPINNING SYSTEMS	CLED
ESİN SARIOĞLU, DENİZ VURUŞKAN, OSMAN YAYLA, EYÜP ALİ SATIL, EBRU ÇELİKTEN	450
NADİR TOPRAK METAL HEKZABORÜRLERLE KOMPOZİT OLUŞUMU cengiz bozada, mikail aslan, halil ibrahim kurt, zihni öztürk	457
DESIGN OF A NEW REWINDING SYSTEM FOR CORELESS STRETCH FILM PRODUCTION HATICE NIDA CIVAN HADI SAMET MUMCULOSMAN YAYLA	<b>M</b> 470
	770
BİR AĞIR VASITA HAVA KOMPRESÖRÜNÜN TERMODİNAMİK DAVRANIŞININ DENEYSEL İNCELENMESİ	
MURAT ŞUKRU AYDINER, ERDEM UNUVAR, METE KALYONCU	475
ELECTROCHEMICAL CORROSION BEHAVIOR OF HEAT-TREATED 2205 DUPLEX STAINLESS STEEL IN CHLORIDE SOLUTION HUSEYIN ZENGIN	487
	107
EFFECT OF DIFFERENT INTERMETALLIC COMPOUNDS ON CORROSION RESISTANCE OF Mg-AI MAGNESIUM ALLOYS	N 402
	470
<b>DESIGN AND ANALYSIS OF MANHOLE COVER FOR TRAILERS</b> ISMAIL BOGREKCI, PINAR DEMIRCIOGLU, DECAN ALPARGUN, EMRE CEYLAN, KADİR BOZKURT	500
ΑΙ ΜαΟ ΟΟΜΒΟΖΙΤΙ ΕΒΙΝ ΠΒΕΤΙΜΙ ΥΕ ΓΑ ΒΕΙΖΤΕΒΙΘΤΙΟΙ	
AI- IVIGO COIVIFOLTI LEKIIN UKETIIVII VE KAKEKTEKISTIGI MIVAILASLAN ADDILLAZIZ VAVA HALILIDDAHIM VIDT NECIDEAZIL VILMAZ	506





AL-MG-SIC KOMPOZITLERIN GÖRÜNÜR YOĞUNLUKLARININ TAGUCHI	
ANALIZI ABDULAZIZ KAYA, MIKAIL ASLAN, NECIP FAZIL YILMAZ, HALIL IBRAHIM KURT	513
TÜMÜYLE ELEKTRİKLİ BİNEK TİPLİ BİR ARAÇTA YUVARLANMA DİREN DEĞİŞİMİNİN İVMELENME PERFORMANSI VE TRANSMİSYON KAVIPLARINA ETKİSİ ÜZERİNE BİR CALISMA	Cİ
M. AKİF KUNT	521
AL-MG-AI2O3 KOMPOZITLERIN YAPAY SİNİR AĞLARI YAKLAŞIMI POROZİTE İÇERİĞİNİN ARAŞTIRILMASI	İLE
HALIL IBRAHIM KURT, ENGIN ERGUL, NECIP FAZIL YILMAZ, MURAT ODUNCUOGLU	532
BURSTING STRENGTH ANALYSIS OF KNITTED FABRICS FROM MULTIFILAMENT/MICROFILAMENT CORE-SPUN YARNS ESIN SARIOĞLU, GÜLBIN FIDAN	542
COMPARISON OF FIVE DEVELOPED POWER CYCLES IN THE FRAME OF WASTE HEAT RECOVERY ALPEREN TOZLU, AYŞEGÜL ABUŞOĞLU, EMRAH ÖZAHİ	549
BOBİNLEME İŞLEMİNİN REJENERE SELÜLOZİK LİF KARIŞIMLI İPLİKLE PERFORMANSI ÜZERİNDEKİ ETKİLERİNİN İNCELENMESİ ESIN SARIOĞLU, GÜLBİN FİDAN	<b>RİN</b> 558
ZrO2-5%CaO TERMAL BARİYER KAPLAMA (TBC) SİSTEMİNİN SICAK KOROZYON DİRENCİ kadir mert döleker	567
ÇEVRİMLİ VE İZOTERMAL SICAK KOROZYONUN NİKEL ESASLI SÜPER ALAŞIM MALZEMEYE ETKİSİ kadir mert döleker, abdullah cahit karaoğlanlı	579
COMPARATIVE ANALYSIS OF CLASSIFICATION TECHNIQUES FOR CRISI MANAGEMENT SYSTEMS SAED ALQARALEH, MERVE IŞIK	591
EFFECT OF SECONDARY AGING of EN AC 43200 ALUMINUM ALLOY to MECHANICAL PROPERTIES BEDRI BAKSAN IBRAHIM CELIKYUREK VIISUE KILIC	600





PERFORMANCE INVESTIGATION OF DISTRIBUTED ORTHOGONAL SPACE TIME BLOCK CODING BASED ON RELAY SELECTION IN WIRELESS COOPERATIVE SYSTEMS	E-
MOHAMMED K.,SIDDEEQY.AMEEN	606
FEASIBILITY STUDY OF AUTONOMOUS FREIGHT PLATOONING PETER JOHN KAY, CERİ MORRİS, KAROLİNA PAWLUCZAK ROCHA	616
<b>BEHAVIOUR OF LASER BEAM CHARACTERISTICS IN DIFFERENT FLUIDS</b> NECIP FAZIL YILMAZ, MUHAMMED PAKSOY, HAKAN ÇANDAR	623
<b>ÖRME KUMAŞLARA SİVRİSİNEK KOVUCU APRE UYGULAMASI</b> Fazlı bulut, kevser günal, esin sarioğlu, ahmet ağaç, şeyda açıkgöz	634
THE EFFECT OF DIFFERENT BED MATERIAL GRAIN SIZES ON THE EQUILIBRIUM SCOUR DEPTH AT THE LABYRINTH SIDE WEIR FLOW MUSTAFA TUNÇ, MUHAMMET EMIN EMIROĞLU	639
A NUMERICAL APPROACH TO ESTIMATE THE TENSILE STRENGTH OF STRUCTURAL LIGHTWEIGHT CONCRETE ESRA TUĞRUL TUNÇ, KÜRŞAT ESAT ALYAMAÇ, ZÜLFÜ ÇINAR ULUCAN	649
EFFECT OF LOADING RATE ON FLEXURAL RESPONSE OF AERATED CONCRETE COMPOSITE BEAMS DERYA BAKBAK, AHMET EMIN KURTOGLU	659
<b>r-GO /TiO2 NANOKOMPOZİTİNİN ÜRETİMİ, KARAKTERİZASYONU VE SERTLİK ÖZELLİKLERİNİN İNCELENMESİ</b> derya tekin, derya birhan, taner tekin	664
<b>PVA – TİO2 NANOKOMPOZİTİNİN ÜRETİMİ, KARAKTERİZASYONU VE ANTİBAKTERİYEL ÖZELLİKLERİNİN İNCELEMESİ</b> derya tekin, derya birhan, taner tekin	671
<b>USING OF MICROCAPSULES IN HOT MIX ASPHALTS FOR SELFHEALING</b> ERKUT YALÇIN, AHMET MÜNİR ÖZDEMİR, MEHMET YILMAZ, BAHA VURAL KÖK	677
DISCONTINUOUS PRECIPITATION IN METALLIC ALLOYS ZAKARIA BOUMERZOUG	687





BONCUKLANMA GÖRÜNTÜLERİNİN İŞLENMESİNDE FARKLI FİLTRELE Kul lanımı	RİN
ABDURRAHMAN TELLİ	695
TUNING THE PROPERTIES OF NANOCOMPOSITE FIBERS BY CONTROLI THE SPINNING SOLUTION AND PROCESSING CONDITIONS	LING
HATICE AYLIN KARAHAN TOPRAKCI, AYSE TURGUT, OZAN TOPRAKCI, MUHSINE ZORCAN	702
EFFECTS OF SOLVENT RATIO ON ELECTRICAL AND MORPHOLOGICAL PROPERTIES OF CARBON NANOFIBER FILLED POLYMER ANOCOMPOS AYSE TURGUT, OZAN TOPRAKCI, HATICE AYLIN KARAHAN TOPRAKCI	<b>ITES</b> 708
ELECTROSPUN POLYVINYLIDENE FLUORIDE FIBERS HATICE AYLIN KARAHAN TOPRAKCI, OZAN TOPRAKCI	713
MECHANICAL PROPERTIES OF POLYPROPYLENE/THERMOPLASTIC ELASTOMER BLENDS AYSE TURGUT, OZAN TOPRAKCI, HUSEYIN CAGDAS ASLAN, HATICE AYLIN KARAHAN TOPRAKCI	719
DESIGN AND PERFORMANCE EVALUATION OF A SELF-COOLING CONCENTRATING PHOTOVOLTAICS SYSTEM SHOUKAT ALİM KHAN, YUSUF BİCER, SAMİ G. AL-GHAMDİ, MUAMMER KOÇ	724
ANTEP FISTIĞI KABUKLARINDAN BİYOKÖMÜR ÜRETİMİ Ali keskin, elif arancı öztürk, nadir şengül, firat elibol, mesut aktaş, mustafa boyrazlı	734
CORROSION PROPERTIES OF DISTALOY AE ALLOYS PREPARED BY P/M METHOD IN 1M KOH SOLUTION AYŞE NUR ACAR, RASİHA NEFİSE MUTLU, ABDUL KADİR EKŞİ, AHMET EKİCİBİL	<b>I</b> 743
SPORTSWEAR FABRIC SELECTION FOR DIFFERENT WEATHER CONDIT USING AHP AND TOPSIS ANALYSIS	TIONS
BURAK SARI, NIDA OGLAKCIOGLU THE EFFECT OF USING SILVER BLENDED YARNS ON THE ABRASION RESISTANCE PROPERTY OF WOVEN FABRICS ZEHRA KAYNAR TAŞCI, NİHAT ÇELİK	752



ME

# EFFECT OF NANO SLICA WITH EPOXY RESIN BONDED SINGLESTRAP REPAIRS

HARUN KARAOĞLAN, AHMET ERKLİĞ, NURETTİN FURKAN DOĞAN





BURAK CIFTCIOGLU, IBRAHİM GOV, M. HANİFİ DOGRU





Cold Street



# AL2024 KOMPOZİTLERİN AŞINMA KARAKTERİSTİĞİ

he International Conference

aterials and Engineering Technology

## ENGİN ERGÜL<sup>\*1</sup>, HALİL İBRAHİM KURT<sup>2</sup>, CAN ÇİVİ<sup>3</sup>, GÖKHAN EYİCİ<sup>3</sup>

<sup>1</sup> Dokuz Eylül Üniversitesi, İzmir Meslek Yüksekokulu, Teknik Programlar Bölümü, İzmir, TÜRKİYE.
<sup>2</sup> Gaziantep Üniversitesi, Mühendislik Fakültesi, Metalürji ve Malzeme Mühendisliği Bölümü, Gaziantep, TÜRKİYE.
<sup>3</sup> Celal Bayar Üniversitesi, Mühendislik Fakültesi, Makine Mühendisliği Bölümü, Manisa, TÜRKİYE.

## Özet

Kompozit malzemelerin geleneksel malzemelerle karşılaştırıldığında dayanıma bağlı olarak yoğunluk ve elastisite modülü özelliklerinin yüksek olması bu malzemelere talebi arttırmıştır. Ek olarak bu malzemelerin daha yüksek dayanım ve rijitliğe sahip olması, makine elamanının ağırlığının azalması anlamına gelmektedir. AL 2024 matrisli kompozit malzemelerin üretiminde kokil kalıba döküm yöntemi kullanılmış olup deneylerdeki aşınma ağırlık kaybı olarak ele alınmıştır. Bu çalışmada ağırlıkça %50 MGO ve %50 MWCNT karıştırılmış ve %0,2 - %0.5 - %1 - %2 oranlarında matris malzemesine ilave edilmiştir. MGO/MWCNT katkılı AL 2024 kompozitlerin aşınma davranışı ball-on-disk aşınma test cihazı kullanılarak incelenmiştir. Elde edilen değerler karşılaştırılarak kompozit malzemelerin aşınma özellikleri üzerindeki etkileri ortaya konulmuştur. 5N yük etkisi altında 250 m mesafede aşınma deneyine tabi tutulan kompozit malzemenin ağırlık kaybı göz önünde bulundurularak yüzde aşınma miktarları incelenmiştir.

Anahtar Kelimeler: Kompozit, Aşınma, Al 2024, CNT.

#### 1. Giriş

Alüminyum alaşımları mühendislik malzemelerinin geliştirilmesinde önemli bir rol oynamaktadır. Bunun nedeni, mekanik işleme, biçimlendirme ve ısıl işlem gibi özelliklerini farklı yollarla geliştirme yeteneklerinden kaynaklanmaktadır. Ek olarak, farklı yapıdaki çok çeşitli takviye malzemeleri kullanılarak alüminyum bazlı kompozitler oluşturma yeteneklerinin yüksek olması bir avantajdır. Alkompozitlerin üretimi için takviye malzemelerinin kullanımındaki amaç, mukavemet, sertlik ve aşınma direnci gibi spesifik özellikleri geliştirerek alüminyumun mekanik performansını en üst düzeye çıkarmaktır [1].

Metal Matris Kompozitlerine takviye edilen elementler, özelliklerinin iyi yönde değiştirebilir ve bunların uygulama alanlarını genişletebilir. Kompozitlerin özelliklerinin değiştirilebilmesi için boyut ve takviye oranları iyi kontrol edilmelidir. Uçağın keşfedilmesiyle birlikte hafif ve yüksek dayanımlı malzemelere olan ihtiyaç artmaktadır [2]. Al alaşımları, havacılık sektöründe düşük yoğunluklu ve yüksek özgül dayanıma sahip olması nedeniyle yüksek talep gören bir malzemedir [3]. Malzemelerin mukavemeti ve sertliği arttıkça, belirli bir yük taşıyıcı uygulama için gerekli olan malzemenin boyutları ve kütle azalmalıdır. Bu durum uçak ve otomobillerde yük artışı ve yakıt verimliliğinin iyileştirilmesi gibi birçok avantaja yol açmaktadır [2].

Takviye malzemesi olarak da dayanımları yüksek olduğu ve hafif olduğu için genellikle SiC, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> ve MgO gibi malzemeler tercih edilmektedir [4, 5]. Karbon nanotüp (CNT), termal iletkenlik, mekanik ve elektriksel özelliklerinin iyi olmasından dolayı malzeme veya dolgu malzemesi olarak kullanılmaktadır [2]. Karbon nanotüpler (CNT'ler) yalnızca seramiklerden çok daha yüksek mekanik özelliklere sahip değildir. Aynı zamanda yüksek termal ve elektriksel iletkenlik gibi diğer işlevsel özelliklere de sahiptir. CNT/Al kompozitler döküm, toz metalurjisi ve sürtünme karıştırma gibi geleneksel malzeme hazırlama teknolojileri kullanılarak üretilen ve yaygın olarak kullanılan bir malzemedir [3].



#### 2. Malzeme ve Yöntem

Bu çalışmada alüminyum 2024 serisi matris malzemesi olarak kullanılmıştır. Ağırlıkça %50 MGO ve %50 MWCNT karıştırılmış ve %0,2 - %0.5 - %1 - %2 oranlarında matris malzemesine ilave edilmiştir. Çoğul duvarlı karbon nanotüp (MWCNT) ve magnezyum oksit (MgO) nano partikül karışımı güçlendirici olarak kullanılmıştır.



Şekil 1. Ball-on-Disk aşınma cihazı

Aşınma testleri oda sıcaklığında bir yağlayıcı olmadan, CSM Instruments marka cihazda ball-ondisk makinesi üzerinde gerçekleştirilmiştir (Şekil 1). Deneylerde; 5 N yük ile 1,0 m/s kayma hızı kullanılmıştır. Kayma mesafesi olarak 250 m belirlenmiştir. Aşınma testlerinin amacı karıştırma döküm yöntemi ile üretilen metal matrisli kompozit malzemelerin içeriğine göre malzemenin ağırlık kaybı göz önünde bulundurularak yüzde aşınma miktarları incelenmesidir.

## 3. Sonuçlar ve Tartışma

Al alaşımları, havacılık sektöründe düşük yoğunluklu ve yüksek özgül dayanıma sahip olması nedeniyle kullanılmaktadır. Yaptığımız çalışmada, Al 2024 metal matris malzemesine farklı oranlarda MGO-MWCNT bileşiği ilave edilerek, malzeme üzerindeki aşınma etkisini görmek amacıyla 5 N yük ile 250 m mesafede aşınma testi yapılmıştır. Tablo 1, ve Şekil 1 ve 2 aşınma testi sonuçlarını göstermektedir.

AĞIRLIK KAYBI (g)		250 m		
MALZEME ÖZELLİKLERİ	YÜK	Ağırlık kaybı(g)	Ağırlık kaybı (%)	
Al 2024 baz		0,0018	-0,0307	
%50 CNT %50 MgO %0,2		0,0023	-0,0583	
%50 CNT %50 MgO 0,5	5 N	0,0046	-0,0521	
%50 CNT %50 MgO %1		0,0022	-0,0235	
%50 CNT %50 MgO %2		0,0028	-0,0355	

Tablo 1. Aşınma	testi sonucu	ağırlık kaybı	ı (veya	yüzdesi)	(g / %)
,		<u> </u>	<pre></pre>		



Şekil 1. Aşınma testi ağırlık kaybı



Şekil 2. Aşınma testi yüzde kayıp

Mekanik özellikleri belirleyecek deneylerde kompozit numunelerin abrasif aşınma davranışları incelenmiştir [6, 7]. Aşınma testi sonucunda malzemelerde aşınma kaybı artış trendinde iken %0,5 güçlendiriciden itibaren azalma eğilimine dönmüştür. Maksimum ağırlık kaybı %0,2 güçlendirici ilavesiyle görülürken minimum ağırlık kaybı %1 güçlendirici ilavesiyle meydana gelmiştir. % ağırlık kaybı ise gram aşınma kaybı aynı trende eğilim göstermiştir. Malzemelerin sertlik değerlerinin artması kompozitin aşınma karakteristiğinin artmasıyla doğru orantılı olması fakat aşırı sertlik

artışının gevrekleşme eğilimini artmasına neden olabileceğiyle aşınma kaybının artmasına yol açması muhtemeledir. Bunun yanısıra mikroyapısal değişimler ve porozite içeriği de aşınma özelliklerinin arttırmaktadır. Al 2024 metal matriste MGO-MWCNT takviyesi sertlik özelliklerinin iyileşmesinde katkı sağlamaktadır [8, 9].

The International Conference of Materials and Engineering Technology

#### 4. Sonuçlar

Bu çalışmada, Al2024/CNT/MgO hibrid kompozitler karıştırma döküm yöntemi ile üretilmiş olup aşınma deneyi sonucunda numunelerde meydana gelen ağırlık kaybı göz önünde bulundurularak aşınma miktarları incelenmiştir. Aşınma davranışlarını lineer bir görüntü sergilemekle birlikte uygun oranlar seçilirse kompozitin aşınma davranışının arttırabileceği görülmüştür. Söz konusu bu çalışma için optimum güçlendirici içeriği ağırlıkça %1 MgO%50+CNT%50 karışımı olduğu görülmüştür.

#### Kaynaklar:

- 1. Pérez-Bustamante, R., et al., The effect of heat treatment on microstructure evolution in artificially aged carbon nanotube/Al2024 composites synthesized by mechanical alloying. Materials Characterization, 2017. **126**: p. 28-34.
- Topçu, İ., et al., Karbon Nanotüp (KNT) Takviyeli Ti-6Al-4V/KNT kompozitlerin aşınma davranışlarının incelenmesi. Gazi Üniversitesi Mühendislik-Mimarlık Fakültesi Dergisi, 2018. 2018(18-2).
- 3. Jiang, L.Y., et al., Preparation and mechanical properties of CNTs-AlSi10Mg composite fabricated via selective laser melting. Materials Science and Engineering: A, 2018. **734**: p. 171-177.
- 4. Vahdettin, K. and M. DEMİREL, Epoksi Reçine-MgO Polimer Matrisli Kompozit Malzemelerin Üretilmesi ve Pin On Disk Abrasiv Aşınma Özelliklerinin İncelenmesi. Fırat Üniversitesi Mühendislik Bilimleri Dergisi. **31**(1): p. 1-10.
- 5. Casati, R., et al., Effect of ball milling on the ageing response of Al2618 composites reinforced with SiC and oxide nanoparticles. Journal of Alloys and Compounds, 2017. **693**: p. 909-920.
- 6. Pul, M., Karbon Nanotüp (CNT) Ve Nano Grafen (G) Takviyeli Al 2024 Kompozitlerin Vorteks Yöntemiyle Üretilerek Aşınma Ve İşlenebilme Özelliklerinin İncelenmesi. Uluslararası Mühendislik Araştırma ve Geliştirme Dergisi. **11**(1): p. 370-382.
- 7. Akbarpour, M., S. Alipour, and M. Najafi, Tribological characteristics of self-lubricating nanostructured aluminum reinforced with multi-wall CNTs processed by flake powder metallurgy and hot pressing method. Diamond and Related Materials, 2018. **90**: p. 93-100.
- 8. Xavior, M.A., H.P. Kumar, and K.A. Kumar, Tribological studies on AA 2024– Graphene/CNT Nanocomposites processed through Powder Metallurgy. Materials Today: Proceedings, 2018. **5**(2): p. 6588-6596.
- 9. Rikhtegar, F., S. Shabestari, and H. Saghafian, Microstructural evaluation and mechanical properties of Al-CNT nanocomposites produced by different processing methods. Journal of Alloys and Compounds, 2017. **723**: p. 633-641.

# AA 8006 ALÜMİNYUM ALAŞIMI MUTFAK FOLYO ÜRETİMİNİN HADDELEME SÜRECİNDE OLUŞAN HATALAR ve ANALİZİ

The International Conference

aterials and Engineering Technology

# HÜSEYİN UZUN<sup>\*1</sup> ve ÖZGE AYAZ<sup>2</sup>

\*1 Sakarya Uygulamalı Bilimler Üniversitesi, Teknoloji Fakültesi, Sakarya, TÜRKİYE.
 2 Sakarya Uygulamalı Bilimler Üniversitesi, Teknoloji Fakültesi, Sakarya, TÜRKİYE.

#### Özet

Bu çalışmada, AA 8006 alüminyum alaşımı kullanılarak üretilen mutfak folyosunun haddeleme imalat sürecinde oluşan hata tiplerinin tespiti, analizi ve ne tür iyileştirmeler yapılabileceği ile ilgili çözüm önerilerinin sunulması hedeflenmiştir. Bu amaçla, benzer üretim parametrelerinin ve deformasyon oranlarının kullanıldığı, başlangıç levha kalınlığı farklı olacak şekilde üç adet mutfak folyo bobini imal edilmiştir. Haddeleme süreci esnasında oluşan hata tiplerinin makro incelemesi, çekme testi, SEM ve EDS analizleri yapılarak, hataların oluşum sebepleri ve hangi üretim kademesinde meydana gelebileceği belirlenmiştir. Sonuç olarak, imal edilen mutfak folyo bobinlerinin %50 deformasyon oranına sahip yedinci haddeleme kademesinde, yüzey kazıntı izi, birbiri peşi sıra takip eden kesikli yırtılma ve iğne deliği (pinhole) olmak üzere üç farklı hata tipi belirlenmiştir. Bu hata tipleri, SEM ve EDS analiz teknikleriyle incelenerek hataların hangi sebeplerden kaynaklandığı değerlendirilmesi yapılmıştır.

Anahtar kelimeler: AA 8006 alüminyum alaşımı, mutfak folyosu, yüzey kazıntı izi, kesikli yırtılma, iğne deliği (pinhole)

#### 1. Giriş

Folyo, 0.2 milimetreden daha ince yapraklar hâline getirilmiş alüminyuma verilen bir addır. Çoğunlukla yiyeceklerin, ilaç, kozmetik ve kimyasal ürünlerin ambalajlanmasında kullanılmaktadır. Gıdaların saklanmasında da tercih edilen bir üründür. Alüminyum folyo esnektir, temiz bir yüzeye sahiptir, yüzeyinde leke ve delik içermez, homojen yüzey rengine sahiptir, hava geçirmez, kolay şekillendirilir, mekanik özellikleri iyidir ve toksik etki göstermez [1]. Alüminyum folyo üretiminde, folyo kalınlığının inceltilerek çok daha ince folyo imalatı günümüz araştırmacılarının konuları arasında yer almaktadır. Bu amaçla alüminyum alaşımı içeriğinin değiştirilerek veya üretim sürecini iyileştirerek, 6.5 mikron ve altı kalınlıklarındaki folyo imalatını gerçekleştirmeye çalışılmaktadır. Folyo imal edilecek alaşımın döküm süreçlerinden paketlemeye kadarki tüm aşamaların herhangi bir basamağında meydana gelebilecek hatalar enerji, zaman ve ekonomik kayıplara sebep olmaktadır. Bu nedenle hasarsız bir folyo üretim süreci büyük bir önem arz etmektedir [2].

Mutfak folyo imalatında yaygın olarak AA 8006 ve AA 8011 alaşımları kullanılmaktadır [3]. Bu çalışma kapsamında, AA 8006 alüminyum alaşımından aynı üretim kademeleri ve parametrelerine sahip olan üç folyo bobinin imalat sürecinde meydana gelebilecek hata türleri ve sebepleri araştırılmıştır. Bobinlerin hatalı bölgelerine yapılan SEM ve EDS analizleri ile makro incelemede yorumlanan hata tiplerinin deneysel tespiti yapılmıştır. Tespit edilen hata tiplerinin üretimin hangi kademesindeki eksikliklerden dolayı meydana gelebileceği ve hataların oluşumunun engellenmesi için alınması gereken önlemlerin neler olabileceği ile ilgili çözüm önerileri sunulmuştur.



## 2. Deneysel Çalışmalar

Bu çalışmada, AA 8006 (AlFe1.5Mn) alüminyum alaşımı kullanılarak, haddeleme yöntemiyle mutfak folyosu üretilmiştir. İyi şekillendirilebilme, yüksek yorulma ve tokluk özelliklerinden dolayı, folyo üretiminde kullanılan AA 8006 alüminyum alaşımının kimyasal içeriği ve mekanik özellikleri Tablo 1'de verilmiştir.

The International Conference of Materials and Engineering Technology

Tablo 1. AA 8006 alüminyum folyo alaşımına ait kimyasal içerik ve mekanik özellikler.

Alüminyum	Kimyasal İçerik (% ağırlık)								
Folyo Alaşımı	Si	Fe	Cu	Mn	Mg	Zn	Ti	Al	
	0.10 - 0.25	1.40 – 1.55	0.03	0.30 - 0.45	0.03	0.02	0.014 - 0.018	Kalan	
AA 8006	Mekanik Özellikler (Folyo kalınlığı 10 - 20 μm arası için)								
(AIF e1.5MIII)		Çekme Mukavemeti (MPa)					% Uzama		
85 - 140				2					

Bu çalışmada, benzer üretim parametrelerinin ve deformasyon oranlarının kullanıldığı, başlangıç levha kalınlığı farklı olacak şekilde üç adet mutfak folyo bobini imal edilmiştir. Tablo 2'de alüminyum folyo üretim kademelerindeki yöntem ve parametreler, her bir haddeleme sonrası ölçülen folyo kalınlıkları verilmektedir. Başlangıç levha kalınlığı, bobin 1 için 6.1200 mm; bobin 2 için 6.3220 mm; bobin 3 için 6.2980 mm olarak ölçülmüştür.



Üretimin birinci kademesinde, 8006 alüminyum alaşımının tane boyutlarını küçültmek ve şekillendirilebilirliğini arttırmak amacıyla 580°C 6 saat tavlanmıştır. İkinci kademede dört faklı deformasyon oranında haddeleme işlemine tabi tutulmuştur. Haddeleme işleminde %0,8 C, %2 Cr içeriğine sahip çelik merdaneler kullanılmıştır. Üçüncü kademede, haddelenen levhanın içyapısının toparlanması amacıyla 450°C 5 saat boyunca ara tavdan geçirilmiştir.

Technology



Üretim esnasında, dedektör yardımıyla hata tespiti yapılmış ve üretim sistemi durdurularak, maket bıçağı ile 1 metre boyunda folyo kesilerek bobinden alınmıştır. Hatalar, 20-50 lux şiddetindeki ışık altında gözle muayene edilmiştir. Folyonun mukavemet değerlerini tespit etmek amacıyla TS EN ISO 6892-1 standardında belirtilen çekme numune ebatları kullanılarak, Zwick Z1.0 çekme cihazında test yapılmıştır. Çekme numuneleri, yedinci kademede folyo hattından alınarak test edilmiştir. Bobin 1'deki numune kalınlığı 11.5 µm, Bobin 2 numune kalınlığı 10.5 µm, Bobin 3 numune kalınlığı 10 µm'dur. Çekme numunesi, hadde yönü doğrultusunda olup, numune ebatları Şekil 1'de gösterilmektedir.

Hatalı numunelerin, SEM fotoğrafları çekilmiş ve hata tipleri belirlenmiştir. EDS elementel analiz tekniği ile hata bölgelerinde hangi tür elementlere rastlandığı tespit edilmiştir. Bu element tespitine göre de hatanın hangi aşamada (döküm veya haddeleme) meydana geldiği değerlendirilerek yorumlanmıştır.



Şekil 1. Çekme numunesi ebatları

#### 3. Sonuçlar ve İrdeleme 3.1 Cekme Denevi Sonuçları

Tablo 3'de son folyo kalınlığı 11.5 µm olan Bobin 1, 10.5 µm olan Bobin 2 ve 10 µm olan Bobin 3'e ait çekme deneyi sonuçları gösterilmektedir. Çekme deneyi sonuçlarının Tablo 1'de verilen folyo kalınlığı 10 - 20 µm arasındaki folyodan beklenen mukavemet değerleri ile uyumlu olduğu görülmektedir. Folyo kalınlığı arttıkça hem akma ve çekme mukavemetleri hem de % uzama değerleri artmaktadır. Folyoya ait çekme eğrisi değişimini göstermek amacıyla, örnek olarak Şekil 2'de Bobin 1'e ait gerilme - % uzama eğrisi verilmiştir.

	Mekanik Özellikler					
Bobin No	bin No Folyo Kalınlığı Akma (μm)	Akma Mukavemeti (MPa)	Çekme Mukavemeti (MPa)	% Uzama		
Bobin 1	11.5	95	108	5.8		
Bobin 2	10.5	91	104	5.3		
Bobin 3	10	87	100	4.9		

Tablo 3. AA 8006 alüminyum folyonun son kalınlığa bağlı olarak değişen çekme deneyi sonuçları.



Şekil 2. Bobin 1'e ait gerilme - % uzama eğrisi

# 3.2 Makro Yüzey İncelemesi ve Hata Tipleri 3.2.1 Yüzey Kazıntı İzi Hata Tipi

Yüzey kazıntı izi hata tipi olarak adlandırılan folyo hatası, Bobin 1'in üretim sürecinde ve 7. kademesinde tespit edilen bir hata tipidir. Yüzey kazıntı iz hata tipi, folyonun haddeleme yönü doğrultusunda uzayarak ilerleyen bir hata tipi şeklindedir [4]. Şekil 3'te yüzey kazıntı izinin, 35 lux ışık şiddetinde çekilmiş makro görünümü gösterilmektedir.



Şekil 3. Yüzey kazıntı iz hata tipi makro görünümü

Meydana gelen yüzey kazıntı izinin SEM incelemesinde, kazıntı oluşmadan önce folyo yüzeyine batmış halde bulunan partiküllerin mevcut olduğu tespit edilmiştir. Bu partiküllerin haddeleme esnasında folyo yüzeyinde yuvarlanması ve hadde miline teması sonucunda folyoda yüzey kazıntı izinin oluşmasına sebep olduğu tahmin edilmiştir. Şekil 4'te tespit edilen bu partiküllerin SEM görüntüsü verilmektedir.

Technology



Şekil 4. Yüzey kazıntı izinin oluşumuna sebep olan partiküllerin SEM görüntüsü.

Yüzey kazıntı izi hata tipinin oluşumuna sebebiyet verdiği düşünülen partiküllerin, üretimin hangi aşamasında oluşabileceği ile ilgili değerlendirme yapılabilmesi amacıyla, elementel analizleri yapılmıştır.

Şekil 4'te gösterilen birinci partikülden alınan elementel analiz sonuçları, Şekil 5 (a)'da ve ikinci partikülden alınan elementel analiz sonuçları da Şekil 5 (b)'de gösterilmektedir. Birinci partikülden Mg ve S sinyalleri alınmıştır. İkinci partikülden Mg, S, Al ve O sinyalleri alınmıştır.

Mg elementi sinyali, AA 8006 folyo alaşımından gelmiş olabileceği tahmin edilmiştir. S elementi sinyali ise döküm esnasındaki cüruftan gelme ihtimali söz konusudur. Yüzey kazıntı izinin iki ihtimale bağlı olarak meydana gelebileceği yorumunda bulunulmuştur. Birinci ihtimal, döküm esnasında alaşımın mikroyapısında Mg elementinin homojen dağılımının sağlanamaması ve Mg topaklanması oluşması sebebiyle, deformasyon sürecinde de sertleşen topaklanmış Mg partikülleri, belirli bir deformasyon sürecinden sonra folyo yüzeyinde kazıntı izi yapmış olmasıdır. İkinci ihtimal ise merdanenin yüzey pürüzlülüğüne bağlı olarak meydana gelmesidir. Periyodik olarak taşlanan merdaneler, gerekli hassasiyet ile taşlanmazlar ise merdane yüzeylerinde pürüzler meydana gelmektedir [5]. Bu pürüzler ise folyo yüzeyinde kazıntı izine sebep olabilir. Bu nedenle periyodik merdane bakımının hassas taşlama işçiliği gerektiği ve taşlama sonrası merdane yüzey muayenesinin dikkatli bir şekilde yapılması gerekliliği önemle önerilmiştir.

## 3.2.2. Kesikli Yırtılma Hata Tipi

Kesikli yırtılma hata tipi olarak adlandırılan folyo hatası, Bobin 2'nin üretim sürecinde ve 7. kademesinde tespit edilen bir hata tipidir. Kesikli yırtılma hata tipi, folyonun haddeleme yönü doğrultusunda meydana gelen bir hata tipi şeklidir. Şekil 6 (a)'da yüzey kesikli yırtılmanın, 35 lux ışık şiddetinde çekilmiş makro görünümü gösterilmektedir. Hata fermuar dişleri görünümündedir. Şekil 6 (b)'de ise bu hata bölgesinin çok küçük bir bölümün SEM görüntüsü gösterilmektedir. SEM görüntüsü incelendiğinde, kopan bölgenin etrafında birden fazla kopmanın meydana geldiği ve tabaka şeklindeki çatlakların varlığı gözlemlenmiştir.



Şekil 5. Şekil 4'te gösterilen (a) birinci ve (b) ikinci partikülden alınan elementel analiz sonuçları



Şekil 6. Kesikli yırtılma hata tipinin (a) makro görünümü (b) SEM görüntüsü

Kesikli hata tipinin meydana gelmesine sebebiyet verdiği düşünülen çatlak görünümündeki bölge içerisinde EDS analizi yapılmıştır. Şekil 7 (a)'da gösterilen 3 numaralı noktadan alınan elementel analiz sonuçları Şekil 7 (b)'de, 4 numaralı noktadan alınan elementel analiz sonuçları ise Şekil 7 (c)'de gösterilmektedir. 3 ve 4 numaralı noktalardan C, O, Al, Si, Mn ve Fe sinyalleri alınmıştır.

Kesikli yırtılma hata tipinin meydana gelme şeklinden oksit esaslı bir hata olabileceği tahmininde bulunulmuştur. EDS analizi sonucunda Oksijen ve Fe elementlerinin sinyalleri yüksek çıkmıştır. Fe elementinin yapıdaki oranının yüksekliğinin döküm prosesinden kaynaklı olabileceği düşünülmüştür. Ergime derecesi yüksek olan Fe elementi, yapıya homojen bir şekilde dâhil olamadığından, kümeleşen Fe elementinin haddelendikçe sertleşen bir yapıya dönüşmüş olabilir. Sertleşen Fe partikülleri kesikli yırtılmaya neden olabilir. Ayrıca Oksijen elementi ile Fe elementinin birleşerek "demiroksit" bileşiğinin meydana gelmiş olabileceği yorumu yapılmıştır. Demiroksit bileşiği haddelendikçe sertleşerek malzemenin oksitlenmeye başlamış zayıf bölgelerinde yırtılmaları meydana getirmiş olabileceği tahmininde bulunulmuştur.

Bu tür bir hatanın önüne geçebilmek için oksitlenme yaparak yüzeyde yırtılmalara sebep olan bu yapının, Fe ile bileşik yapmasını engellemek için Fe elementinin yapıya homojen bir şekilde karışmasını sağlamak gerekir. Döküm prosesinde sıvı metale tabletler halinde katılan Fe elementinin ergime derecesinin yüksek olması nedeniyle alaşımda homojen olması önem arz eder. Kullanılan Fe tabletlerinin kalitesi, yapıya homojen bir şekilde dahil olmasıyla orantılıdır. Bunun için döküm prosesinde kullanılan Fe tabletlerin kalitesine önem verilmesi gerektiği önerisinde bulunulmuştur.


Şekil 7. Şekil (a)'da gösterilen (b) 3 numaralı (c) 4 numaralı noktalardan alınan elementel analiz sonuçları

# 3.2.3 İğne Deliği (Pinhole) Hata Tipi

İğne deliği (pinhole) hata tipi olarak adlandırılan folyo hatası, Bobin 3'ün üretim sürecinde ve 7. kademesinde tespit edilen bir hata tipidir. İğne deliği (pinhole) hata tipi, folyonun haddeleme yönü doğrultusunda meydana gelen bir hata tipi şeklidir. Şekil 8'de iğne deliği (pinhole) hata tipinin 35 lux ışık şiddetinde çekilmiş makro görünümü gösterilmektedir.



Şekil 8. İğne deliği (pinhole) hata tipinin makro görünümü.



İğne deliği (pinhole) hata tipi olarak adlandırılan folyo hatası, Bobin 2'nin üretim sürecinde ve 7. kademesinde tespit edilen bir hata tipidir. İğne deliği (pinhole) hata tipi, folyonun haddeleme yönü doğrultusunda meydana gelen bir hata tipi şeklidir. Folyonun belirli bir bölgesel alanında yoğunlaşan, iğne deliği şeklindeki hatalardan oluşmaktadır [7]. Folyoda "pinhole" hatasını tanımlamak için standartlarda belirtilen kabul kriterleri dikkate alınır. Folyo kalınlığına bağlı olarak 1 m²'lik alan içerisinde yer alan delik sayısı, standartta belirtilen maksimum değeri geçiyor ise folyo hasarlı olarak tanımlanır. Bobin 3'ün folyo kalınlığı 10 µm'dur. Bu kalınlıktaki bir folyoda, 1 m²'lik alan içerisinde A kalitesi için 50'den fazla delik sayısı olması durumunda folyo hasarlı olarak tanımlanır [6]. Şekil 9'da iğne deliği (pinhole) hata tipinin SEM görüntüsü gösterilmektedir. SEM incelemesi sonucunda, 1 m²'lik alan içerisinde 60 – 70 delik sayısı tespit edildiği için folyo pinhole delik hata tipi nedeniyle hasarlı olarak belirlenmiştir.



Şekil 9. İğne deliği (pinhole) hata tipinin (a) SEM ve (b) EDS alınan noktaların görüntüleri

Şekil 9 (b)'de, SEM görüntüsü verilen "pinhole" hata tipinin yer aldığı hatalı bölgesinin 3 ve 4 nolu noktalarından alınan EDS elementel analiz sonuçları Şekil 10'da verilmiştir. 3 numaralı noktasından alınan Şekil 10 (a)'da, 4 numaralı noktasından alınan elementel analiz sonuçları ise Şekil 10 (b)'de gösterilmektedir. 3 numaralı noktadan O, Al, Mg ve Fe sinyalleri alınmıştır. 4 numaralı noktadan ise O, Al, Si, Cr ve Fe sinyalleri alınmıştır.

Fe, O ve Cr elementlerinin sinyalleri, AA 8006 alaşımın içeriğindeki oranlarından daha yüksek olduğu tespit edilmiştir. Fe ve O elementinin birlikte yüksek sinyal vermesi "demiroksit" bileşiği partiküllerinin varlığını hatırlatmaktadır. Haddelemede kullanılan merdanelerin krom kaplı olmasından dolayı Cr elementinden alınan sinyalin, merdane kaynaklı olabileceği düşünülmüştür. Meydana gelen demiroksit bileşiğinin sertleşerek yüzeyden kopmuş ve merdaneye yapışarak haddeleme esnasında pinhole adı verilen deliklerin meydana gelmesine sebep olabileceği tahmin edilmiştir. Kopan bu parçanın merdane yüzeyine yapışmış olmasıyla haddelenen malzemede izler meydana getirmiş olabileceği yorumunda bulunulmuştur.



**Şekil 10.** Şekil 9 (a)'da gösterilen (a) 3 numaralı ve (b) 4 numaralı noktalardan alınan elementel analiz sonuçları

## 4. Genel Sonuçlar

AA 8006 alüminyum alaşımı kullanılarak üretilen mutfak folyosunun haddeleme imalat sürecinde oluşan hata tipleri belirlenmiş, SEM ve EDS analizleri yapılmış ve ne tür iyileştirmeler yapılabileceği gerektiği ile ilgili önerilerde bulunulmuştur. İmal edilen mutfak folyo bobinlerinin %50 deformasyon oranıyla son kalınlıklarına haddelendikleri yedinci kademesinde, yüzey kazıntı izi, birbiri peşi sıra takip eden kesikli yırtılma ve iğne deliği (pinhole) olmak üzere üç farklı hata tipi belirlenmiştir.

Yüzey kazıntı izi, döküm esnasında alaşımın mikroyapısında Mg elementinin homojen dağılımının sağlanamaması neticesinde deformasyon sürecinde sertleşen topaklanmış Mg partikülleri, belirli bir deformasyon sürecinden sonra folyo yüzeyinde kazıntı izi yapmıştır. Ayrıca merdanenin yüzey pürüzlülüğüne bağlı olarak yüzey kazıntı izi oluşmuştur.

Kesikli yırtılma hata tipinin demiroksit esaslı bir hata olabileceği tahmininde bulunulmuştur. Oksijen elementi ile Fe elementinin birleşerek "demiroksit" bileşiğinin folyo haddeleme sürecinde sertleşerek folyo yüzeyinde yüzey kazıntı izi hatasının oluşumuna sebep olmuştur.

İğne deliği (pinhole) hata tipi, demiroksit bileşiğinden ve merdane yüzeyine yapışan partiküller nedeniyle meydana gelebileceği değerlendirilmiştir.

## Teşekkür

Teknik desteklerinden dolayı, ASAŞ Alüminyum Sanayi ve Ticaret A.Ş. şirket yetkililerine teşekkür ederiz.

## Kaynaklar

- 1. Rahimi, M. Fojan, P., Gurevich, L., Afshari, A. Aluminium Alloy 8011: Surface Characteristics", Applied Mechanics and Materials, **2015**, Vols. 719-720, pp. 29-37.
- 2. <u>https://www.assanaluminyum.com/tr-tr/surdurulebilirlik/her-yonuyle-aluminyum</u>, 2019.
- 3. http://www.aluminum-foil.org/product/Aluminium-Household-Foil.html, 2019.
- 4. Çatalbaş B. Z. Olası Hata Türü Ve Etkisi Analizinin Alüminyum Levha Ve Folyo Üretiminde Uygulanması, Yüksek Lisans Tezi, İstanbul Teknik Üniversitesi, **2001.**
- 5. Yıldız Y. Alüminyum Folyo Haddelemede Kullanılan Merdanelerin Taşlama Operasyonu İle Kazandıkları Yüzey Özelliklerinin Haddeleme Prosesine Etkileri, Yüksek Lisans Tezi, İstanbul Teknik Üniversitesi, **2001.**
- 6. <u>http://aluminiumfoils.blogspot.com/2015/06/light-gauge-aluminium-foils.html</u>, 2019.
- Kalyoncu Z. İnce Alüminyum Folyolarda İğne Deliği Oluşumları ve Solvent Bazlı Mürekkeplerle Islanma Kabiliyetleri, Yüksek Lisans Tezi, İstanbul Teknik Üniversitesi, 2001.



## UFUK ASIL, ABDULLAH SECGİN

Dokuz Eylül University, Department of Nanoscience and Nanoengineering, İzmir, TURKEY Dokuz Eylül University, Department of Mechanical Engineering, İzmir, TURKEY

### Abstract

Now the number of components on integrated circuits has reached the limits of Moore's law. Memristor technology has taken attentions from the researchers as it is a future technology. Memristor have several advantages over its competitors due to the fact that their low power consumption, low space occupation and its ability to hold data even when the system power is cut. In this study, several type of logic designs with memristors in crossbar array was simulated in Virtuoso Cadence Design Environment program.

Keyword: Memristor, Logic gates, Crossbar, Virtuoso Cadence.

### 1. Introduction and Literature Summary

The word Memristor was first introduced by L.O. Chua in 1971 in addition to Resistor, Capacitor and Inductor. Memristor is the fourth basic two terminal circuit element that shows the nonlinear connection between magnetic flux and electric charge.[1] The relation between the memristor, capacitor, inductor and resistor are shown on the Figure 1.



Figure 1. The relation between the memristor, capacitor, inductor and resistor[1]

Besides Chua predicted theoretically, memristor was physically found in 2008. However, it was only in 2008 when researchers in HP Lab could confirm L.O. Chua's ideas with a memristor device manufactured using Pt/TiO2/Pt. Structure of HP memristor is shown in the Figure 2. Later, there has been lot of research in memristors, for various memory implement and logic design applications. Memristors can be produced on a crossbar cluster with very small cell, which potentially can result in big storage density combine with less power consumption and reasonably rapid switching times[2].



Figure 2. Structure of HP memristor[2]

From 2008 to this day, a lot of logic design pattern using memristors have been proposed like "IMPLY", "MAGICNOR", "OR", "AND" which can be mapped to the crossbar. However, these logic designs cannot be directly mapped to crossbar and hereby have restricted applicability.



Figure 3. Simple crossbar array

A memristor crossbar array is showed in the figure 3. These bitlines and wordlines are bonded by memristors and they are suitable to hold logical values such as logic 1 and logic 0. In this paper, several type of logic designs with memristors in crossbar systems was simulated in Virtuoso Cadence Design Environment program.

## 1. Imply Gate In Crossbar Array

This process, however, wants consecutive voltage activation at different locations within the circuit. Additionally, with imply, the result is stored by one of the inputs and not by a dedicated output memristor. Stateful logic also requires additional circuit components (for example, a controller and an extra resistor within every row of the crossbar), distributes high power, and has high computational complication. Truth table of IMPLY is given in Table 1 and Schematic of IMPLY is shown in the Figure 4.[4]

Table 1.	Truth	table	of IMPLY	gate
----------	-------	-------	----------	------

	Р	Q	P IMP Q
1	1	1	1
2	1	0	0
3	0	1	1
4	0	0	1



Figure 4. Schematic of a IMPLY gate

 $\begin{array}{ll} V\_cond & : 500 \mbox{ mV} \\ V\_set & : 2V \\ R_{off} & : 100 \mbox{ Ohm} \\ R_{on} & : 100k \mbox{ Ohm} \\ R_{g} & : 200 \mbox{ Ohm} \end{array}$ 

UWE ditte

values were used on imply Schematic in Cadence Virtuoso and IMPLY gate within a crossbar array is shown Figure 5 and Figure 4.



**Figure 5.** IMPLY gate within a crossbar array

18



Figure 6. Analysis of IMPLY gate within a crossbar array

The results of our analysis using IMPLY gate on a crossbar in Cadence Virtuoso are shown in the Figure 6.

## 2. MagicNor Gate In Crossbar Array

The logical condition in a MagicNor gate is offered as a resistance, where the high and low resistances are considered, respectively, as logical zero and one (logic 0 is  $R_{off}$  and logic 1 is  $R_{on}$ ). The inputs and output of the logic gates are the logical states of the memristors. Before performing gate operations, the input memristors must be initialized to their needed states, and the output memristor initialized to logic 1 for noninverting gates such as "OR" and "AND" logic 0 for inverting gates such as "NOR", "NAND" or "NOT" gates. During assessment, a voltage  $V_{dc} = 1V$  is applied to the input memristors, and the output terminal is grounded. Truth table of MagicNor is given in table 2 and Schematic of MagicNor is shown in the Figure 7.[3]

	Р	Q	P MagicNor Q
1	1	1	0
2	1	0	0
3	0	1	0
4	0	0	1

Table	2.	Truth	table	of	MagicNor	gate
I GOIC		11um	uuuu	O1	magiorior	Suco



Figure 7. Schematic of a MagicNor gate

- $V_{dc}$  : 1v
- $R_{on}$  : 50 Ohm
- R<sub>off</sub> : 1k Ohm

Valu es were used on MagicNor Schematic in Cadence Virtuoso and MagicNor gate within a crossbar array is shown Figure 8.



Figure 8. MagicNor gate within a crossbar array

¢.



Figure 9. Analysis of MagicNor gate within a crossbar array

The results of our analysis using MagicNor gate on a crossbar in Cadence Virtuoso are shown in the Figure 9.

## 3. NOT Gate In Crossbar Array

To make NOT gate, we are using MagicNor gate. If we make one of the inputs "0" at gate MagicNor, we can get NOT gate. Truth table of NOT is given in table 3 and Schematic of NOT is shown in the Figure 10.[3]

Tablo 3. Truth table of NOT gate



Figure 10. MagicNor gate schema with single input "0"

High resistance of one of the inputs provides a design as in Figure 11.



Figure 11. NOT gate, simplified single entry MagicNor gate



Figure 12. NOT gate within a crossbar array

Not gate within a crossbar array is shown Figure 12.

UWE Bristol

# 4. OR Gate In Crossbar Array

To make OR gate in crossbar system, we must use NOT gate, MagicNor gate and De Morgan Rules. Truth table of NOT is given in table 4. P OR Q = (P MagicNor Q)'



# Table 4. Truth table of OR gate

	Р	Q	P OR Q
1	1	1	1
2	1	0	1
3	0	1	1
4	0	0	0

- V<sub>dc</sub> : 1v
- $R_{on}$  : 50 Ohm

UWE ditter Bristol

•  $R_{off}$  : 1k Ohm

values were used on OR Schematic in Cadence Virtuoso and OR gate within a crossbar array is shown Figure 13 and Figure 14.



Figure 13. schematic of a OR gate



Figure 14. OR gate within a crossbar array



Figure 15. Analysis of OR gate within a crossbar array

The results of our analysis using OR gate on a crossbar in Cadence Virtuoso are shown in the Figure 15.

## 5. AND Gate In Crossbar Array

During assessment, a voltage  $V_{dc} = 1V$  is applied to the input memristor, and the output terminal is grounded. Truth table of AND gate is given in table 5 and schematic of AND gate is shown in the Figure 16.

Table 5.	Truth	table	of AND	gate
----------	-------	-------	--------	------

	Р	Q	P And Q
1	1	1	1
2	1	0	0
3	0	1	0
4	0	0	0

•  $V_{dc}$  : 1v

- $R_{on}$  : 50 Ohm
- R<sub>off</sub> : 1k Ohm

values were used on AND Schematic in Cadence Virtuoso and AND gate within a crossbar array is shown Figure 17.



Figure 16. schematic of a AND gate



Figure 17. AND gate within a crossbar array



Figure 18. Analysis of AND gate within a crossbar array

The results of our analysis using AND gate on a crossbar in Cadence Virtuoso are shown in the Figure 18.

## References

- 1. Chua, L. (1971). Memristor-the missing circuit element. IEEE Transactions on circuit theory, 18(5), 507-519.
- 2. Strukov, D. B., Snider, G. S., Stewart, D. R., ve Williams, R. S. (2008). The missing memristor found. nature, 453(7191), 80.
- Kvatinsky, S., Belousov, D., Liman, S., Satat, G., Wald, N., Friedman, E. G., ve Weiser, U. C. (2014). MAGIC—Memristor-aided logic. IEEE Transactions on Circuits and Systems II: Express Briefs, 61(11), 895-899
- 4. Kvatinsky, S., Satat, G., Wald, N., Friedman, E. G., Kolodny, A., ve Weiser, U. C. (2013). Memristor-based material implication (IMPLY) logic: Design principles and methodologies. IEEE Transactions on Very Large Scale Integration (VLSI) Systems, 22(10), 2054-2066.



aterials and Engineering Technology

## HÜSEYİN UZUN<sup>\*1</sup> ve MÜGE FİLİZ<sup>2</sup>

\*1 Sakarya Uygulamalı Bilimler Üniversitesi, Teknoloji Fakültesi, Sakarya, TÜRKİYE.
 2 Sakarya Uygulamalı Bilimler Üniversitesi, Teknoloji Fakültesi, Sakarya, TÜRKİYE.

### Özet

Bu çalışmada, %30 cam elyaf takviyeli PA66 kompozit malzemeden bağlantı destek parçasının, plastik enjeksiyon yöntemiyle çeşitli kalıp sıcaklıkları kullanılarak imalatında uygun kalıp sıcaklığının belirlenmesi hedeflenmiştir. Enjeksiyon yöntemiyle farklı kalıp sıcaklıklarında (20°C, 40°C, 60°C ve 80°C) üretilen parçanın kalıp sıcaklığına bağlı olarak oluşabilen hatalı veya hatasız ürünler makroskobik olarak incelenmiş ve optimum kalıp sıcaklık değeri belirlenmiştir. Bağlantı destek parçasının uygun geometrik tasarıma sahip olup olmadığını belirlemek amacıyla, bilgisayar destekli sanal çekme testi uygulanmıştır. Ayrıca kompozit ürünün yanma davranışını belirlemek amacıyla alev testi yapılmıştır. Sonuç olarak, tasarlanan bağlantı destek parçası, 60°C seçilen kalıp sıcaklığında başarıyla üretilmiştir. Diğer kalıplama sıcaklıklarında ise istenilen hatasız geometrik boyut elde edilememiştir. Alev testi sonucunda kompozit malzemenin yanma davranışının standartlarda belirtilen kriterleri sağladığı sonucuna varılmıştır. Sanal çekme testi sonucunda da tasarlanan geometrik boyutun uygun olduğu ve seçilen kompozit malzemenin istenilen mekanik özellikleri sağladığı belirlenmiştir.

Anahtar kelimeler: Cam elyaf takviyeli PA66 kompozit, plastik enjeksiyon kalıplama, kalıp sıcaklığı, alev testi, sanal çekme testi.

### 1. Giriş

Otobüs tavan rafı montajında kullanılabilecek bağlantı destek parçasının (bracket) tasarımsal geometrik şekli ve birleştirme elemanı olarak kullanılması sebebiyle en ekonomik ve seri üretim tekniği olarak plastik enjeksiyon kullanılmaktadır [1]. Enjeksiyonla üretiminin mümkün, tedarikinin kolay ve maliyetinin nispeten ucuz olması sebebiyle bağlantı destek parçası (bracket) üretimi için %30 cam elyaf içeren PA66 kompozit malzeme tercih edilmektedir.

Kompozit malzemeden enjeksiyon yöntemi ile başarılı bir şekilde ürün imal edilebilmesi ve tasarlanan geometrik ölçülere uygunluğunun sağlanabilmesi için enjeksiyon parametrelerinin uygun seçilmesi, büyük önem taşımaktadır [2]. Uygun seçilmeyen parametreler, ergimiş malzemenin erken katılaşarak kalıp boşluğunu tam olarak dolduramaması, kalıplanan üründe yanmaların oluşması, maksimum çekme ve eğilmelerin meydana gelmesi gibi hataların meydana gelmesi mümkün olmaktadır [3, 4]. Uygun seçilmesi gereken parametrelerden birisi de kalıp sıcaklığıdır. İmal edilecek parçanın geometrik şeklinin verildiği boşlukların yer aldığı iki veya daha fazla plakadan oluşan kalıplamada önemli bir parametre olarak değerlendirilmektedir. Kalıp sıcaklığının düşük veya yüksek olması enjeksiyon ürününde hataların oluşumuna sebep olan etkenlerden bir tanesidir.

Bu çalışmada, %30 cam elyaf içeren Poliamid 66 (PA66) kompozit malzemeden, otobüs tavan rafı montajında kullanılan bağlantı destek parçasının (bracket) enjeksiyon kalıplama tekniği ile üretilmesinde, en uygun kalıp sıcaklığının belirlenmesi hedeflenmiştir. Bu amaçla kalıp sıcaklığının

dışındaki enjeksiyon parametreleri sabit tutularak düşük, orta ve yüksek kalıp sıcaklıkları (20°C, 40°C, 60°C ve 80°C) seçilip optimum değerin tespit edilmesi amaçlanmıştır. Ayrıca kullanılan malzemenin yanma direnci ve tasarlanan bağlantı destek parçasının (bracket) geometrik şeklinin uygun olup olmadığı incelenmiştir.

he International Conference of Materials and Engineering Technology

## 2. Deneysel Çalışmalar

Bu çalışmada, %30 cam elyaf içeren Poliamid 66 (PA66) kompozit malzemeden, otobüs tavan rafi montajında kullanılan bağlantı destek parçasının (bracket) enjeksiyon kalıplama tekniği ile üretilmesi hedeflenmiştir. Üretilecek bağlantı destek parçasının tasarlanan geometrik teknik resmi Şekil 1'de ve otobüs tavan rafındaki montaj kısmı da Şekil 2'de gösterilmektedir.

Öncelikle %30 cam elyaf ve %70 Poliamid 66 mikser yardımıyla karıştırılarak, homojen kompozit karışım elde edilmiştir. Daha sonra hammadde kurutucusunda 80°C'de 4 saat bekletilerek kurutma işlemine tabi tutularak, kompozit karışımın nemi giderilmiştir.

Plastik enjeksiyon makinasına, Şekil 3'te gösterilen ve bağlantı destek parçasının üretilmesi amacıyla tasarlanan kalıp bağlanmıştır. Tablo 1'de verilen enjeksiyon kalıplama parametreleri kullanılarak, farklı kalıp sıcaklıklarında bağlantı destek parçası üretilmiştir. Kalıp sıcaklığı değiştirilirken diğer tüm parametreler (sıcaklık, basınç ve hız gibi) sabit tutulmuştur. Kalıp sıcaklığı 20°C, 40°C, 60°C ve 80°C olmak üzere dört farklı sıcaklık parametresi kullanılmıştır. Kalıp sıcaklığının ayarlanan değerlerde olup olmadığının takibi için temassız ultra-yüksek kızılötesi termometre kullanılmıştır. Enjeksiyon işlemi, HAITIAN marka plastik enjeksiyon makinesi ile gerçekleştirilmiştir. Farklı kalıp sıcaklığında üretilen bağlantı destek parçaları incelenerek en uygun kalıp sıcaklığı belirlenmiştir. Plastik enjeksiyon yöntemiyle üretilen bağlantı destek parçasının TSE ISO 3795 standardına göre alev direnci testi gerçekleştirilmiştir.

Ayrıca kompozit malzemeden üretimi gerçekleştirilen bağlantı destek parçasının geometrik tasarımının, malzeme seçiminin ve parçanın tasarım yüklerini karşılayacak sağlamlıkta olup olmadığını belirlemek amacıyla bilgisayar destekli tasarım ve analiz programı ANSYS ile sanal çekme testi gerçekleştirilmiştir.



Şekil 1. Üretilmesi tasarlanan bağlantı destek parçasının geometrik teknik resmi.

Şekil 2. Otobüs tavan rafında bağlantı destek parçasının montaj kısmı.

27



<b>Table 1.</b> Enjeksiyon kalıplama parametreleri.					
Kalıp sıcaklığı	Ergimiş kompozit sıcaklığı	Enjeksiyon basıncı	Enjeksiyon hızı		
(°C)	(°C)	(MPa)	(m/dak)		
20	240	50	45		
40	240	50	45		
60	240	50	45		
80	240	50	45		



Şekil 3. Bağlantı destek parçasının üretilmesi amacıyla tasarlanan kalıp.

## 3. Sonuçlar ve İrdeleme

3.1. Kalıp Sıcaklığının Bracket Üretimine Etkisi

3.1.1 Kalıp Sıcaklığı 20°C'de Üretilen Bracket

Plastik enjeksiyon yöntemiyle 20°C kalıp sıcaklığında %30 cam elyaf takviyeli Poliamid 66 kompozit malzemeden üretilen bağlantı destek parçasının (bracket) ön yüzey görünümü Şekil 4 (a)'da ve arka yüzey görünümü de Şekil 4 (b)'de gösterilmektedir.

Şekillerden de görüleceği gibi 20°C kalıp sıcaklığı kullanılarak üretilen bağlantı destek parçasının tasarlanan geometrik şeklini kalıp boşlukları içerisinde tamamlamadığı ve malzemenin homojen bir şekilde dağılımının olmadığı belirlenmiştir. Özellikle sol orta ve uç bölgelerinde daha belirgin bir şekilde eksik ürün hatasının mevcut olduğu görülmektedir.



Şekil 4. Kalıp sıcaklığı 20°C kullanılarak PA66 kompozit malzemeden üretilen bağlantı destek parçasının (bracket) (a) ön yüzey ve (b) arka yüzey görünümü.

Bu hata türü, düşük kalıp sıcaklığı olan 20°C de yapıldığından, kalıp boşluklarını malzemenin yeterli düzeyde dolduramamasından kaynaklanmaktadır. Eriyik malzeme kalıp içerisine dolduğu esnada, 20°C gibi düşük kalıp sıcaklığının olması sebebiyle, malzeme kalıp boşluklarını doldurmadan katılaşma gerçekleşmiş ve bu hata meydana gelmiştir. Kalıp sıcaklığının düşük olması malzemenin viskozitesini arttırarak ergiyik malzemenin yolluk ağzına uzak olan kısımlarında, özellikle de parçanın sol orta ve uç kısmında daha belirgin olduğu ve ergiyik malzemenin kalıbın tamamına ulaşamadan katılaşmaya sebep olduğu tahmin edilmiştir. Sonuç olarak, plastik enjeksiyon



yöntemiyle 20°C kalıp sıcaklığında %30 cam elyaf takviyeli Poliamid 66 kompozit malzemeden bağlantı destek parçasının üretimi için yetersiz olduğu tespitinde bulunulmuştur.

3.1.2 Kalıp Sıcaklığı 40°C'de Üretilen Bracket

Kalıp sıcaklığı 40°C kullanılarak üretilen bağlantı destek parçasının (bracket) ön yüzey görünümü Şekil 5 (a)'da ve arka yüzey görünümü de Şekil 5 (b)'de gösterilmektedir.



Şekil 5. Kalıp sıcaklığı 40°C kullanılarak PA66 kompozit malzemeden üretilen bağlantı destek parçasının (bracket) (a) ön yüzey ve (b) arka yüzey görünümü.

Şekillerden de görüleceği gibi 40°C kalıp sıcaklığı kullanılarak üretilen bağlantı destek parçasının tasarlanan geometrik şeklini kalıp boşlukları içerisinde tamamlamadığı ve malzemenin homojen bir şekilde dağılımının olmadığı belirlenmiştir.

Kalıp sıcaklığı 40°C kullanıldığında da malzemenin kalıp boşluklarını yeterli düzeyde doldurmadığı ve kalıp boşluklarının oluştuğu görülmektedir. Dolayısıyla 40°C'lik kalıp sıcaklığı da PA66 kompozit malzemeden bağlantı destek parçasının üretimi için yetersiz olduğu tespitinde bulunulmuştur.

## 3.1.3 Kalıp Sıcaklığı 60°C'de Üretilen Bracket

Kalıp sıcaklığı 60°C kullanılarak üretilen bağlantı destek parçasının (bracket) ön yüzey görünümü Şekil 6 (a)'da ve arka yüzey görünümü de Şekil 6 (b)'de gösterilmektedir.

Şekillerde de görüleceği gibi kalıp sıcaklığı 60°C kullanılarak üretilen bağlantı destek parçasının geometrik şekli tasarlanan ebatlardadır. Arzu edilen kalitenin de sağlanmış olduğu görülmüştür.

Kalıp sıcaklığı 60°C kullanıldığında, enjeksiyon malzemesinin kalıp boşluklarını tamamen doldurduğu görülmektedir. Kalıp sıcaklığı, 60°C'ye çıkarıldığı zaman enjeksiyon malzemenin viskozitesini düşürerek en ince cidarlardan akışı kolaylaştırarak kalıbın tamamını doldurmak ve eksik ürün oluşumunu önlemek mümkün olmuştur. Kalıp sıcaklığı 60°C seçildiğinde, 20°C veya 40°C kalıp sıcaklıklarında gözlemlenen tüm kalıp boşluklarının doldurulmaması hataları giderilmiş ve istenilen kalitede ürün üretimi gerçekmiş bulunmaktadır. Sonuç olarak, plastik enjeksiyon yöntemiyle 60°C kalıp sıcaklığında %30 cam elyaf takviyeli PA66 kompozit malzemeden bağlantı destek parçasının üretimi için yeterli olduğu tespitinde bulunulmuştur.



Şekil 6. Kalıp sıcaklığı 60°C kullanılarak PA66 kompozit malzemeden üretilen bağlantı destek parçasının (bracket) (a) ön yüzey ve (b) arka yüzey görünümü.

## 3.1.4 Kalıp Sıcaklığı 80°C'de Üretilen Bracket

Kalıp sıcaklığı 80°C kullanılarak üretilen bağlantı destek parçasının (bracket) ön yüzey görünümü Şekil 7 (a)'da ve arka yüzey görünümü de Şekil 7 (b)'de gösterilmektedir.

Kalıp sıcaklığı 80°C gibi yüksek bir sıcaklık değeri seçilerek yapılan bağlantı destek parçasının enjeksiyonunda, istenilen ebatlardaki geometrik şekil sağlanamamıştır. Parçasın sağ ve sol taraflarında bulunan toplam dört deliğin ebatlarının istenilen boyutta olmadığı belirlenmiştir. Malzeme kalıp boşluklarını tam doldurmuştur. Fakat enjeksiyon işlemi sonrasında kalıptan ürün çıkarıldıktan sonra gerçekleşen soğuma esnasında parçanın özellikle delik bölgelerinde minimal düzeyde şekil değişiklikleri meydana gelmekte ve geometrik bozukluk oluşmaktadır. Enjeksiyon işlemi belirlenen optimum kalıp sıcaklığından daha yüksek bir değer seçilmesi halinde arzu edilen verimim alınmadığı belirlenmiştir. Dolayısıyla 80°C'lik kalıp sıcaklığının, PA66 kompozit malzemeden bağlantı destek parçasının üretimi için yüksek olduğu tespitinde bulunulmuştur.



Şekil 7. Kalıp sıcaklığı 80°C kullanılarak PA66 kompozit malzemeden üretilen bağlantı destek parçasının (bracket) (a) ön yüzey ve (b) arka yüzey görünümü.

## **3.2.** Alev Direnci Sonuçları

Kalıp sıcaklığı 60°C'de üretilen bağlantı destek parçasının TSE ISO 3795 standardına göre yapılan alev direnci testi sonucunda, ateş çekildikten sonra 6 saniyede söndüğü ve damlama olmadığı tespit edilmiş olup istenilen yanma dayanımının sağladığı belirlenmiştir.

## 3.3 Sanal Çekme Testi Sonuçları

Şekil 8'de bilgisayar destekli tasarım ve analiz programı ANSYS R19.0 ile gerçekleştirilen sanal çekme testi sonucunda tasarlanan bağlantı destek parçasına ait toplam deformasyon analizi gösterilmektedir. Yapılan sanal çekme testi sonucunda tasarlanan bağlantı destek parçasının geometrik şeklinin uygun ve kullanılan kompozit malzemenin istenilen mekanik özelliklere sahip olduğu tespit edilmiştir.



Şekil 8. Sanal çekme testi sonucunda tasarlanan bağlantı destek parçasına ait toplam deformasyon analizi.

## 4. Genel Sonuçlar

Bu çalışmada, %30 cam elyaf içeren PA66 kompozit malzemeden, otobüs tavan rafı montajında kullanılan bağlantı destek parçasının (bracket) enjeksiyon kalıplama tekniği ile dört farklı kalıp sıcaklıklarında (20°C, 40°C, 60°C ve 80°C) üretilerek, en uygun kalıp sıcaklığının belirlenmesi doğrultusunda yapılan deneysel çalışmalardan elde edilen sonuçlar şunlardır:

- 1. Bağlantı destek parçasının (bracket) enjeksiyonla üretiminde 20°C ve 40°C kalıp sıcaklıklarının düşük olduğu ve erken katılaşma sebebiyle kalıp boşluklarının doldurmamasına sebep olan hataların oluşmasına neden olmaktadır.
- 2. Bağlantı destek parçasının (bracket) enjeksiyonla üretiminde 80°C kalıp sıcaklığı ise yüksek olduğu ve parçanın delik bölgelerinde ihmal edilemeyecek düzeyde geometrik şekil değişikliklerine sebep olmaktadır.
- **3.** Bağlantı destek parçasının (bracket) enjeksiyonla üretiminde 60°C kalıp sıcaklığının en uygun sıcaklık değeri olduğu tespit edilmiştir.
- **4.** Kalıp sıcaklığı 60°C'de üretilen bağlantı destek parçasının alev direnci testi sonucunda, ateş çekildikten sonra 6 saniyede söndüğü ve damlama olmadığı tespit edilmiş olup istenilen yanma dayanımının sağladığı belirlenmiştir.
- **5.** Bilgisayar destekli tasarım ve analiz programı ANSYS R19.0 ile gerçekleştirilen sanal çekme testi sonucunda tasarlanan bağlantı destek parçasının geometrik şeklinin uygun ve kullanılan kompozit malzemenin istenilen mekanik özelliklere sahip olduğu belirlenmiştir.

### Teşekkür

Teknik desteklerinden dolayı, KNS Otomotiv San. ve Tic. A.Ş. şirket yetkililerine teşekkür ederiz.

### Kaynaklar

- Eker Aydoğan, A., Plastik Matrisli Kompozitler, 2019, http://www.yildiz.edu.tr/~akdogan/lessons/imalattakompozit/Plastik\_Matrisli\_Kompozitler. pdf
- Hüner, Ü. ve Erdoğan, S., Cam Elyaf Takviyeli Polipropilen Kompozit Malzemelerin Üretim ve Bilgisayar Ortamında Kalıplama ve Takviye Malzemesine Bağlı Parametrelerinin Karşılaştırılması, 13. International Materials Symposium, 13-15 October 2010, Pamukkale University, Denizli, Turkey.
- 3. Kuldaşlı, A. B., Plastik Enjeksiyon Yöntemi ile Parça İmalatı, İmalat Hataları ve Hata Tespiti, **2005**, Yüksek Lisans Tezi, İ.T.Ü. Fen Bilimleri Esntitüsü, İstanbul.
- 4. Cansever, C.C., Effects of Injection Molding Conditions on the Mechanical Properties of Polyamide/glass Fiber Composites, **June 2007**, Master Thesis, Middle East Technical University, Turkey.



# POLİETİLEN FİLM YÜZEYİNİN, REAKTİF GAZ ORTAMINDA FONKSİYONELLEŞTİRİLMESİ

# CELAL SÖYLEMEZ<sup>\*1</sup>, İLKER TÜRKMEN<sup>2</sup>,

<sup>1</sup>Lidersan Sağlık ve Gıda A.Ş. ,Kimya Mühendisi, Gaziantep, TÜRKİYE <sup>2</sup>Lidersan Sağlık ve Gıda A.Ş. ,Kimyager, Gaziantep, TÜRKİYE

## Özet

Üretimini yaptığımız düşük yoğunluklu polietilen film (LDPE) ve poliolefin filmler genel anlamda bazı dezavantajlara sahiptirler. Bunlar; zayıf ısıl yapışma, düşük yüzey enerjisine sahip olduklarından dolayı basıla bilirliklerinin mümkün olmaması, sürtünme katsayılarının yüksek olması,film içerisindeki bazı katkılarının yüzeye migrasyonunun kolay gerçekleşebilmesi gibi bahsettiğimiz bu dezavantajları yüzeye doğrudan florlama işlemi yapılarak iyileştirilebilmesi amaçlanmıştır. . Doğrudan florlama, gaz halinde olan moleküler florin F2 (seyreltilmemiş veya başka bir karışım halinde) arasında heterojen bir reaksiyon olarak tanımlanır. Yaptığımız uygulamada;düşük yoğunluklu polietilen (LDPE) filme oda sıcaklığı ortamında %10 Flor ve %90 Azot gazı ile doğrudan florlama işlemi yapılmıştır. LDPE kimyasal bileşimindeki değişiklikler FTIR yapılarak incelenmiştir. Yüzey morfolojisi çeşitli mikroskopi teknikleri (SEM ve EDX Maping) ile incelenmiştir. Film özelliklerini daha da karakterize etmek için, yüzey enerjisi ölçümleri özel solisyonlar ile incelenip bant ile adezyon testi yapıldı. O2 bariyerliği ile ilgili analizler yapılmıştır. Isıl dirençliği ve mukavemet özellikleri incelenmiştir.

Anahtar Kelimeler : Düşük yoğunluklu polietilen, Doğrudan Florlama, Poliolefin film, Yüzey enerjisi, Adezyon,

### 1. Giriş

Yaygın olarak kullanılan polimerler, düşük maliyet ve işlenebilirlik gibi birçok avantaja sahiptir, ancak aynı zamanda birçok dezavantaja sahiptirler (genellikle zayıf yapışma, zayıf basılabilirlik ve bariyer özellikleri, düşük kimyasal direnç, vb.).). Ticari özellikleri geliştirilmiş özel polimerler (örneğin, flor içeren polimerler) tüm makale imal etmek mümkündür. Florlu polimerler, geliştirilmiş Kimyasal stabilite, termal stabilite, iyi bariyer özellikleri vb.gibi benzersiz özelliklere sahiptir. [1-2]. Bununla birlikte, flor içeren polimerler gibi özel olarak sentezlenmiş polimerlerin pratik kullanımı, yüksek maliyet ve sentez karmaşıklığı nedeniyle kısıtlanmıştır. Ancak çoğu zaman, polimer malların uygulama özellikleri esas olarak yüzey özellikleri ile tanımlanır. Bu nedenle, imal etmek gerekli değildir. Floropolimerlerden ancak daha basit, daha ucuz ve yaygın olarak kullanılan polimerlerden yapılmış eşyaların yüzey muamelesini uygulamak için daha uygun. Bu durumda, doğrudan florlama etkili bir şekilde kullanılabilir. Polimerlerin doğrudan florlanması, gaz halindeki F2'nin heterojen bir reaksiyonu ve bir polimer yüzeyi ile karışımlarıdır. Bu bir yüzey modifikasyonu yöntemidir: camsı polimerlerin çoğunluğu için, sadece üst yüzey tabakası modifiye edilir (kalınlıkta ~ 0.01–10  $\mu$ m), ancak dökme özellikleri değişmeden kalır.

Florlama, polimerlerin fizikokimyasal özelliklerini geniş bir aralıkta değiştirmek ve kontrol etmek için en etkili kimyasal yöntemlerden biri olduğundan, bu işlem büyük ilgi gören önemli bir araç haline gelmiştir. Doğrudan florlama endüstride kullanıldığında birçok avantaja sahiptir. Ana temel aşamaların yüksek ekzotermitesi nedeniyle, florlama, endüstriyel uygulamalar için yeterli bir oranda oda sıcaklığında kendiliğinden ilerlemektedir. Doğrudan florlama kuru bir teknolojidir. Herhangi bir şekle sahip polimer ürünler tedavi edilebilir Herhangi bir şekildeki polimer eşyaların yapışma özellikleri önemli ölçüde geliştirilebilir. Ayrıca, sürtünme katsayısı olabilirdüşük ve antibakteriyel özellikler ve kimyasal direnç geliştirilebilir [2,3,4,5].

The International Conference

aterials and Engineering Technology

### 2. Malzeme ve Yöntem

50 Mikron kalınlığında düşük yoğunluklu polietilen film kullanılarak Florlu ve Florsuz olmak üzere iki örnek üzerinden uygulamalar yapılmıştır.

Flor uygulaması oda sıcaklığında kuru olarak %90 Azot ve %10 Flor gazı ortamında in-line proses şeklinde yapılmıştır.

İki ürün arasında karşılaştırma yapabilmek için OTR, SEM,EDX, FT-IR, COF ve adezyon analizleri yapılmıştır.



Şekil.1 Flor elementinin Hidrojen elementi ile etkileşimi

### 3. Sonuçlar ve Tartışma

Plastik yüzeyler, bir florin karışımına maruz bırakılarak florlanır. Yüksek reaktivitesine bağlı olarak flor, malzeme yüzeyindeki hidrojen atomlarını kısmen değiştirir. Florlama, kimyasal bir reaksiyondur, kaplama değildir. Buna göre, flor atomları substratın moleküler yapısına nüfuz eder. İşlem, temel malzemenin özelliklerini etkilemez ve boyutları değişmeden kalır.

Mürekkep ve polimerik malzemelerin çokça kullanıldığı ambalaj sektöründe geçerli olan adezyon teorisinin temeli, yüzey enerjisi veya adzorpsiyon teorisidir. Bu teorinin temelini yüzey enerjileri ve kendiliğinden yayılma oluşturur. Bu teorinin anlaşılabilmesi için "yüzey temas açısı"nın anlamının açıklanması gerekir.[6]



Şekil.2 Temas açısı ölçümü

Şeklende görüleceği üzere katı-sıvı ile sıvı-gaz arasındaki açı derecesinin arttırılması amaçlanmış ve başarılı olunmuştur. Florsuz uygulamanın yüzey enerjisi 38 dyn/cm den düşük olduğu,Florlu uygulamanın yüzey enerjisi ise 42 dyn/cm olarak tespit edilmiş olup Kabul edilebilecek değerler arasında yer almaktadır.

e International Conference of Materials and Engineering Technology

Literatür araştırmalarına göre COF değerinin düşmesi beklenirken aksi şekilde arttığı gözlemlenmiştir.[7]

Tablo I. COF analiz sonucu				
Materyal	COF değeri			
Florsuz yüzey/Florsuz yüzey	0,13 N			
Florlu yüzey/ Florlu yüzey	0,42 N			

50 mikron kalınlığındaki aynı LDPE film numunesine Flor tedavisi uygulayarak ve uygulanmamış olarak MOCON cihazında OTR testi yapılmış olup büyük farklılıklar gözlemlenmemiştir.

Standart Numun		Numune	OTR (cc/m <sup>2</sup> -gün)
	ASTM D 3985	Florlu LDPE film	2348,5
		Florsuz LDPE film	2197,8

Florlu LDPE ile Florsuz LDPE arasındaki yüzey farklılığı SEM analizi yapılarak analiz edilmiş olup florlu yüzeyin daha düzgün bir yapıya sahip olduğu görülmüştür.



Şekil.3 SEM görüntülerinin karşılaştırılması

Flor uygulanmış LDPE film yüzeyinde oluşan CF, CF<sub>2</sub> ve CF<sub>3</sub> bağlarının tespiti amacı ile EDX maping işlemi yapılmış olup Flor elementine rastlanılmıştır.

34



Şekil.4 SEM EDX maping yapılarak Flor elementinin tespiti

Literatüre göre 1350-850 cm-1 bölgeleri arasında CHF, CF<sub>2</sub> ve CF<sub>3</sub> bağlarının oluştuğu tespit edilmiştir.[8]



Şekil.5 FT-IR Analizi yapılarak Flor bağlarının tespiti sağlanmıştır.

Mukavemet testleri ZWİCK cihazında 300 m/dk hızında yapılmıştır. Isıl Yapışma değerleri HOT-TACK cihazında yapılmıştır. Sonuçlar incelendiğinde mukavemet değerlerinde değişiklik görülmemiş olup ısıl yapışma değerinin yükseldiği tespit edilmiştir.

Standart		Florlu LDPE	Florsuz LDPE
ASTM D 882	Kopma (N)	25,6	24,1
	Uzama (%)	1016	1036
	Isıl yapışma (°C)	147	126

Tablo 2.	Mukavemet ve isil	vapısma analiz sonucu
1 abit 2.	With a chief ve isin	yapışına ananz sonucu

Flor gazı ile LDPE film yüzeyinin fonksiyonelleştirilmesi başarılmıştır. Literatürde belirtilenin aksine COF değerinin arttığı ve oksijen bariyeri kazandırmadığı gözlemlenmiştir. Fakat yüzey enerjisini arttırdığı ve bu sayede sanayide kullanılan Korona ve plazma teknolojilerine alternatif olabileceği tespit edilmiştir.



**Teşekkür:** Mali ve Teknik desteklerinden dolayı Lidersan Sağlık ve Gıda Ürünleri A.Ş. ' ye teşekkür ederim.

### Kaynakça:

- 1. R. J. Lagow, J. L. Margrave. Prog. Inorg. Chem. 26, 162 (1979).
- 2. Z. Hruska. International Conference "Fluorine in Coatings-IV", Brussels, Belgium, 5–7 March 2001, Conference paper no. 34.
- 3. A. P. Kharitonov. Direct Fluorination of Polymers, Nova Science Publishers (2008).
- 4. A. P. Kharitonov, L. N. Kharitonova, R. Taege, G. Ferrier, E. Durand, A. Tressaud. L'Actualite Chimique Nos. 301–302, 130–134 (2006).
- 5. P. A. B. Carstens, S. A. Marais, C. J. Thompson. J. Fluorine Chem. 104, 97 (2000).
- 6. Şekercioğlu, T., Kaner, S. 2014. "Plastiklerin Yapıştırılmasında Yüzey Hazırlama Yöntemlerinin İncelenmesi," Mühendis ve Makina, cilt 55, sayı 648, s. 37-43.
- 7. J. Peyroux et al. / Surface & Coatings Technology 292 (2016) 144-154
- 8. J. Peyroux et al. / European Polymer Journal 66 (2015) 18-32



# DETERMINATION OF WEIBULL COEFFICIENTS FOR HATAY REGION BY USING THE MOVING LEAST SQUARES APPROACH

## Yusuf Alper KAPLAN<sup>1,\*</sup>Ayşe Gül KAPLAN<sup>2</sup>

<sup>1</sup>Department of Energy Engineering, Osmaniye Korkut Ata University, Osmaniye, Turkey <sup>2</sup> Department of Mathematics, Osmaniye Korkut Ata University, Osmaniye, Turkey

### ABSTRACT

Today, the continuous increase in energy needs increases our need for new energy sources. In particular, renewable energy sources have become a very important energy heading for developed and developing countries. Wind energy is one of the most important renewable energy sources, but the constant variation of wind speed creates some question marks in this regard. In this study, wind energy potential of Hatay region was evaluated. Using the wind data obtained from the meteorology general directorate, the potential of the selected region in terms of wind energy was evaluated with the moving least squares method. The most important reason for the selection of Hatay region is the high wind energy investments made in the region due to its wind potential. The coefficients of the Weibull distribution function will be calculated using the moving least squares method. By means of these coefficients, the average wind speed of the region will be estimated so that the average wind power of the selected region will be determined. Different statistical error analysis methods will be used to evaluate the performance of this method. The results will be compared with the actual wind speed data.

Keywords: Weibull Distribution, Graphical Method, Moving Least Squares Method, Statistical Test

### **1** Introduction

Nowadays, the need of energy has been increasing day by day with the population growth and the advancements of technology [1]. Hence, the search of new energy resources becomes very important subject for the entire world. Energy is the most important factor which affects economic structure of a country. As is known, energy is seen as not only the internal dynamics of the countries but also a strategic case which affects International Relations, including political as well as military conflicts. Energy is also primary element of economic and social development in the world [2]. Wind energy has been used in irrigation, wheat-grinding, vessels and many other fields because it is an environment-friendly future energy resource. Furthermore, wind energy is used for meeting the energy requirement that will be the most important problem of the future world. They are also powerful political player, thanks to the economic power. Currently, wind energy is seen as a positive alternative to fossil fuels and also a way to assist the expansion of local economies in future. The world will use renewable energy instead of using fossil fuels in order to meet the demands of the world's energy [3,4]. This study aims to determine the wind energy potential in Osmaniye. The five



Kantar and Usta [5] analyzed the use of the minimum cross entropy principle in the estimation of wind speed distribution and wind power density functions. Moreover, they compared the Weibull pdf (probability density function) with the MinxEnt (minimum cross-entropy principle) pdfs. Akdag and Dinler [6] reviewed different methods, i.e. the graphical, maximum likelihood, moment methods and energy pattern method. Bilgili and Sahin [7] investigated wind energy density in the southern and southwestern region of Turkey by using the Weibull and Rayleigh probability density functions. Rocha et al. [8] deal with the evaluation and comparison of different numerical methods for the assessment of efficiency in determining the parameters for the Weibull distribution function, using wind speed data collected in Camocim and Paracuru cities. Freitas et al. [9] introduced a new approach for the analyzing of numerical methods used in calculating the Weibull distribution parameters for the prediction of wind energy source. Chang [10] reviewed the six kinds of numerical methods commonly used for estimating Weibull parameters. Bilir et al. [11] collected wind data for a one year period between June 2012 and June 2013. Wind speed data, collected for two different heights (20 m and 30 m) from a measurement station installed in Atılım University campus area (Ankara, Turkey), were recorded using a data logger as one minute average values. They determined shape (k) and scale (c) parameter of Weibull using five different methods.

## 2 The Wind Characteristic of Hatay Region

Hatay is located in the south of our country, the location of it is shown in Fig. 1. It is surrounded by the Mediterranean from the west, Syria from the south and east, Adana from the northwest, Osmaniye from the north and Gaziantep from the northeast. This study aims to determine the wind energy potential in Hatay. We have collected the five years data of the wind speed measured at 10-meter height from the General Directorate of State Meteorology. Based on the hourly-measured wind speed data, the region's wind energy potential has been statistically analyzed by using Weibull Distribution Functions.



Figure 1. The map of Hatay

Fig. 2 shows the density of wind directions according to wind frequency. The dominant wind direction of Hatay region is found by using a five-year wind data. The summers are hot and dry and the winters are mild and rainy. In Hatay, where the average annual temperature varies between 15.1 - 20  $^{\circ}$  C, the average monthly temperature reaches the highest in summer and lowest in winter.



Figure 2: The variation of the wind directions.

39

## 3. Statistical Analysis

There are many distribution functions for determining the wind speed distribution. Two-parameter Weibull and Rayleigh distribution functions are the most popular of all. The Rayleigh distribution is less flexible than the Weibull as it is one-parameter. However, the calculation of parameters is easier in the Rayleigh distribution.

The International Conference of Materials and Engineering

## **3.1 Weibull Distribution Function**

The Weibull Distribution Function provides a close approximation to the probability laws of many natural phenomena. This function has been used to represent wind speed distributions for application in wind turbines studies for a long time. For more than half a century the Weibull Distribution Function has attracted the attention of statisticians working on theory and methods as well as various fields of statistics [12]. The wind data analysis of a region is prepared by the prediction of the region's potential performance through the pre-measured values. Hourly wind speed and wind direction details are observed in a place and statistical results are calculated for modelling the frequency and probability of the obtained results [7, 13]. First, as is seen in Table 1, the periodical frequency (the blowing number) and probability of wind speed are determined. Wind speeds are grouped periodically in the second column of Table 1. The third column shows average wind speed for each speed ratio. The blowing number or frequency of each speed ratio is given in the fourth column.

$$p(v_i) = \frac{f_i}{\sum_{i=1}^{N} f_i}$$
(1)

Here,  $f_i$  is frequency of occurrence of each speed class and N is number of hours in the period of time considered.  $P(v_i)$  is the cumulated probability density which is illustrated in the sixth column of the Table 1.

The two-parameter Weibull distribution function is described for wind speed as follow Eq. (2) and as a cumulative distribution function given by Eq. (3) [14, 15]:

Actual probability density function and cumulative probability distributions derived from the long-term wind speed data of Hatay depicted in Fig. 3.

Technology



Figure 3: The wind speed probability density and cumulative distribution data of Hatay

The general expression of the two-parameter Weibull is given by,

$$p(v) = \left(\frac{k}{c}\right) \left(\frac{v}{c}\right)^{k-1} \exp\left[-\left(\frac{v}{c}\right)^k\right]$$
(2)

The cumulative function of wind speed can be attained by computing the integral of the probability dencity function is given by,

$$P(v) = 1 - \exp\left[-\left(\frac{v}{c}\right)^k\right]$$
(3)

Where p(v) is the observed probability density function, P(v) is the cumulative probability density function , shape (k) and scale (c) parameter of Weibull distribution function [15, 16].

The average wind speed 'Vm' and wind power density ' $P_W$ ' of Weibull Distribution Function can be estimated by the following equations:

$$Vm = c\Gamma\left(1 + \frac{1}{k}\right) \tag{4}$$
$$P_{w} = \frac{1}{2}\rho c^{3}\Gamma\left(1 + \frac{3}{k}\right) \tag{5}$$

Here,  $\Gamma$  shows the gamma function.

$$\Gamma(x) = \int_{0}^{\infty} e^{-u} u^{x-1} du \quad \text{ve} \quad \Gamma(1+x) = x \,\Gamma(x) \tag{6}$$

### **3.2 Moving Least Squares Approximation**

In this paper, the moving least squares approximation presented. The method is primarily proposed to solve the curve fitting problem to the interpolation conditions. The method was extended to solve the surface fitting problem. In this study, the MLSA is used for curve fitting to the interpolation conditions. It is seen that the proposed method gives effective results [17, 18]

The International Conference of Materials and Engineering Technology

In the MLSA, the trial function can be written as [19]

$$u^{h}(x) = \sum_{i=0}^{m} p_{i}(x) a_{i}(x) = \boldsymbol{p}^{T}(x) \, \boldsymbol{a}(x)$$
(7)

where  $p_i(x)$  is a monomial basis of order m,  $a_i(x)$  is the unknown coefficient of basis. The coefficient vector a(x) is determined by minimizing a weighted discrete  $L_2$  norm. Therefore, the quadratic form is defined as

$$J(x) = \sum_{i=1}^{N} w(x - x_i) [u^h(x) - u(x_i)]^2$$

$$J(x) = \sum_{i=1}^{N} w(x - x_i) \left[ \sum_{j=0}^{m} p_j(x) a_j(x) - u(x_i) \right]^2$$
(8)
(9)

where  $x_i$  is nodes of spatial coordinate x,  $w(x - x_i)$  is the weight function and  $w(x - x_i) > 0$  for all x in the support of  $w(x - x_i)$ . To find the value of vector a(x) by minimizing the J we obtain

$$\frac{dJ}{da} = A(x)a(x) - B(x)u = 0 \tag{10}$$

So this can be written in the equation system

$$A(x)a(x) = B(x)u,$$
(11)

Where the matrices A(x), B(x), u, W(x) and p(x) are defined as follow:

$$A(x) = \boldsymbol{p}^{T}(x)W(x)\boldsymbol{p}(x), \qquad (12)$$

$$\boldsymbol{B}(\boldsymbol{x}) = \boldsymbol{p}^{T}(\boldsymbol{x})W(\boldsymbol{x}), \tag{13}$$

$$\boldsymbol{u}^{T} = (u_{1}, u_{2}, \dots, u_{N}), \tag{14}$$



Therefore, substituting a(x) into Eq. (4) approximation function of u(x) is obtained as

$$u(x) \approx u^h(x) = \boldsymbol{p}^T(x)A^{-1}(x)B(x)u \tag{15}$$

There are different weight functions in the literatüre. However, Gaussian weight function with compact supported is chosen in our algorithms. Gaussian weight function is defined as follows:

$$w(x - x_i) = \begin{cases} \frac{e^{-(d_i/c_i)^2} - e^{-(r_i/c_i)^2}}{1 - e^{-(r_i/c_i)^2}}, & 0 \le d_i \le r_i \\ 0, & d_i > r_i \end{cases}$$
(16)

where  $c_i$  is the shape parameter and  $d_i = |x - x_i|$  is the distance between nodes x and  $x_i$ . The support size  $r_i$  for weight functions  $w(x - x_i)$  determines the support of node  $x_i$ .

### 4. Results and Discussions

In this study, two distribution functions are compared that it was compatible with actual data. A linear equation was obtained by using the MLSA. This graph is shown in Fig.4.



Figure 4: The linear regression results of MLSA

43



The long term wind data must be measured in order to determine the wind potential of the selected area. The shape and scale parameters are separately calculated for each month. The MLSA is used to determine the parameters of distribution functions, the calculated parameters are given in Table 1.

	MLSA			
Months	k	с		
January	1,3811	0,8839		
February	1,2745	0,8651		
March	1,5247	1,0536		
April	1,6637	1,2414		
May	1,6666	1,3802		
June	1,9817	1,7305		
July	2,09	1,9749		
August	2,0874	1,7459		
September	1,5276	1,2315		
October	1,4641	0,8788		
November	1,3209	0,7706		
December	1,495	0,9008		
Yearly	1,566	1,2967		

## Table 1. Weibull parameters for MLSA

### **4.1 Statistical Error Analysis**

In this study, two different statistical error tests were performed to show the accuracy and suitability of the proposed method. The used statistical indicators are: The root mean square error (RMSE) and Mean percentage error (MPE) [2].

• The root mean square error (RMSE)

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}(y_i - x_i)^2\right]^{\frac{1}{2}}$$
(17)

• Mean percentage error (MPE)

$$MPE = \frac{1}{N} \sum_{i=1}^{N} \left( \frac{x_i - y_i}{y_i} \right) * 100\%$$
(18)

Actual Values		MLSA Values		Statistical Error Tests	
Vm	Pm	Vw	Pw	RMSE	MPE
1.5244	2.1697	1.1650	2.4745	0.07899	1.12618

Table 2. Comparison of Real and Estimated Values

Average wind speed and average wind power values were calculated with real data and estimated by weibull distribution function. These values were compared in Table 2 and the performance of the method used was evaluated in two separate statistical error tests.

The International Conference of Materials and Engineering Technology

### **5.** Conclusions

In this study, especially Hatay region which is rich in wind energy potential has been selected. The wind characteristics of the region were examined and the average wind speed and average wind energy potential were determined. The actual wind speed and the actual wind power values were compared with the values obtained using the Weibull distribution function. The parameters of the Weibull distribution function were determined by MLSA method and the average wind energy power of the region was estimated by these parameters. The concordance of the results with the real data and the performance of the method were examined with different statistical error analysis tests. According to the results, the method used was successful. In future studies, the scope of this paper can be expanded and detailed with comparing other numerical methods to determine wind energy potential of this region.

### 6. References

- 1. Kaplan Y.A., "Overview Of Wind Energy In The World And Assessment of Current Wind Energy Policies in Turkey", Renewable and Sustainable Energy Reviews, **43** C, pp. 562-568, (2015).
- 2. [Kaplan, Y. A. "Determination of the best Weibull methods for wind power assessment in the southern region of Turkey" IET Renewable Power Generation, **11**, pp. 175-182, (2017).
- 3. Çapika M, Yılmaz A.O., Çavusoglu I., "Present situation and potential role of renewable energy in Turkey", Renewable Energy; **46**, pp. 1-13, (2012).
- 4. Gabbasa M, Sopian K, Yaakob Z, Zonooz M, Fudholi A, Asim N., "Review of the energy supply status for sustainable development in the Organization of Islamic Conference", Renewable and Sustainable Energy Reviews, **28**, pp. 18–28, (2013).
- 5. Kantar Y.M., Usta I., "Analysis of wind speed distributions: wind distribution function derived from minimum cross entropy principles as better alternative to Weibull function", Energy Convers Manage, **49**, pp. 962–973, (2008).
- 6. Akdağ S.A., Dinler A., "A new method to estimate Weibull parameters for wind energy applications", Energy Convers Manag, **50**, pp. 1761-1766, (2009).
- Bilgili M., Şahin B., "The finding of weibull parameters at the determination of Wind Power density", New and Renewable Energy / Energy Management Symposium, Kayseri, 229-234, (2005).
- 8. Rocha P. A. C. R., Sousa R. C. D., Andrade C. F.D, Silva M. E. V. D., "Comparison of seven numerical methods for determining Weibull parameters for wind energy generation in the northeast region of Brazil", Applied Energy, **89**, pp. 395–400, (2012).
- 9. Freitas de Andrade C., Maia Neto H. F., Costa Rocha P. A., Vieira da Silva M. E., "An efficiency comparison of numerical methods for determining Weibull parameters for wind energy applications: A new approach applied to the northeast region of Brazil", Energy Convers Manage, **86**, pp. 801–808, (2014).
- 10. Chang T.P., "Performance comparison of six numerical methods in estimating Weibull parameters for wind energy application", Appl Energy, **88**, pp. 272–282, (2011).



- Azad A.K., Rasul M.G., Alam M.M., Uddin S.M.A., Mondal S.K., "Analysis of Wind Energy Conversion System Using Weibull Distribution", Procedia Engineering., 90, pp. 725–732, (2014).
- 13. Kim J.S., Yum B.J., "Selection Between Weibull and Lognormal Distributions: A Comparative Simulation Study", Computational Statistics&Data Analysis, **53**, pp. 477-485, (2008).
- 14. Ahmet Shata S.A., Hanitsch R. "Evaluation of wind energy potential and electricity generation on the coast of Mediterranean Sea in Egypt", Renew Energy, **31**, pp. 1183–1202, (2006).
- 15. Kose R., Arif M.O., Erbas O., Tugcu A., "The analysis of wind data and wind energy potential in Kutahya, Turkey", Renewable and Sustainable Energy Reviews, **8**, pp. 277–288, (2004).
- 16. Yıldırım U., Gazibey Y., Güngör A., "Wind Energy Potential of Niğde", Journal of Niğde University, **1**, pp. 37-47, (2012).
- 17. Zeng Q.H., Lu D.T., "Curve and Surface Fitting Based on Moving Least-Squares Methods", Journal of Engineering Graphics, pp. 84–89, (2004).
- 18. Zuo C.W., Nie Y.F., Zhao M.L., "The Selection About the Radius of Influence in MLS", Chinese Journal of Engineering Mathematics, pp. 833–838, (2005).
- 19. Lancaster P., Salkauskas K., "Surfaces generated by moving least squares methods", Mathematics of Computation, **37**, pp. 141–158, (1981).



## HALİDE KÖKLÜ<sup>\*1</sup>, OKAN ÖZER<sup>2</sup>

<sup>1,2</sup> University of Gaziantep, Engineering Faculty, Engineering of Physics, Gaziantep, TURKEY.

#### Abstract

Critical thicknesses for one-speed neutrons are calculated for triplet anisotropic scattering in plane geometry neutron transport equation using Chebyshev polynomials of first type approximation  $(T_N)$  method. Triplet anisotropic scattering is the fourth term of the scattering function. The neutron flux moments in the neutron transport equation comprises the eigenfunction of the neutron flux. By solving the eigenfunctions, the eigenvalues are obtained from Chebyshev polynomial solution. The resultant neutron flux equation composes of the eigenfunction, Chebyshev polynomial term and the number of secondary neutrons "c". The solution of the eigenvalues gives imaginary root for c is smaller than one. So in this study we study with bigger than one c values. The system critic size is solved with the Mark boundary condition. The critical size is calculated for one more scattering types. So the relation is obviously observed in the tables. It is shown that our results are in agreement with the existing literature.

Keyword: Critical Thickness, Slab Reactor, Neutron Transport Theory

#### 1. Introduction

The neutron transport equation is the general form of the describing the behavior of the neutrons in a reactor core. The equation includes position, velocity and time variables with seven unknown parameter. For this reason some approximations are assumed and the steady-state neutron transport equation for one speed plane system can be written as [1],

$$\mu \frac{\partial \psi(x,\mu)}{\partial x} + \psi(x,\mu) = \frac{c}{2} \int_{-1}^{1} f(\mu,\mu') \psi(x,\mu') d\mu'$$
(1)

where  $\mu'$  is the direction of the scattering,  $f(\mu, \mu')$  is the scattering function and defines the scattering probability of neutrons,  $\psi(x, \mu)$  is the number of the neutrons at position x- and direction  $\mu$  with distance measured in units of mean free path (mfp), c is the number of secondary neutrons per collision related with the material cross sections by the equation  $c\sigma_t = \upsilon \sigma_f + \sigma_s$ . Here  $\sigma_f$  is the fission cross section and  $\sigma_s$  is the scattering cross section,  $\upsilon$  is the number of neutrons per fission. The distance measured for neutrons is in units of total mean free path, "mfp". The scattering function in Eq. (1) is expanded with the Legendre polynomials [2] as

$$f(\mu,\mu') = \sum_{n=0}^{N} (2n+1) f_n P_n(\mu) P_n(\mu')$$
(2)

where  $P_n(\mu)$  and  $P_n(\mu')$  are Legendre polynomials and  $f_n$  is the scattering coefficient.

47

The scattering function defines the probability of scattering then the sum of these scatterings must equal to one. Therefore the interval values of  $f_n$  must be determined for every scattering situation. The reactor core critical thickness calculations are very complicated for real media and very hard to solve in neutron transport equation. By making this calculation for triplet anisotropic scattering, we have taken into account more scattering probabilities.

e International Conference

### 2. P<sub>N</sub> Solution of the Neutron Transport Equation for Triplet Anisotropic Scattering

The series expansion of the scattering function given in Eq. (2) for triplet anisotropic scattering can be written as,

$$f(\mu,\mu') = f_0 P_0(\mu) P_0(\mu') + 3f_1 P_1(\mu) P_1(\mu') + 5f_2 P_2(\mu) P_2(\mu') + 7f_3 P_3(\mu) P_3(\mu')$$
(3)

Legendre moments of the flux is defined as [3]

$$\phi_n(x) = \int_{-1}^{1} P_n(\mu')\psi(x,\mu')d\mu'$$
(4)

Technology

If the scattering function in Eq. (3) is defined in Eq. (1) with the definition of the Legendre moments,

$$\mu \frac{\partial \psi(x,\mu)}{\partial x} + \psi(x,\mu) = \frac{c}{2} \Big[ P_0(\mu) f_0 \phi_0(x) + 3P_1(\mu) f_1 \phi_1(x) + 5P_2(\mu) f_2 \phi_2(x) + 7P_3(\mu) f_3 \phi_3(x) \Big]$$
(5)

The angular flux can be expanded in terms of the Legendre polynomials [4]

$$\psi(x,\mu) = \sum_{n=0}^{\infty} \frac{2n+1}{2} \phi_n(x) P_n(\mu)$$
(6)

One can insert the Eq. (6) into Eq. (5) by multiplying the resulting equation with  $P_m(\mu)$  and integrating over  $\mu \in (-1,1)$ . The recursion relation

$$\mu P_{n}(\mu) = \frac{1}{2n+1} \Big[ (n+1) P_{n+1}(\mu) + n P_{n-1}(\mu) \Big]$$
(7)

and the orthogonality of the Legendre polynomials

$$\int_{-1}^{1} P_m(x) P_n(x) dx = \begin{cases} 0 & m \neq n \\ \frac{2}{2n+1} & m = n \end{cases}$$
(8)

are used in this application [5]: After some algebra, one can obtain the moments  $\phi_n(x)$  in general form as

$$(n+1)\frac{d\phi_{n+1}(x)}{dx} + n\frac{d\phi_{n-1}(x)}{dx} + (2n+1)(1 - cf_n\delta_{n0} + cf_n\delta_{n1} + cf_n\delta_{n2} + cf_n\delta_{n3})\phi_n(x) = 0, \qquad n = 0, 1, 2, \dots, N$$

$$(9)$$
The kronecker delta is defined as  $\delta_{nm} = \begin{cases} 1, & n = m \\ 0, & n \neq m \end{cases}$  A well-known ansatz for the solutions of Eq. (9) is employed of the form [6],

$$\phi_n(x) = A_n(v)e^{-\sigma_r x/v} \tag{10}$$

where  $A_n$  is the eigenfunctions corresponding to v eigenvalues. We represent a method and corresponding computer code to get all eigenvalues for tetra anisotropic scattering. Using Eq. (10) in Eq. (9), a system of equations is obtained for the analytic expressions of  $A_n(v)$ 

$$A_{1}(v) = vA_{0}(v)(1 - cf_{0})$$
(11)

$$A_2(v) = \frac{3vA_1(v)(1-cf_1) - A_0(v)}{2}$$
(12)

$$A_3(v) = \frac{5vA_2(v)(1-cf_2) - 2A_1(v)}{3}$$
(13)

$$A_4(v) = \frac{7vA_3(v)(1 - cf_3) - 3A_2(v)}{4}$$
(14)

The general form of Eq. (11-14) may be given by

$$(n+1)A_{n+1}(v) + nA_{n-1}(v) + (2n+1)(1 - cf_n\delta_{n0} + cf_n\delta_{n1} + cf_n\delta_{n2} + cf_n\delta_{n3})vA_n(v) = 0, \qquad n = 0, 1, 2, ..., N$$
(15)

As shown in Eq. (15) the analytical solution of the  $A_n(v)$  gives the eigenvalues by solving  $A_{n+1}(v) = 0$ , for any c. The discrete eigenvalues  $v_k$  and eigenfunctions of the neutron flux are included in the equation of criticality. If c is equal 1, one pair of roots is imaginary ( $\pm \infty i$ ), other pairs are in the range [-1, +1]. If c is between 0 and 1, all roots are imaginary but one pair of them is greater than 1. If c is greater than 1, one pair of roots are pure imaginary, and the other pairs are in the range [-1, +1]. After determining the discrete eigenvalues  $v_k$  the general solution of the flux moments in the Eq. (15) can be written for odd numbers of N

$$\phi_n(x) = \sum_{k=1}^{\frac{N}{2}} \beta_k A_n(v_k) \Big[ e^{\sigma_i x/v_k} + (-1)^n e^{-\sigma_i x/v_k} \Big] \qquad (N+1)/2 < k \le (N+1)$$
(16)

where  $\beta_k$  are the coefficients comes from the linear combinations of the solutions corresponding to each  $v_k$  eigenvalues, and they are determined from the boundary conditions of the system using the parity relation of  $A_n(-v) = (-1)^n A_n(v)$ . Therefore, the general solution to Eq. (1) for the neutron angular flux can be found by replacing the flux moments Eq. (16) into Eq. (6),

$$\psi(x,\mu) = \sum_{n=0}^{\infty} \sum_{k=1}^{\frac{N+1}{2}} \frac{2n+1}{2} \beta_k A_n(v_k) \left[ \left(1 + (-1)^n \right) \cosh(\frac{\sigma_t x}{v_k}) + \left(1 - (-1)^n \right) \sinh(\frac{\sigma_t x}{v_k}) \right] P_n(\mu) \quad (17)$$

# 3. T<sub>N</sub> Solution of the Neutron Transport Equation for Tetra Anisotropic Scattering

The angular flux of the neutron transport equation can be written in terms of first type Chebyshev polynomials like that; [7]

$$\psi(x,\mu) = \frac{\phi_0(x)}{\pi\sqrt{1-\mu^2}} T_0(\mu) + \frac{2}{\pi\sqrt{1-\mu^2}} \sum_{n=1}^N \phi_n(x) T_n(\mu)$$
(18)

he International Conference

laterials and Engineering Technology

Here  $\phi_n(x)$  is called as the flux moment and  $T_n(\mu)$  is the first type Chebyshev polynomial term. The Eq. (18) is substituted into neutron transport equation as shown in Eq.(1)

$$\mu \frac{\partial}{\partial x} \left( \frac{\phi_0(x)}{\pi \sqrt{1 - \mu^2}} T_0(\mu) + \frac{2}{\pi \sqrt{1 - \mu^2}} \sum_{n=1}^N \phi_n(x) T_n(\mu) \right) + \frac{\phi_0(x)}{\pi \sqrt{1 - \mu^2}} T_0(\mu) + \frac{2}{\pi \sqrt{1 - \mu^2}} \sum_{n=1}^N \phi_n(x) T_n(\mu) = \frac{c}{2} \int_{-1}^1 f\left(\mu, \mu'\right) \left( \frac{\phi_0(x)}{\pi \sqrt{1 - \mu^2}} T_0(\mu') + \frac{2}{\pi \sqrt{1 - \mu^2}} \sum_{n=1}^N \phi_n(x) T_n(\mu') \right) d\mu'$$
(19)

Here the scattering function is enlarged up to n=3 for triplet anisotropic scattering such as in Eq. (3). All the variables in Eq. (3) and Eq. (19) is replaced in Eq. (1) to be solved and the resultant form is obtained as

$$\mu \frac{\partial}{\partial x} \left( \frac{\phi_0(x)}{\pi \sqrt{1 - \mu^2}} T_0(\mu) + \frac{2}{\pi \sqrt{1 - \mu^2}} \sum_{n=1}^N \phi_n(x) T_n(\mu) \right) + \frac{\phi_0(x)}{\pi \sqrt{1 - \mu^2}} T_0(\mu) + \frac{2}{\pi \sqrt{1 - \mu^2}} \sum_{n=1}^N \phi_n(x) T_n(\mu)$$

$$= \frac{c}{2} \int_{-1}^1 f_0 P_0(\mu) P_0(\mu') + 3f_1 P_1(\mu) P_1(\mu') + 5f_2 P_2(\mu) P_2(\mu') + 7f_3 P_3(\mu) P_3(\mu')$$

$$\left( \frac{\phi_0(x)}{\pi \sqrt{1 - \mu^2}} T_0(\mu') + \frac{2}{\pi \sqrt{1 - \mu^2}} \sum_{n=1}^N \phi_n(x) T_n(\mu') \right) d\mu'$$

		_	
	$\sim$	$\mathbf{n}$	17
- (			11
۰.	_	U	, ,

Some definite integrals known as

$$\int_{-1}^{1} \frac{T_{0}(\mu')}{\sqrt{(1-\mu'^{2})}} d\mu' = \pi \qquad \qquad \int_{-1}^{1} \frac{\mu' T_{0}(\mu')}{\sqrt{(1-\mu'^{2})}} d\mu' = 0$$

$$\int_{-1}^{1} \frac{T_{n}(\mu')}{\sqrt{(1-\mu'^{2})}} d\mu' = 0 \qquad n \ge 1 \qquad \qquad \int_{-1}^{1} \frac{\mu' T_{n}(\mu')}{\sqrt{(1-\mu'^{2})}} d\mu' = \begin{cases} \frac{\pi}{2}, & n = 1\\ 0, & n > 1 \end{cases}$$

$$(21)$$



$$T_{n+1}(\mu) - 2\mu T_n(\mu) + T_{n-1}(\mu) = 0$$
<sup>(22)</sup>

Technology

and the orthogonally relation is

$$\int_{-1}^{1} T_{m}(\mu) T_{n}(\mu) (1-\mu^{2})^{-1/2} d\mu = \begin{cases} 0, & m \neq n \\ \pi/2, & m = n \neq 0 \\ \pi, & m = n = 0 \end{cases}$$
(23)

The recursion in Eq. (22) and the orthogonally relation in Eq. (23) are used and multiplied with  $\int_{-1}^{1} T_m(\mu) d\mu$ . So the differential equations can be found for varying m values that are related with the  $\phi_n(x)$  flux moments.

$$m = 0, \quad \frac{d\phi_{1}(x)}{dx} + \phi_{0}(x) = cf_{0}\phi_{0}(x)$$

$$m = 1, \quad \frac{d\phi_{2}(x)}{dx} + \frac{d\phi_{0}(x)}{dx} + 2\phi_{1}(x) = 2cf_{1}\phi_{1}(x)$$

$$m = 2, \quad \frac{d\phi_{3}(x)}{dx} + \frac{d\phi_{1}(x)}{dx} + 2\phi_{2}(x) = \frac{2}{3}cf_{2}\phi_{0}(x) - \frac{2}{3}cf_{0}\phi_{0}(x) + 2cf_{2}\phi_{2}(x)$$

$$m = 3, \quad \frac{d\phi_{4}(x)}{dx} + \frac{d\phi_{2}(x)}{dx} + 2\phi_{3}(x) = -\frac{6}{5}cf_{1}\phi_{1}(x) + \frac{6}{5}cf_{3}\phi_{1}(x) + 2cf_{3}\phi_{3}(x)$$
(24)

The solution of the recursion equations gives the result of the flux moments of the differential equation. A general expression is proposed to solve the recursion equations.

$$\phi_n(x) = G_n(v) \exp(x/v) \tag{25}$$

This expression is used into Eq. (24) to obtain  $G_n(v)$  the eigenfunctions of the flux moments and then the eigenvalues v can be found from the solution.

$$G_{0}(v) = 1$$

$$G_{1}(v) = cvf_{0}G_{0}(v) - vG_{0}(v)$$

$$G_{2}(v) = 2cvf_{1}G_{1}(v) - 2vG_{1}(v) - G_{0}(v)$$

$$G_{3}(v) = \frac{2}{3}cvf_{2}G_{0}(v) - \frac{2}{3}cvf_{0}G_{0}(v) + 2vcf_{2}G_{2}(v) - 2vG_{2}(v) - G_{1}(v)$$

$$G_{4}(v) = -\frac{6}{5}cvf_{1}G_{1}(v) + \frac{6}{5}cvf_{3}G_{1}(v) + 2cvf_{3}G_{3}(v) - 2vG_{3}(v) - G_{2}(v)$$
(26)

The eigenvalues can be obtained from  $G_{n+1}(v) = 0$ . From the approximation of T<sub>1</sub>, the eigenvalues can be determined by solving  $G_2(v) = 0$ . The coefficients of the eigenfunctions occur (N+1)x(N+1) square matrices and  $\mathbf{G} = [G_0, G_1, G_2, ..., G_N]^T$  is a column vector.



$$\phi_n(x) = \sum_{k=1}^{\frac{N+1}{2}} \beta_k G_n(v_k) \Big[ \exp(x/v_k) + (-1)^n \exp(-x/v_k) \Big] \qquad n = 1, ..., N$$
(27)

The International Conference of Materials and Engineering

Here the rule that  $G_n(-v_k) = (-1)^n G_n(v_k)$  is used and  $\beta_k$  can be determined from the boundary condition of the system.

$$\psi(x,\mu) = \frac{T_0(\mu)}{\pi\sqrt{1-\mu^2}} \sum_{k=1}^{N+1/2} \beta_k G_0(v_k) \left[ (2) \cosh(\frac{x}{v}) \right] + \frac{2}{\pi\sqrt{1-\mu^2}} \sum_{n=1}^{N} \sum_{k=1}^{N+1/2} \beta_k G_n(v_k) \left[ (1+(-1)^n) \cosh(\frac{x}{v}) + (1-(-1)^n) \sinh(\frac{x}{v}) \right] T_n(\mu_k)$$
(28)

#### 4. Criticality Conditions

The aim in criticality problem is to solve the equation of transport related with the number of secondary neutrons "c". In this study eigenvalues corresponding to the case c > 1 are used. Eq. (17) shows that neutron flux expression obtained by  $P_N$  method. Half slab thickness is obtained by applying Mark boundary condition to the Eq. (17). Mark maintains the continuity of neutron flux along the boundaries encircled in a vacuum. He used the idea of continuity of the angular flux, and for the special values of  $\mu$ . It is showed that the angular flux from the boundary is equal to zero. Here, Mark-type boundary condition is used [8].

$$\psi(a, \mu_m) = 0, \quad 1 \le m \le (N+1)/2$$
 (29)

Here,  $\mu_m$  is the root of the Legendre polynomials that can be found from  $P_{N+1}(\mu_m) = 0$ . Then, the criticality equation can be obtained by using Eq. (18) in the Mark boundary condition given in Eq. (29)

$$\sum_{n=0}^{\infty} \sum_{m,k=1}^{\frac{N+1}{2}} \frac{2n+1}{2} \beta_k A_n(v_k) \left[ \left( 1 + (-1)^n \right) \cosh(\frac{\sigma_t a}{v_k}) + \left( 1 - (-1)^n \right) \sinh(\frac{\sigma_t a}{v_k}) \right] P_n(\mu_m) = 0$$
(30)

Eq. (29) can also be written in a matrix form as  $\left[M_m^k(a)\right]B_k = [0]$ , where  $B_k$  is a vector with elements  $\beta_k$  and  $\left[M_m^k(a)\right]$  is a square matrix with elements  $\left[(N+1)/2\right]^2$ . The determinant of  $\left[M_m^k(a)\right]$  must be equal to zero for the criticality condition for a non-trivial solution of Eq. (30).

By applying same procedure for  $P_N$  method the critical thickness equation can be obtained for  $T_N$  method.

#### 5. Numerical Results

We can calculate the critical thickness by using Eq. (30) for Mark boundary condition. The neutron transport equation with triplet anisotropic scattering has three scattering terms. It means that three different scattering coefficients must have been added to general transport equation. The maximum value of the literature study are used for the linear, quadratic and triplet anisotropic scattering coefficients ( $f_1=0.3$ ,  $f_2=0.2$  and  $f_3=0.142$  respectively) [9,10]. It is seen from the Table 1., the critical thickness results for changing number of secondary neutrons "c" show that the critical thickness decreases with increasing c values, as expected.

Technology

Then the critical thickness values are tabulated with fixed c values for isotropic, linear, quadratic pure quadratic, triplet, and pure triplet anisotropic scattering types. So the changing in critical thickness is observed from the Tables 2,3,4,5 (1.01, 1.1, 1.5, and 2.0 respectively). Legendre polynomial solutions are solved up to thirteenth order, however the convergence is observed up to three digits in the results. In literature, slab critical thickness solutions are done for strongly anisotropic scattering by C. Yıldız [10]. In his study, the solutions have been done up to fifteenth order with  $P_N$  method, and the convergence was obtained up to three digits. ; In Table 6 the critical thickness values are tabulated for  $T_N$  method. In Table 7 four different scattering types are showed for fixed and changing c values with  $T_N$  method.

The International Conference of Materials and Engineering Technology

In literature, the solutions of the critical thickness for neutron transport equation are done in plane geometry for triplet anisotropic scattering with  $F_N$  method by G. Türeci [11] (as shown in Table 8). We compared pure triplet anisotropic critical thickness values of our calculations with the literature study done by G. Türeci [11] in order to see the compatibility of our method. To obtain the critical thickness values as shown in Table 8, the peak order of  $P_{13}$  in our results and the peak order of  $F_3$  in G. Türeci results were used.

С	<b>P</b> <sub>1</sub>	<b>P</b> 3	<b>P</b> 5	<b>P</b> 7	<b>P</b> 9	<b>P</b> <sub>11</sub>	<b>P</b> 13	-
1.01	10.0384	9.82526	9.81031	9.80604	9.80423	9.80329	9.80222	-
1.05	4.07718	3.83286	3.81531	3.81064	3.80868	3.80767	3.80709	
1.10	2.68134	2.43080	2.40904	2.40381	2.40166	2.40057	2.39994	
1.20	1.71227	1.47377	1.44256	1.43586	1.43331	1.43203	1.43130	
1.40	1.05213	0.852709	0.809563	0.798604	0.794817	0.793103	0.792162	
1.60	0.774859	0.607738	0.562009	0.547607	0.542141	0.539718	0.538471	
1.80	0.61747	0.474534	0.430248	0.414186	0.407336	0.404099	0.402421	
2.00	0.51481	0.39021	0.34864	0.33215	0.32447	0.32055	0.31843	_
	<b>Table</b> 2	2. Critical half	f-thickness for	r different so	cattering type	es for $c=1.01$	l	
Scattering	<b>P</b> <sub>1</sub>	<b>P</b> 3	P5	<b>P</b> 7	<b>P</b> 9	<b>P</b> <sub>11</sub>	]	P <sub>13</sub>
types								
Isotropic	8.493	56 8.346	35 8.336	16 8.33	309 8.3	3175 8.	.33104 8	3.33064
Lin.ans.	10.03	84 9.835'	75 9.821	33 9.81	695 9.8	1505 9.	.81404 9	9.81297
Pure quadrat	tic 8.493	56 8.347	50 8.325	48 8.32	258 8.3	2133 8.	.32068 8	3.32032
Quadratic	10.03	84 9.821	95 9.808	46 9.80	9.8	0201 9.	.80109 9	9.80012
Pure triplet	8.493	56 8.348	68 8.337	84 8.33	466 8.3	3329 8.	.33257 8	3.33214
Triplet	10.03	84 9.8252	<b>26 9.810</b>	<b>3</b> 1 <b>9.8</b> 0	604 9.8	0423 9.	.80329 9	9.80222
	Table	<b>3</b> . Critical hal	f-thickness fo	or different s	cattering typ	bes for c=1.1		
Scattering	<b>P</b> <sub>1</sub>	<b>P</b> 3	<b>P</b> 5	<b>P</b> 7	<b>P</b> 9	<b>P</b> <sub>11</sub>		<b>P</b> 13
types								
Isotropic	2.30869	2.13534	2.12100	2.11734	4 2.1158	30 2.11	501 2	2.11454
Lin.ans.	2.68134	2.44941	2.43108	2.42613	3 2.4240	02 2.422	292 2	.42227
Pure quadratic	2.30869	2.11492	2.09910	2.09586	5 2.0938	33 2.093	305 2	.09260
Quadratic	2.68134	2.42299	2.40329	2.39831	1 2.3962	24 2.393	517 2	
Pure triplet	2.30869	2.14065	2.12481	2.12097	7 2.1193	38 2.11	857 2	2.11809
Triplet	2.68134	2.43080	2.40904	2.40381	2.4016	56 2.400	057 2	.39994

Table 1. Critical half-thickness for triplet anisotropic scattering in P<sub>N</sub> method



The International Conference

ing Technology

Scattering	<b>P</b> 1	<b>P</b> 3	<b>P</b> 5	$\mathbf{P}_7$	<b>P</b> 9	<b>P</b> <sub>11</sub>	<b>P</b> <sub>13</sub>
types							
Isotropic	0.78001	0.64949	0.62089	0.61223	0.60904	0.60762	0.60686
Lin.ans.	0.89092	0.71651	0.68261	0.67284	0.66925	0.66759	0.66689
Pure quadratic	0.78001	0.63423	0.59956	0.58880	0.58483	0.58311	0.58222
Quadratic	0.89092	0.69594	0.65485	0.64278	0.63840	0.63646	0.63544
Pure triplet	0.78001	0.65802	0.62654	0.61727	0.61389	0.61240	0.61161
Triplet	0.89092	0.70879	0.66356	0.65065	0.64603	0.64399	0.64292
				1:00		2.0	

**Table 5**. Critical half-thickness for different scattering types for c=2.0

Scattering	<b>P</b> 1	<b>P</b> 3	<b>P</b> 5	<b>P</b> 7	<b>P</b> 9	<b>P</b> <sub>11</sub>	<b>P</b> 13
types							
Isotropic	0.45343	0.36197	0.33477	0.32350	0.31817	0.31545	0.31396
Lin.ans.	0.51481	0.39320	0.36065	0.34775	0.34184	0.33887	0.33728
Pure quadratic	0.45345	0.35219	0.32014	0.30656	0.29993	0.29647	0.29455
Quadratic	0.51481	0.38020	0.34204	0.32651	0.31919	0.31543	0.31338
Pure triplet	0.45345	0.36889	0.33939	0.32745	0.32186	0.31902	0.31748
Triplet	0.51481	0.39021	0.34864	0.33215	0.32447	0.32055	0.31843

**Table 6**. Critical thickness for triplet anisotropic in  $T_N$  method

с	$T_1$	T <sub>3</sub>	$T_5$	$T_7$	T9
1.01	12.2945	9.82545	9.81274	9.80669	9.80455
1.05	4.99350	3.83626	3.81813	3.81133	3.80902
1.1	3.28396	2.43973	2.41233	2.40456	2.40202
1.2	2.09710	1.49057	1.44695	1.43674	1.43371
1.4	1.28859	0.874008	0.816173	0.800052	0.795386
1.6	0.949004	0.628321	0.569675	0.54973	0.543002
1.8	0.756243	0.493401	0.438082	0.41676	0.408494
2.0	0.630507	0.407329	0.356231	0.334941	0.325837

	Bristol	HAMAD BIN KHALIFA UNIVERSITY		ternational Conference of	Materials and Engineering	Technology
	Table 7 Cr	itical thickne	ess for different	t scattering type	es in T <sub>N</sub> metho	ł
с	Scattering types	<b>T</b> 1	<b>T</b> 3	<b>T</b> 5	<b>T</b> 7	Т9
	Isotropic	10.4024	8.34422	8.33808	8.33356	8.33203
1.01	Lin.ans.	12.2945	9.83830	9.82369	9.81770	9.81542
1.01	Quadratic	12.2945	9.82396	9.81017	9.80448	9.80236
	Triplet	12.2945	9.82545	9.81274	9.80669	9.80455
	Isotropic	2.82755	2.13832	2.12361	2.11786	2.11613
1 1	Lin.ans.	3.28396	2.45749	2.43397	2.42694	2.42443
1.1	Quadratic	3.28396	2.43288	2.40625	2.39912	2.39664
	Triplet	3.28396	2.43973	2.41233	2.40456	2.40202
	Isotropic	0.95531	0.66222	0.626001	0.613477	0.609675
15	Lin.ans.	1.09115	0.73503	0.687943	0.674389	0.669907
1.5	Quadratic	1.09115	0.718871	0.661233	0.644734	0.639185
	Triplet	1.09115	0.729982	0.6708487	0.652456	0.646735
	Isotropic	0.55536	0.373162	0.340069	0.32546	0.319265
2.0	Lin.ans.	0.630507	0.408875	0.366441	0.350112	0.342973
2.0	Quadratic	0.630507	0.398921	0.34877	0.329438	0.320599
	Triplet	0.630507	0.407329	0.356231	0.334941	0.325837

Table 8. Critical thickness (2a) results for pure triplet anisotropic scattering with Ref. [11].

с	<b>P</b> <sub>N</sub> Method	T <sub>N</sub> Method	<b>R. G. Türeci [11]</b>
1.1	4.23866	4.239246	4.2430863
1.3	1.891516	1.892303	1.8924836
1.5	1.227636	1.228802	1.2252848
1.7	0.90406	0.905672	0.8986480
2.0	0.643614	0.6457373	0.6322023

#### 6. Conclusion

In this study, we examine the critical half-thickness of the slab by changing scattering types with  $P_N$  and  $T_N$  method. We have been seeking an answer for the question what are the effects of anisotropic scattering on the critical thickness. In previous studies, this effect was investigated for each scattering type individually. In contradistinction, this study presents the results from isotropic to triplet anisotropic scattering calculations that are applied simultaneously. Comprehensive and comparative results of all scattering types provided in this study, offers a good source for further studies and researches.

We have extended the neutron scattering function up to  $f_3$ , called as triplet anisotropic scattering. It is quite difficult to obtain the analytical solutions for this kind of scattering. When the number of terms in scattering function is increased, the equation becomes more complicated. In this study, finding the eigenvalues of the neutron transport equation was the first step. Then they are substituted into neutron flux equation to obtain the critical thickness by using a computer code. The thickness required for the criticality depends on the scattering parameters in the solutions. In our solutions, the Legendre polynomials are used since it provides suitable and rapid direct results. The critical halfthickness of the uniform-medium slab for one-energy group with triplet anisotropic scattering is computed by using the well-known and widely used boundary conditions which is Mark boundary condition. The critical thickness decreases by increasing the number of secondary neutrons c as expected. The deviation between each scattering coefficients is getting smaller by increasing the order of anisotropic scattering. Hence, critical thickness results are converging which means less difference between the values of each next orders scattering results.

The International Conference of Materials and Engineering Technology

# **References:**

- 1. Duderstadt J J and Martin W R, Transport Theory. New York: Wiley, 1979.
- 2. Mika, J., "Neutron transport with anisotropic scattering", Nucl. Sci.and Engineering, **1961**, 11: 415.
- 3. Sahni D C, Dahl E B and Sjöostrand N G, "Real criticality eigenvalues of the one-speed linear transport operator" Transp. Theory Statist. Phys. **1995**, 24 1295-1317
- 4. Lewis EE, Miller WF. Computational methods of neutron transport, Wiley, New York, 1984.
- 5. Case K M and Zweifel P F, Linear Transport Theory, New York: Addison Wesley, 1967
- 6. Davison, B., Neutron transport theory, Oxford University Press, London, 1958.
- Yaşa, F., Anlı F., "Eigenvalue spectrum with Chebyshev polynomial approximation of the transport equation in slab geometry" Journal of Quantitative Spectroscopy & Radiative Transfer, 2004, 97 51–57
- 8. Anlı F., Yaşa F., Güngör S., Öztürk H., "T<sub>N</sub> approximation to neutron transport equation and application to critical slab problem", Journal of Quantitative Spectroscopy & Radiative Transfer, **2005**, 101: 129-134,.
- 9. Rashis, P. A. H., "Investigation of the solution by using P<sub>N</sub> method of transport equation for triplet anisotropic scattering" Ms Thesis, Publication of Kahramanmaraş of Sütçü Imam University, Kahramanmaraş, **2013**, 35-50.
- Yıldız, C., "Variation of the Critical Slab Thickness with the Degree of Strongly Anisotropic Scattering in One -peed Neutron Transport Theory" Ann. Nucl. Energy, **1997**, 25,(8): 529-540.
- 11. Türeci, R. G.," Solving the criticality problem with the reflected boundary condition for the triplet anisotropic scattering with the modified  $F_N$  method" Kerntechnik, **2015**, 80(6): 583-591.

# GRAFİT KATKILI POLİPROPİLEN KOMPOZİTLERİN MEKANİK VE TRİBOLOJİK ÖZELLİKLERİNİN İNCELENMESİ

The International Conference

aterials and Engineering Technology

# SALİH HAKAN YETGİN<sup>1\*</sup>, MURAT ÇOLAK<sup>2</sup>

<sup>1\*</sup> Kütahya Dumlupınar Üniversitesi, Simav Teknoloji Fakültesi, Makine Mühendisliği Bölümü, KÜTAHYA, TÜRKİYE
<sup>2</sup> Bayburt Üniversitesi, Mühendislik Fakültesi, Makine Mühendisliği Bölümü, BAYBURT, TÜRKİYE

# Özet

Bu çalışmada, grafit katkılı Polipropilen (PP) kompozitlerinin mekanik ve tribolojik özellikleri incelenmiştir. Bu amaçla, PP polimerine ağırlıkça %1, 3, 5 ve 10 oranlarında grafit ilave edilmiştir. PP/Grafit granülleri çift vidalı ekstruderde elde edildikten sonra çekme ve aşınma numuneleri enjeksiyon kalıplama tekniği ile elde edilmiştir. Farklı oranlarda grafit katkılı PP kompozitlerin aşınma ve sürtünme davranışları pim-disk sisteminde ve kuru ortam şartları altında 1.0m/s ve 1.5m/s kayma hızı ve 20N ve 40N yük altında gerçekleştirilmiştir. Çalışma sonucunda, PP polimerine ilave edilen grafit minerali ile çekme dayanımı azalırken çekme modülü artmıştır. Grafit katkılı PP kompozitin sürtünme katsayısı ve aşınma oranı uygulanan yük ve kayma hızından etkilenmiştir.

Anahtar kelimeler: Polipropilen, Grafit, Mekanik Özellikler, Aşınma, Sürtünme

#### 1. Giriş

Polimerik malzemeler, yüksek dayanım, iyi termal kararlılık ve korozyon direnci gibi özellikleri nedenleriyle birçok endüstride geniş bir şekilde kullanılmaktadır. Özellikle polipropilen (PP) polimeri, fiziksel ve mekanik özelliklerinin çok iyi bir şekilde dengelenmesi, kolay üretilebilmesi ve diğer malzemelere göre oldukça düşük maliyetli olması nedeniyle en çok kullanılan polimer türlerinden birisidir. Ayrıca, PP polimerinin diğer mühendislik polimerlerine göre düşük yoğunluğa sahip olması ağırlık azalımının gerekli olduğu alanlarda da kullanımını ön plana çıkarmaktadır [1,2]. Ancak, PP polimeri, yüksek kalıpta çekme, düşük rijitlik, düşük darbe dayanımı ve kuru kayma şartları altında yüksek sürtünme katsayısı gibi dezavantajlara da sahiptir. PP polimerinin mekanik ve tribolojik özelliklerini geliştirmenin en basit yöntemi ise cam fiber, karbon fiber ve aramit fiber gibi takviye elemanları ile farklı geometrilere sahip katkı elamanlarının kullanılmasıdır. Fiberlerin yük taşıma kapasiteleri nedeniyle mekanik özellikler gelişirken, fiberler kırılsa dahi yük taşıma kapasitelerini koruyarak aşınma direncini artırabilirler [3-5].

Katı yağlayıcı olarak kullanılan politetrafloretilen (PTFE), grafit ve molibden disülfit (MoS<sub>2</sub>) ise sürtünme katsayısının azaltılması ve aşınma direncinin geliştirilmesi için en çok tercih edilen katkı elamanlarıdır [6,7]. Karbonun üç formundan birisi olan grafit, her bir tabakasındaki hekzogonal birim hücrede düzenlenmiş atomların bulunduğu tabakalı bir yapıya sahiptir. Bu tabakalar, kayma şartları altında, kesme kuvveti uygulandığında kolaylıkla kırılabilen zayıf Van der Waals bağları ile bağlıdır [6, 8]. Grafit katkılı polimer malzemelerin mekanik ve tribolojik özellikleri üzerine birçok çalışma yapılmıştır. Chang ve arkadaşları [9] kısa karbon fiber, grafit plakaları ile mikron-altı TiO<sub>2</sub> ve çinko sülfür (ZnS) katkılı polietereterketon (PEEK) ve polieterimit (PEI) polimerlerinin tribolojik özelliklerini incelemişlerdir. Çalışma sonucunda geleneksel katkılar olan karbon fiber ve grafit plakalarının hem aşınma direncini hem de polimerlerin yük taşıma kapasitelerini artırdıklarını Xintao ve arkadaşları [6] politetrafloretilen (PTFE) ve grafit katkılı belirtmişlerdir. poly(phthalazinone ether sulfone ketone) (PPESK) polimerinin mekanik ve tribolojik özelliklerini incelemişlerdir. Çalışma sonucunda, PPESK polimerine ilave edilen katkılar ile sürtünme katsayısının ve aşınma oranının azaldığı belirtilmiştir. %20'nin üzerindeki katkı oranlarında düşük sürtünme katsayısı ve aşınma oranı elde edildiği belirtilmiştir. Uygulanan yükün artması ile sürtünme



12

Bu çalışmada ise PP polimerine ağırlıkça %1-10 oranlarında ilave edilen grafit katkısının mekanik ve tribolojik özelliklere etkisi incelenmiştir. Üretimlerde ekstrüzyon prosesini takiben enjeksiyon kalıplama yöntemi kullanılmıştır. Mekanik özelliklerin belirlenmesi amacı ile çekme testleri yapılmıştır. Tribolojik özelliklerin belirlenmesi için ise kuru ortam şartları altında pim-disk sistemini kullanarak aşınma testleri gerçeklestirilmiştir. Testlerde, 1.0 ve 1.5m/s kayma hızı ile 30 ve 40N yük uygulanmıştır.

#### 2. Deneysel Çalışmalar

Farklı ağırlık oranlarında grafit katkılı Polipropilen kompozitlerin üretiminde Lyondellbasell firmasından temin edilen 0.9g/cm<sup>3</sup> yoğunluğa ve 1.2g/10dak. ergiyik akış indeksine (MFI) sahip Polipropilen (Hostalen PP H1850) polimeri ile Alfa Aesar firmasından temin edilen 2-15µm partikül boyutuna sahip grafit partikülleri kullanılmıştır. Ağırlıkça %1, 3, 5 ve 10 oranlarında grafit katkılı PP kompozitleri 4mm kalıp çapına sahip AYSA LAB30 çift vidalı ekstruder ile üretilmiştir. Besleme bölgesinden kalıba kadar olan sıcaklık dağılımı 170-210°C olarak belirlenmiştir. Silindirik çubuk formunda ekstrude edilen karşımlar soğuk suda soğutulmuş, sonrasında tekrar granül haline getirilmiştir. Çekme ve tribolojik test numuneleri bir enjeksiyon kalıplama makinesi kullanılarak standartlara uygun olarak üretilmiştir. Enjeksiyon ile kalıplama işleminde besleme hunisi ve kalıp arasındaki sıcaklık dağılımı 170-215°C olarak belirlenmiştir. Kalıp sıcaklığı 30°C olarak sabitlenmiştir. Çekme testleri ASTM D638 standardına uygun olarak Zwick Roell Z-100 makinesinde, oda sıcaklığında ve 10mm/dak. cekme hızında yapılmıştır. Sürtünme katsayısı ve aşınma oranlarının belirlenmesi için tribolojik testler, oda sıcaklığında, kuru ortam şartları altında, 1.0 ve 1.5m/s kayma hızı ile 20 ve 40N yük altında pim-disk sistemi kullanılarak gerçekleştirilmiştir. 6mm çapa ve 40mm uzunluğa sahip silindirik pim numuneleri 1040 çeliğine karşı test edilmiştir. Şekil 1'de pim-disk aşınma cihazı resmi verilmiştir. Tablo 1'de ise çalışmada kullanılan malzemeler, yoğunluk değerleri ve deney şartları verilmiştir. Her bir test öncesi, pim ve disk yüzeyleri alkol ile



aterials and Engineering Technology

$$K_o = \frac{\Delta m}{L * \rho * F} \quad (\text{mm}^3/\text{Nm}) \tag{1}$$



Şekil 1. Pim-disk aşınma cihazı.

Tablo 1.	Çalışmada	kullanılan	malzemeler	ve deney	şartları.

Malzemeler	Yoğunluk (g/cm <sup>3</sup> )	Yük (N)	Kayma hızı (m/s)	Sıcaklık (°C)	Nem (%)
РР	0.90				
PP/1Gr	0.91	20	1.0		
PP/3Gr	0.92	20	1.0	23±2	$40 \pm 5$
PP/5Gr	0.94	40	1.5		
PP/10Gr	0.99				

#### 3. Deney Sonuçları

Şekil 2'de farklı oranlarda grafit katkılı PP polimerinin çekme dayanımı sonuçları verilmiştir. Şekilde görüldüğü gibi PP polimerine ilave edilen grafit katkısı çekme dayanımını azaltmıştır. PP polimerinin 29.52MPa olan çekme dayanımı %1, 3, 5 ve 10 oranlarında grafit ilavesi ile azalarak 21.4, 23.6, 25.1 ve 26.9MPa olarak elde edilmiştir. Bu azalma grafit miktarına bağlı olarak %37, %24, %17 ve %9 oranlarında elde edilmiştir. Benzer bir sonuç Xintao ve arkadaşları [6] tarafından yapılan PTFE ve grafit katkılı PPESK polimerinde de elde edilmiştir. Bunun sebebi, katkılar ile PPESK polimeri arasındaki zayıf arayüzeye ve ayrıca PTFE ve grafit katkılarının %15'in üzerinde kolaylıkla topaklanmasına bağlamışlardır. Mekanik özelliklerdeki azalmanın, grafit kristallerinin zayıf mekanik dayanıma sahip olmasından da kaynaklanabileceği belirtilmiştir. Katkılara fonksiyonelleşme işlemi yapılmadığında, katkı ile polimer arasında zayıf bağlanmanın oluşacağı ve dolayısıyla mekanik özelliklerin azalacağı belirtilmiştir. Çatlakların, polimer matris ile hemen hemen hiç bağlanmayan bu alanlar içerisinde kolaylıkla başlayabileceği ve yayılabileceği, dolayısıyla dayanımın azalacağı belirtilmiştir [17]. PP polimerine ilave edilen grafit miktarının artması ise çekme dayanımını artırmıştır. %1 ile %10 grafit katkısı karşılaştırıldığında bu artış %25.4 oranında elde edilmiştir. Guo-Hua ve arkadaşları [11] genleştirilmiş grafit katkılı polistren (PS) polimerinin mekanik özelliklerini inceledikleri çalışmada artan grafit miktarı ile çekme dayanımının arttığını belirlemişlerdir. Bunun sebebinin ise rijit grafit tabakalarının çekme modülünü artırması ve grafit plakalarının aynı zamanda takviye elemanı gibi davranmasına bağlamışlarıdır.

59



Şekil 2. Grafit miktarına bağlı olarak çekme dayanımı sonuçları.

Sekil 3'te ise grafit miktarına bağlı olarak elastiklik modülü sonucları verilmistir. Sekilde görüldüğü gibi PP polimerine ilave edilen grafit ile elastiklik modülü artmıştır. PP polimerinin 1248MPa olan elastiklik modülü %3 grafit miktarında 1531MPa, %10 grafit miktarında ise 1835MPa olarak elde edilmiştir. %10 oranında grafit ilavesi ile PP polimerinin elastiklik modülü %47 oranında artmıştır. PP polimerine ilave edilen rijit grafik katkısı elastiklik modülünü artırmıştır. Aynı zamanda PP polimerinin kristallenme davranışı elastiklik modülünü etkilemiş olabilir. Benzer sonuçlar Zheng [18] ve Krupa [19] tarafından yapılan çalışmada da elde ediliştir. İnorganik partiküller polimer malzemelere ilave edildiğinde, bu partiküller matriste iskelet görevi görecektir ve matris ile partikül arasında fiziksel çapraz bağlanma nedeniyle zincirlerin moleküler hareketi sınırlanarak kompozit sistemlerin rijitliği artacaktır. PP gibi kristal özelliğe sahip polimerler için inorganik partiküller, heterojen çekirdeklenmeye sebep olarak kristallenme derecesini veya kristal yapıyı değiştirerek kompozitlerin rijitliğinin artmasına katkı sağlayabilir [2]. Şekil 4'te ise grafit miktarına bağlı olarak kopma uzaması sonuçları verilmiştir. Şekilde görüldüğü gibi sünek olan PP polimeri grafit eklenmesi ile gevrek davranış göstermiştir. PP polimerinin %10.65 kopma uzaması değeri farklı oranlarda grafit ilavesi ile ortalama %144 oranında azalarak yaklaşık %4.4 civarında elde edilmiştir. Genel olarak, katkı miktarına bağlı olarak kopma uzamasının azalması beklenilen bir durumdur. Krupa ve arkadaşları [19] kopma uzamasındaki azalmayı kullanılan grafitin düzensiz bir yapıda olmasına, partiküllerin keskin köşelere sahip olmasına ve partiküllerin homojen olarak dağılmamasına bağlamışlardır. Bu geometrik faktörlerin kopma uzamasının azalmasına katkıda bulunacağı belirtilmiştir.



UWE Bristol

Şekil 3. Grafit miktarına bağlı olarak elastiklik modülü sonuçları.



Şekil 4. Grafit miktarına bağlı olarak kopma uzaması sonuçları.



Şekil 5. PP ve kompozitlerinin yüke bağlı olarak sürtünme katsayısı değişimi (Hız: 1.0m/s).

Technology

gineering

and En

C



Şekil 6. PP ve kompozitlerinin kayma hızına bağlı olarak sürtünme katsayısı değişimi (Yük:20N).

Şekil 5 ve 6'da sırasıyla PP ve grafit katkılı PP kompozitlerin farklı kayma hız ve yük altındaki sürtünme katsayısı sonuçları verilmiştir. Şekil 5 incelendiğinde, tüm numuneler için artan yüke bağlı olarak sürtünme katsayısının azaldığı belirlenmiştir. PP polimerinin 0.66 olan sürtünme katsayısı yükün 40N'a artırılması ile %20.5 oranında azalarak 0.55 elde edilmiştir. Benzer şekilde, PP/1Gr, PP/3Gr, PP/5Gr ve PP/10Gr kompozitleri için bu azalma %6.13, %7.90, %11.9 ve %11.7 oranlarında elde edilmiştir. Yükün artması ile sürtünme ısısı nedeniyle temas sıcaklığı genellikle artacaktır. Artan bu arayüzey sıcaklığı polimer matrisi yumuşatarak kayma gerilimini azaltacaktır ve böylelikle sürtünme katsayısı azalacaktır [20]. PP polimerine ilave edilen grafit katkısı da sürtünme katsayısının azalmasında etkili olmuştur. 40N yük altında, PP polimerinin sürtünme katsayısı grafit miktarına bağlı olrak %14, %40, %76 ve %118 oranlarında azalmıştır. Suresha ve arkadaşları [10] artan grafit miktarı ile cam elyaf/epoksi kompozitlerin sürtünme katsayısının azaldığını belirtmişlerdir. Sürtünme katsayısındaki bu azalmanın, yağlayıcılık etkisini artıran, grafit partiküllerin parçalanması ve/veya topaklanmış grafitin dağılımı nedeniyle olduğunu belirtmişlerdir. Katiyar ve arkadaşları da [15] epoksi bazlı polimerin 0.54 olan sürtünme katsayısının %10 grafit ilavesi ile 0.25'e düştüğünü ve bu azalmanın sebebini ise grafitin mükemmel bir katı yağlayıcı olmasına bağlamışlardır. Şekil 6 incelendiğinde ise artan kayma hızına bağlı olarak sürtünme katsayısının arttığı belirlenmiştir. PP polimeri için bu artış %4.6 oranında elde edilirken PP/1Gr, PP/3Gr, PP/5Gr ve PP/10Gr kompozitleri için %7.2, %8.5, %8.9 ve %9.9 oranlarında elde edilmiştir. Sudheer ve arkadaşları [20] grafit gibi katı yağlayıcıların transfer film tabakası kalınlığını değiştirerek kompozit ile disk yüzeyi arasındaki temas alanını etkilediği belirtilmiştir. Artan kayma hızı ile birlikte transfer film tabakasının disk yüzeyinde dağıldığı ve sürtünme katsayısının etkilendiği belirtilmiştir. En yüksek sürtünme katsayısı 1.5m/s kayma hızı ve 20 N yük altında 0.69 değeri ile PP polimerinde elde edilirken en düşük sürtünme katsayısı 1.0m/s kayma hızı ve 40N yük altında 0.25 değeri ile PP/10Gr kompozitinde elde edilmiştir. Uygulanan kayma hızı ve yük aralıklarında sürtünme katsayısını etkileyen en önemli parametrenin uygulanan yük olduğu belirlenmiştir.

Technology



Sekil 7. PP ve kompozitlerinin yüke bağlı olarak spesifik aşınma oranı değişimi (Hız: 1.5m/s).

PP/3Gr

PP/5Gr

PP/10Gr

PP/1Gr

0,0E+00

PP



Şekil 8. PP ve kompozitlerinin kayma hızına bağlı olarak aşınma oranı değişimi (Yük:40N).

Şekil 7 ve 8'de PP ve grafit katkılı PP kompozitlerinin uygulanan yüke ve kayma hızına bağlı olarak spesifik asınma oranları verilmistir. Genel olarak PP polimeri ile PP/1Gr kompoziti için asınma oranı 10<sup>-4</sup> mm<sup>3</sup>/N.m elde edilirken PP/3Gr, PP/5Gr ve PP/10Gr kompozitleri için 10<sup>-5</sup> mm<sup>3</sup>/Nm elde edilmiştir. Grafikler incelendiğinde, artan yüke bağlı olarak aşınma oranı azalırken kayma hızının artması ile aşınma oranı artmıştır. PP polimerine ilave edilen grafit ve artan grafit miktarına bağlı olarak aşınma oranı önemli oranda azalmıştır. Bu azalma, PP/1Gr, PP/3Gr, PP/5Gr ve PP/10Gr kompozitleri için %47, %71, %104 ve %140 oranlarında elde edilmiştir. Benzer bir sonuç Xintao ve arkadaşları [6] tarafından yapılan çalışmada da elde edilmiştir. Artan grafit miktarına bağlı olarak sürtünme katsayısının ve aşınma oranının azaldığı belirtilmiştir. Çalışma sonucunda, grafitin karşı disk yüzeyinde transfer film tabakası oluşturan katı yağlayıcılardan birisi olduğu belirtilmiştir. Artan grafit miktarı ile kayma yüzeyi üzerinde şekillenen grafit miktarının arttığı ve disk yüzeyi ile polimer arasında teması keserek sürtünme katsayısının ve aşınma miktarının azaldığı belirtilmiştir [6]. Goyal and Yadav [16] grafit katkılı PTFE kompozitin tribolojik özelliklerini incelemişlerdir. Çalışma sonucunda, aşınma oranının %5 ve %10 grafit içeriğinde 22 ve 45 kez daha düşük olduğu belirtilmiştir. Bu azalmanın karşı disk yüzeyinde oluşan ince ve sürekli transfer film tabakasının oluşumuna bağlamışlardır. Çalışma sonucunda en düşük aşınma oranı 1.0m/s kayma hızı ve 40N yük altında, 5.81x10<sup>-5</sup> mm<sup>3</sup>/Nm değeri ile PP/10Gr kompozitinde elde edilmiştir. Bilindiği gibi grafit, birbirine zayıf Van der Waals kuvvetleri ile bağlanmış ince paralel düzlemler (grafen) oluşturan



altıgen halkalardan oluşan tabakalı yapıya sahip katı bir yağlayıcıdır. Kayma süresince, sürtünme kuvvetleri grafit partiküllerinin kayma yönünde hizalanmasına sebep olmaktadır. Grafen plakaları arasındaki zayıf bağ kayma yönünde düşük kayma gerilmelerine neden olurken kayma yönüne dik yönde yüksek basma dayanımı sağlamaktadır [10]. Düşük sürtünme katsayısı ve grafitin yüksek yük taşıma kapasitesi grafit katkılı PP kompozitlerin kuru kayma şartları altında aşınma performansının artmasında etkili olmuştur.



Şekil 9. PP ve PP kompozitlerin makro disk resimleri.

Şekil 9'da PP ve grafit katkılı PP kompozitlerin makro disk resimleri verilmiştir. Şekilde görüldüğü gibi tüm numuneler için disk yüzeyinde transfer film tabakasının oluştuğu belirlenmiştir. Oluşan bu transfer film tabakası pim ile disk temasını engellemiştir. Xintao ve arkadaşları [6] tarafından yapılan çalışmada da sürtünme katsayısının ve aşınma oranının kontrol edilmesinde politetrafloretilen (PTFE) ve grafit katkılarının en önemli katı yağlayıcılar olduğu belirtilmiştir.

# 4. Sonuçlar

Farklı oranlarda grafit katkılı PP kompozitlerinin mekanik ve tribolojik özelliklerinin incelendiği çalışmada aşağıdaki sonuçlar elde edilmiştir.

PP polimerine ilave edilen grafit, çekme dayanımını ve kopma uzamasını azaltırken elastiklik modülünü artırmıştır. En yüksek sürtünme katsayısı 1.5m/s kayma hızı ve 20 N yük altında 0.69 değeri ile PP polimerinde elde edilirken en düşük sürtünme katsayısı 1.0m/s kayma hızı ve 40N yük altında 0.25 değeri ile PP/10Gr kompozitinde elde edilmiştir. Uygulanan kayma hızı ve yük aralıklarında sürtünme katsayısını etkileyen en önemli parametrenin uygulanan yük olduğu belirlenmiştir. Genel olarak PP polimeri ile PP/1Gr kompoziti için aşınma oranı 10<sup>-4</sup> mm<sup>3</sup>/N.m elde edilirken PP/3Gr, PP/5Gr ve PP/10Gr kompozitleri için 10<sup>-5</sup> mm<sup>3</sup>/Nm elde edilmiştir. Düşük sürtünme katsayısı ve grafitin yüksek yük taşıma kapasitesi grafit katkılı PP kompozitlerin kuru kayma şartları altında aşınma performansının artmasında etkili olmuştur.

#### Kaynaklar:

1. Kyriaki, K., Hiroyuki, F., Lawrence, T.D. Mechanical properties and morphological characterization of exfoliated graphite–polypropylene nanocomposites. Composites: Part A, **2007**, 38:1675-1682.

The International Conference of Materials and Engineering Technology

- 2. Ji-Zhao, L., Qiang, D., Gary Chi-Pong, T., Chak-Yin, T. Tensile properties of graphene nanoplatelets reinforced polypropylene composites. Composites Part B, 2016, 95:166-171
- Jia, J.H., Chen, J.M., Zhou, H.D., Hu, L.T., Chen, L. Comparative investigation on the wear and transfer behaviors of carbon fiber reinforced polymer composites under dry sliding and water lubrication, Compos. Sci. Technol., 2005, 65(7-8):1139–1147.
- 4. Zhang, X.R., Pei, X.Q., Wang, Q.H. Friction and wear studies of polyimide composites filled with short carbon fibers and graphite and micro SiO2. Mater. Des., **2009**, 30(10):4414-4420.
- Shi, Y.J., Mu, L.W., Lu, X.H., Feng, X. Tribological behavior of carbon nanotube and polytetrafluoroethylene filled polyimide composites under different lubricated conditions, J.Appl.Polym.Sci., 2011, 121(3):1574-1578.
- 6. Xintao, Z., Gongxiong, L., Qifeng, J., Xuebin, F., Xigao, J. On dry sliding friction and wear behavior of PPESK filled with PTFE and graphite. Tribology International, **2008**. 41:195-201.
- 7. Ben Difallah, B., Kharrat, M., Dammaka, M., Monteil, G. Mechanical and tribological response of ABS polymer matrix filled with graphite powder. Materials and Design, **2012**, 34:782-787.
- Yu-Xun, P., Zhong-Zhen, Y., Yu-Chun, O., Guo-Hua, H. A New Process of Fabricating Electrically Conducting Nylon 6/Graphite Nanocomposites via Intercalation Polymerization. Journal of Polymer Science Part B Polymer Physics, 2010, 38(12):1626-1633.
- 9. Chang, L., Zhong, Z., Lin, Y., Klaus, F. Tribological properties of high temperature resistant polymer composites with fine particles. Tribology International, **2007**, 40:1170-1178.
- Suresha, B., Chandramohan, G., Renukappa, N.M., Siddaramaiah, H. Mechanical and Tribological Properties of Glass–Epoxy Composites with and Without Graphite Particulate Filler. Journal of Applied Polymer Science, 2007, 103:2472-2480.
- Guo-Hua, C., Da-Jun, W., Wen-Gui, W., Bin, H., Wen-li, Y. Preparation of polystyrene– graphite conducting nanocomposites via intercalation polymerization. Polymer International, 2001, 50:980-985.
- 12. Difallah, B.B., Kharrat, M., Dammaka, M., Monteil, G. Mechanical and tribological response of ABS polymer matrix filled with graphite powder. Materials and Design, **2012**, 34:782-787.
- 13. Jia, Z., Caizhe, H., Yanhong, Y., Yulin, Y. Effects of nano scale expanded graphite on the wear and frictional behaviors of polyimide-based composites. Wear, **2015**, 338-339:282-287.
- 14. Golchin, A., Klaus, F., Andreas, N., Braham, P. Tribological behavior of carbon-filled PPS composites in water lubricated contacts. Wear, **2015**, 328-329:456-463.
- 15. Katiyar, J.K., Sujeet, K.S., Arvind, K. Friction and wear durability study of epoxy-based polymer (SU-8) composite coatings with talc and graphite as fillers. Wear, **2016**, 362-363:199-208.
- 16. Goyal, R.K., Yadav, M. Study on wear and friction behavior of graphite flake-filled PTFE composites, Journal of Applied Polymer Science, **2013**, 127(4):3186-3191.
- 17. Bryan, D., Khalid, L., Use of exfoliated graphite filler to enhance polymer physical properties. Carbon, **2007**, 45:1727-1734.
- Zheng, W., Xuehong, L., Shing-Chung, W. Electrical and Mechanical Properties of Expanded Graphite-Reinforced High-Density Polyethylene. Journal of Applied Polymer Science, 2004, 91:2781-2788.
- 19. Krupa, I., Novak, I., Chodak, I. Electrically and thermally conductive polyethylene/graphite composites and their mechanical properties. Synthetic Metals, **2004**, 145:245-252.
- Sudheera, M., Hemantha, K., Rajua, K., Thirumaleshwara, B. Enhanced Mechanical and Wear Performance of Epoxy/glass Composites with PTW/Graphite Hybrid Fillers. Procedia Materials Science, 2014, 6:975-987.



# SICAKLIK DEĞİŞİMİNİN POLİÜRETAN ÇARPIŞMA KUTULARININ DARBE PERFORMANSINA ETKİSİNİN DENEYSEL OLARAK İNCELENMESİ

# **MURAT REİS**

Makine Mühendisliği Bölümü, Bursa Uludağ Üniversitesi, Bursa, Türkiye

# Özet

Bu çalışmada, sıcaklık değişiminin poliüretan malzemeden imal edilmiş silindirik bir çarpışma kutusunun darbe performansı üzerine etkisi deneysel olarak incelenmiştir. Özel olarak asansörlerde emniyet elemanı olarak kullanılan poliüretan durdurucuların çalışma sıcaklık aralıkları ölçümlerle tespit edilmiş ve bu sıcaklık aralıkları için düşme testleri yapılmıştır. İçi kısmi olarak boşaltılmış, silindirik formda hazırlanan poliüretan çarpışma kutusu numunesi için düşürme testi sırasında kuvvet, enerji, yer değiştirme ve zaman verileri kaydedilmiş. Sonuçlar enerji emilimi, tepe kuvveti ve maksimum deplasman değerleri açısından karşılaştırılmıştır. Sonuç olarak, poliüretan malzemenin çarpışma performansının belirlenen çalışma sıcaklığı aralığında değişken olduğu, özellikle de 50 °C den sonra bu değişimin hızla arttığı gözlenmiştir. Ayrıca, enerji emme yeteneğinin sıcaklıkla bir miktar arttığı ve yine 50 °C den sonra enerji emiliminin de azaldığı görülmüştür.

Anahtar Kelimeler: poliüretan, çarpışma, kutu, sıcaklık

# 1. Giriş

Ucuzluğu ve üretim kolaylığı sayesinde polimer esaslı malzemelerin kullanımı hızla artmakta ve metalik malzemeler yerlerini hızla polimer esaslı malzemelere bırakmaktadır. Bu avantajlarına rağmen, polimer malzemelerin mekanik özellikleri metalik malzemelere kıyasla sıcaklık değişimlerine karşı çok daha hassastır. Uzun yıllardır otomobillerde ve asansörlerde kullanılan pasif güvenlik elemanları (çarpışma kutuları veya durdurucular) genellikle silindirik ve prizmatik kutular olarak çelik veya alüminyum gibi metal bazlı malzemelerden yapılmaktaydı. Ancak, düşük üretim maliyetleri ve çok karmaşık geometrilerde üretilebilme yetenekleri nedeniyle polimer esaslı çarpışma kutularını e durdurucular son yıllarda hızla yaygınlaşmaktadır. Polimer çarpışma kutularının ana avantajları, çarpışma performansını optimize edebilecek karmaşık geometrilerde üretilebilmeleri ve elastik özellikleri sayesinde, değiştirilme ihtiyacı olmaksızın kullanılabilmeleridir. Özellikle poliüretan bazlı sönümleyiciler birer ideal yay gibi davranmakta ve çarpışma sonrası yük ortadan kalktıktan kısa bir süre sonra kalıcı deformasyona uğramadan eski formuna tamamen geri dönmektedir. Bununla birlikte, bu malzemelerin mekanik özellikleri, gerilme hızına ve sıcaklığına bağlı olarak metal malzemelere kıyasla önemli ölçüde değişebilmektedir.

Polimerlerin sıcaklığa bağlı mekanik özelliklerini belirleme ve iyileştirme çalışmaları oldukça yeni bir araştırma alanıdır [1,2]. Bouvard ve diğ. deneysel olarak farklı gerilmeler, sıcaklıklar ve nominal zorlanma hızları altında iki yarı kristalize polimerin (polipropilen, PP ve kopolimer polipropilen, ko-PP) mekanik davranışlarını incelemiştir [3]. Grala ve diğ. çekme testleriyle -20 ila 120 derecede ısıtılan polipropilenin viskoelastik davranışını gözlemlemişlerdir [4]. Li ve diğ. sıcaklığın polipropilen polimerinin mekanik davranışı üzerindeki etkisini incelemiştir [5]. Belirli polimerlerin mekanik özelliklerini sırasıyla düşük sıcaklık ve yüksek sıcaklık için tek eksenli çekme testleriyle incelemişlerdir. Sonuç olarak, sıcaklık arttıkça polimerlerin elastik modülünün belirli oranlarda azaldığını göstermişlerdir. Polimerlerin mekanik özellikleri bazı katkı maddeleri ile geliştirilebilmektir. Ancak, bu katkı maddeleri kullanılırken bunların insan sağlığı üzerindeki etkileri

de dikkate alınmalıdır. Son yıllarda, termo-plastiklerin zorlanma hızı oranına [6, 7] ve sıcaklığa [8, 9, 10, 11] bağlı mekanik özelliklerini belirlemek için sayısız çalışmalar yapmıştır.

The International Conference of Materials and Engineering Technology

Polimer malzemelerin sıcaklığa bağlı mekanik özelliklerinin incelenmiş olmasına rağmen, polimerlerden imal edilmiş elemanların çarpışma performansları henüz yeteri kadar incelenmemiştir. Zira son yıllara kadar, otomotivdeki tüm pasif güvenlik elemanları, sıcaklığa bağımlı olmayan (-50 ° C ile 100 ° C arası) mekanik özelliklere sahip metal bazlı malzemelerden oluşuyordu. Bu nedenle, performansi sıcaklık carpisma problemlerinde etkileven bir faktör aracın olarak değerlendirilmemekteydi. Oysa günümüzde otomobillerde ve daha pek çok alanda polimer bazlı pasif güvenlik elemanı sayısı hızla artmaktadır. Bununla beraber iklim koşullarına bağlı olarak polimer malzemeler otomotiv ve yapı sektöründe 80 °C dereceyi bulabilen bir sıcaklık aralığında calısabilmektedir. Polimer yapıların yaygın olarak kullanılmasıyla, pasif emniyet elemanlarının mekanik davranışlarının, farklı iklim koşullarındaki kazalarda farklılık göstereceği açıktır.

Hücresel tamponlar olarak ta isimlendirilen poliüretan çarpışma kutuları, uzun sıkıştırma uzunlukları ve yüksek enerji depolama ile karakterize edilen pasif emniyet elamanlarıdır. Hücresel çarpışma kutuları (veya tamponları) yüksek yapısal direnci olan hücresel poliüretan elastomerden üretilirler. Bunların temel avantajları, yük etkisi altında nispeten düşük bir enine genişleme ile sonuçlanan hacimsel sıkıştırılabilirlikleridir. Hücresel tamponlar yağ ve gres gibi alifatik hidrokarbonlara ve Bu nedenle teknik uvgulamalarda karsı dayanıklıdırlar. vaslanmava yaygın olarak kullanılmaktadırlar. Ancak kuvvetli asit ve özütleyicilere karşı dayanıklı değillerdir. Çalışma sıcaklığı -20 °C ila 80 °C arasındadır. Hücresel gövdenin malzemesi özgül ağırlığı 0,35 g/cm<sup>3</sup> (yumuşak poliüretan) ila 0,65 g/cm<sup>3</sup> (sert poliüretan) aralığındadır. Hücresel tamponlarda kullanılan poliüretan, poliol ve izosiyanatların belirli bir oranda ve sıcaklıkta karışımı ile oluşturulan ve ekzotermik bir reaksiyonla şişen bir polimer üründür. İki temel gruba ayrılabilir ki, bunlardan polieter bazlı poliüretan köpükler, güçlü hidroliz direncine sahiptir ve formülasyona eklenecek katkı maddeleri ile uyumludur. Ancak, yangın dirençleri ve mekanik özellikleri düşüktür. Polyester bazlı poliüretan köpükler ise polieter poliol bazlı poliüretan köpüklere alternatif olarak geliştirilen yanma direnci ve termal stabilitesi nispeten yüksek polimerlerdir. Poliüretan bazlı çarpışma kutuları (veya hücresel tamponlar) yüksek enerji depolama özelliklerinin yanında enerji sönümleme özellikleri düşüktür yani, çarpışma sonrasında çarpışma enerjisinin bir kısmını sisteme geri verirler. Hacimsel sıkıştırılabilirlikleri ve uzun sıkıştırma uzunlukları sayesinde, darbe yüklerinin etkisinin zamana yayılmasını sağlayarak sistemi çarpışma kuvvetlerinin tepe değerlerinden korurlar.

Çarpışma dayanıklılığı, araçlarda pasif emniyet elemanlarının çarpışma sırasında yolculara (veya sisteme) zarar vermeyecek güvenli bir alan sağlayarak çarpma enerjisini emme kapasitesini tanımlar [12]. Benzer bir tanımlama asansörler için de yapılabilir zira benzer bir risk asansör kazaları için de geçerlidir. Dahası asansör pasif emniyet elemanlarından beklenen görev ile otomotivde kullanılan pasif emniyet elamanlarından beklene görev birbirinin aynıdır. Ancak otomotivde emniyet elemanları için kütlenin önemli bir tasarım kriteri olmasına karşın asansörlerde kütle artışı problem teşkil etmemektedir. Otomotiv endüstrisinde kullanılan çarpışma kutuları çoğunlukla ince duvarlı metal boş kutulardır [13, 14]. Buna karşılık asansörlerde kullanılan durdurucularda kısmen boşaltılmış dolu profiller kullanılmaktadır. Bunun temel sebebi otomobillerdeki gibi kütle kısıtının asansörlerde bir anlam ifade etmemesidir. Bu çalışmada, poliüretan bazlı bir polimerden üretilen silindirik bir çarpışma kutusu kullanılmış (Şekil 1.b ve c), darbe performansı ve bu performansın sıcaklıkla değişimi deneysel düşme testleri ile incelenmiştir. Düşürme testleri Şekil 1.a da gösterilen, Bursa Uludağ Üniversitesi Makine Mühendisliği Laboratuvarı'nda tasarlanıp üretilen test cihazı kullanılarak gerçekleştirilmiştir (maksimum 500 kN dinamik yük hücresi, maksimum 10 kJ enerji).

67



# 2. Materyal ve Metot

Deneylerde HKS Has Asansör Kauçuk Plastik Metal San. ve Tic. A. Ş. 'den temin edilen poliüretan çarpışma tamponları kullanılmıştır. Numuneler farklı sıcaklıklardaki sıvı içerisinde ısıl denge sağlanıncaya kadar bekletilerek test sıcaklığına ısıtılmıştır. Her bir test numunesinin test sıcaklığında bekletildikleri sıvı ortam içerisinden alınarak test platformuna yerleştirilmeleri ve düşürme testinin tamamlanması yaklaşık 10 saniye zaman almaktadır. Bu süre zarfında numunelerin dış yüzeylerindeki sıcaklık düşüşünün ihmal edilebilecek kadar az olduğu ve test sonuçlarına ciddi bir etkisinin olmadığı değerlendirilmiştir. Her sıcaklık testi üçer kez tekrarlanarak üç testin ortalaması alınmıştır. Düşürme test cihazı çarpma başlığı üzerindeki kuvvet sensörü ile çarpışma kutusu ile çarpan kütle arasında ortaya çıkan kuvveti ve deplasman sensörü ile kutunun ezilme miktarını (deplasman) kaydetmektedir. Bununla birlikte deneyler eş zamanlı olarak hızlı kamera ile kayıt edilmiştir.

The International Conference

Deneylerde kullanılan poliüretan darbe sönümleyicinin boyutları Şekil 2 de verilen teknik resimlerle gösterilmektedir. Buna göre 155 mm boyunda ve 580 gram kütlesindeki ve 0,58 g/cm<sup>3</sup> yoğunluklu poliüretan tampon üzerine 0.45 m yükseklikten 450 kg yük düşürülerek test numunesinden 2 kJ enerjiyi sönümlemesi beklenmektedir. Deney numunesinin oda sıcaklığındaki testlerinde çarpışmadan önceki ve çarpışma anında en yüksek ezilme anındaki fotoğrafları Şekil 1.a ve Şekil 1.b'de verilmiştir.













ials and Engineering Technology

Şekil 2. Poliüretan çarpışma kutusunun boyutları.



# 3. Sonuçlar

Şekil 3 'te poliüretan çarpışma kutusu test örneklerinin sırası ile 20-30-40-50-60 (°C) sıcaklık değerlerindeki düşme testlerinden elde edilen kuvvet-deplasman eğrileri verilmektedir. Eğrilerden sıcaklık artışı ile birlikte kuvvet değerlerinde belirgin bir değişimin oluşmadığı, deplasman değerlerinde ise gözle görülür bir farkın ortaya çıktığı ve özellikle 50 °C den sonra bu farkın oldukça belirginleştiği görülmektedir. Bununla birlikte, özelikle 50°C den sonra tepe kuvvettin de hızla azaldığı fark edilmektedir. Şekil 3'de verilen kuvvet-deplasman eğrilerinin altında kalan alanlar her bir test numunesinin geçici olarak absorbe ettiği enerji değerini vermektedir ve bu enerji değişimleri Şekil 4 'te enerji-deplasman grafiği olarak ayrıca gösterilmektedir. Şekil 4'te verilen sönümlenen enerji-deplasman grafiğine göre sıcaklık arttıkça sönümlenen enerjinin arttığı görülmektedir. Bu artışın temel sebebi kuvvet değerinin aynı seviyelerde kalmasına karşın deplasman miktarındaki artış sayesinde nispeten daha büyük bir enerjinin poliüretan çarpışma kutusu tarafından emilmesidir. Ancak 50 °C'den sonra kuvvet değerindeki belirgin düşüş sönümlenen enerjiyi azalmakta ve 20 °C ile 50 °C sıcaklık değerleri arasında 0,2 kJ lük bir enerji farkı ortaya çıkmaktadır. 20°C'deki elastomer malzeme ile 60°C'deki elastomer malzeme için tepe kuvvet değerleri arasında 20 kN fark vardır. Benzer şekilde 20 °C'deki elastomer malzeme ile 60°C'deki elastomer malzeme arasında 5 mm deplasman farkı görülmektedir. Bu değerlere göre özgül enerji emilimi değeri en yüksek durum 50 °C de ölçülmüştür ve özgül enerji emilim değeri OEE = 2,2 kJ/kg şeklinde elde edilmiştir.

The International



Şekil 3. Farklı sıcaklıklar için çarpışma kuvvetinin deformasyon miktarı ile değişim eğrileri



Şekil 4. Farklı sıcaklıklar için çarpışma kutusu tarafından sönümlenen enerjinin deformasyon miktarı ile değişim eğrileri.

Technology



# 4. Tartışma

Günümüzde üretim kolaylığı ve maliyeti gibi faktörlerin etkisi ile metalik pasif güvenlik elamanlarının yerini hızla polimer esaslı güvenlik elamanları almaktadır. Bu çalışmada taşıt ve asansör kazalarında darbe yüklerinin etkisini zamana yayarak azaltan ve bir miktarını da sönümleyen elastomer (poliüretan) silindirik çarpışma kutularının sıcaklığa bağlı çarpışma performansları düşürme testleri ile değerlendirilmiştir. Düşürme testi deneylerinden her malzeme için kuvvet-zaman, deplasman-zaman, kuvvet-deplasman ve sönümlenen enerji-deplasman grafikleri elde edildi. Deneyler sonucunda sıcaklığın poliüretan çarpışma kutusunun mekanik özelliklerini 20-50°C aralığında nispeten az bir miktar etkilediği gözlenmiştir. Çarpışma anında ortaya çıkan tepe kuvvetin çok değişmediği buna karşın ezilme miktarının sıcaklık ile birlikte belirgin bir şekilde arttığı gözlenmiştir. 50 °C nin üzerindeki sıcaklık değerlerinde ise hem tepe kuvvetin hem de deplasman değerlerinin değiştiği ve buna bağlı olarak ta sönümlenen enerji değerinin hızla düştüğü gözlenmiştir. Sonuç olarak poliüretan bazlı çarpışma kutularının kullanımında sıcaklığın dikkat göz ardı edilmemesi gereken bir faktör olduğu özellikle yüksek güneş ışınımı altında çalışan makine ve yapı elamanlarında kullanımında dikkat edilmesi gereği anlaşılmaktadır.

The International Conference of Materials and Engineering Technology

# Teşekkür

Poliüretan çarpışma kutusu deney numunelerinin temini konusundaki yardımlarından dolayı HKS HAS ASANSÖR KAUÇUK PLASTİK METAL SAN. VE TİC. A.Ş. 'ne teşekkür ederim.

# Kaynaklar:

- 1. Richeton, J. Ahzi, S. Vecchio, K.S., Influence of temperature and strain rate on the mechanical behavior of three amorphous polymers: characterization and modeling of the compressive yield stress. Int. J. Solids Struct., **2006**, 43, 2318–2335.
- 2. Moy, P., Gunnarsson, C.A., Weerasooriya, T., Chen, W., Stress-Strain Response of PMMA as a Function of Strain-Rate and Temperature, Dynamic Behavior of Materials, Conference Proceedings of the Society for Experimental Mechanics, **2011**, 1, 125-133.
- 3. Bouvard, J.L., Denton, B., Freire, L. and Horstemeyer, M.F., Modeling the mechanical behavior and impact properties of polypropylene and copolymer polypropylene, Journal of Polymer Research, **2016**, 23, 4.
- Grala, M., Bartczak, Z., Morphology, R.A., Thermal and mechanical properties of polypropylene/SiO 2, nano composites obtained by reactive blending. J. Polym. Res., 2016, 23, 1–19.
- 5. Li, G., Chen, Y., Ruan, B., A constitutive model of polymer at different temperature. Aust. J. Mech. Eng., **2016**, 52, 67–73.
- Zhou, Y., Mallick, P.K., Effects of temperature and strain rate on the tensile behavior of unfilled and talc-filled polypropylene Part II: constitutive equation. Polym. Eng. Sci., 2002, 42, 2461–2470.
- Dar, U.A., Zhang, W., Xu, Y., Thermal and strain rate sensitive compressive behavior of polycarbonate polymer – experimental and constitutive analysis. J. Polym. Res., 2014, 21, 1– 10.
- 8. Mahieux, C.A., Reifsnider, K.L., Property modeling across transition temperatures in polymers: a robust stiffness-temperature model. Polymer, **2001**, 42, 3281–3291.
- Gibson, A.G., Torres, M.E.O., Browne, T.N.A., High temperature and fire behavior of continuous glass fiber / polypropylene laminates. Compos A Appl. Sci. Manuf., 2010, 41,1219–1231.
- Sardon, H. Irusta, L. Santamaría, P., Thermal and mechanical behaviour of self-curable water borne hybrid polyurethanes functionalized with (3-aminopropyl) triethoxysilane (APTES). J. Polym. Res., 2012, 19, 1–9.



- 11. Cao, K., Wang, Y., Wang, Y., Experimental in vestigation and modeling of the tension behavior of polycarbonate with temperature effects from low to high strain rates. Int.J.Solids Struct., **2014**, 51:2539–2548.
- 12. Rao, C.L., Narayanamurthy, V., Simha, K.R.Y., Applied Impact Mechanics, John Wiley & Sons, West Sussex, United Kingdom, **2016**.
- Nia, A.A., Hamedani, J.H., Comparative analysis of energy absorption and deformations of thin walled tubes with various section geometries, Thin-Walled Structures, 2010, 48, 946– 954.
- 14. Kuznetcov, A., Telichev, I., Wu, C.Q., Effect of thin-walled tube geometry on its crashworthiness performance", 14th International LS-DYNA Users Conference, Detroit, USA. **2016**.



# EXTRACTION OF INTERFACIAL PROPERTIES FOR DIFFERENT TYPES OF FRC WITH AIDS OF MODERN TECHNIQUES

# AMJAD KHABAZ

Hasan Kalyoncu University, Faculty of Engineering, Civil Engineering Department, Gaziantep, TURKEY.

#### Abstract

Due to the challenges of measuring the interfacial properties of Fiber Reinforced Concrete-FRC by direct methods using measurement devices, it is important to find alternative methods such as special micro or macro-experiments in concrete laboratory in addition to theoretical computations. This presentation introduces an alternative method can be used to find the friction factor between the fibers and the concrete in composites of FRC. The principle of this method depends on pull-out test of fibers embedded into concrete matrix; this test might be applied on laboratory samples and computer simulations using different types of fibers such as (steel, glass, carbon, polymer and aramid), where in the creation process of computer simulations; different values of friction coefficient should be applied (as an expected values) versus each case of laboratory samples, therefore stress-strain curves can be obtained and drawn as a result of these pull-out tests, and the value of interfacial-sliding-friction factor can be found through a simple comparison between the sliding part of laboratory stress-dispalcement curve and the computer simulation stress-displacement curves. This technique can be applied to find another interfacial-properties such as poisson's ratio and bond strength.

**Keyword:** Steel Fiber, Glass Fiber, Fiber Reinforced Concrete, Friction, Pull-Out, Sliding Mechanism, Interfacial Property.

#### **1. Introduction**

Fiber Reinforced Concrete - FRC is classified as a composite material, where the concrete occupies the place of material with high compressive strength property and the fiber occupies the place of material with high tensile strength property. Generally, the strength of FRC can be evaluated using the interfacial properties of contacted surfaces between the concrete and the fibers.

The shape of fiber usually affect on bond strength and sliding mechanism of fibers in concrete matrix [1-3]. In case of sophisticated forms of fiber (with hooked ends), the results showed enhancing in bond strength between the fiber and the concrete, even using same concrete mix components [4]. Evolution in bond strength also can be found in case of fibers with corrugates along its length [5]. Different types of fibers with different shapes are usually used in FRC such as Glass Fiber, Steel Fiber, Carbon Fiber and Aramid Fiber [6-8]. Improvements in the design of FRC can be conducted when the properties of the concrete and the used fibers are considered to use by its best values such as Poison's-ratio and Elasticity Modulus [9-14]. In concrete and materials laboratories pull-out tests are usually used to observe the mechanical behavior at the interfacial surfaces between the concrete and the fiber, where bonding and debonding behavior in addition to sliding mechanism are usually shown on the pull-out curves [15-21]. Computer simulations and finite element modeling can be used to compare and validate the laboratory results [22-28].

According to literatures some properties of FRC can be obtained by direct methods or experiments such as compressive strength, tensile strength, splitting strength and flexural strength. But some other properties are not possible to be obtained by same direct methods or measurements, one of these important properties are interfacial properties such as friction coefficient or Poison's-ratio. Therefore, in this paper, an alternative method will be introduced to extract some interfacial

properties at the surfaces between the concrete and the fiber using laboratory experimental results and computer aids including modern techniques.

#### 2. Materials and Methods

Generally, bond strength at the interfacial surfaces along the fiber in concrete composites comes from three sources. First one because of chemical reaction of cement past along the total area of outer surfaces of the fiber. Second one because of fiber shape and connecting joints between the fiber and the concrete matrix, such as hooked ends or corrugates along the length of the fiber. Third source is sliding movement of the fiber inside the concrete matrix when the fiber is exposed to tensile forces try to extract this fiber from the concrete, such as flexural stresses in case of beams. This third source can be considered as sliding-mechanism, which is controlled by the value of friction at the interfacial surfaces between the fiber and the concrete. Therefore, to evaluate the sliding strength against tensile or compressive forces, which might be applied on the fiber in the concrete, it is important to know the value of interfacial properties such as friction factor and Poisson's ratio.

#### 2.1 Sliding-Mechanism governing equations

Tensile or compressive forces might apply on the fiber in concrete members, in this case, the chemical bond resist until breaking, after that, the debonding strength works to resist the sliding movement until become not sufficient to resist the applied forces, then the friction at the interfacial surfaces starts to resist the sliding movement. The speed and the strength of this sliding movement are related to the smoothing at the interface, what means, in other words, the friction governs this movement.

The applied tensile force,  $F_t$ , on each fiber (or compressive force) usually transferred to the interface as shear stresses,  $\tau_s$ , along the embedded length of the fiber inside the concrete,  $l_{emb}$ , (See Figure 2.1.1).



Figure 2.1.1 Applied shear stresses at the interface.

The concrete resist the fiber sliding movement by frictional stresses,  $\tau_{fr}$ , which are distributed along the embedded length at the interface as well but in opposite direction. The resultant of these stresses



can be called as, *S*, which must be under balance with the applied tensile force,  $F_t$ , (See Figure 2.1.2), these frictional forces are produces because of concrete pressure in (*x*) direction on the interface, which can be called as  $\sigma_x$ .



Figure 2.1.2 Resistance stresses at the interface.

In both cases, if we assume the friction factor at the interface between the fiber and the concrete is,  $Fri_{(f.c)}$ , the governing equations will be as the following:

$$\tau_{s} = \frac{F_{t}}{\pi \times d \times l_{emb}}$$
(1)  
$$\tau_{fr} = \sigma_{x} \times Fri_{(f.c)}$$
(2)

To obtain sufficient resistance against sliding movement, the produced frictional stresses,  $\tau_{fr}$ , must be greater than the applied shear stresses,  $\tau_s$ . Therefore, the concrete pressure in (*x*) direction, which is applied on the interface,  $\sigma_x$ , should satisfies the balance equation (3) as the following:

$$\sigma_x \ge \frac{F_t}{Fri_{(f,c)} \times \pi \times d \times l_{emb}}$$
(3)

#### 2.2 Laboratory pull-out test

The experimental program of this research was prepared to conduct pull-out tests, in aiming to monitor the mechanical behavior the fiber in the concrete when applying an extracting tensile force on the fiber, and obtaining stress-displacement curves, which can be used to find the interfacial properties along the fiber such as friction factor or Poisson's ratio.

Samples of straight steel fiber and single glass fiber embedded into concrete matrix are prepared (See Figurse 2.2.1, 2.2.2).

The density of steel fiber is 7850  $kg/m^3$ , the modulus of elasticity is 200000  $N/mm^2$ , the ultimate strength is 1200  $N/mm^2$ , the length is 50 mm, the diameter is 0.8 mm, Poisson's ratio is 0.28.



The elasticity modulus of glass fiber is 70000  $N/mm^2$ , and for the concrete is 30000  $N/mm^2$ . Poisson's ratio of the used glass fiber and concrete is 0.20.

In all cases, the embedded length of the fiber will equal to 50% of its total length.



**Figure 2.2.1** Preparation of samples for embedded steel and glass fibers into concrete.



**Figure 2.2.2** Pull-out experiment of steel and glass fiber samples.

#### 2.3 Finite element modeling

As a modern technique in this research work, finite element modeling are prepared with respect to same dimensions which are used for the fibers and the concrete in the concrete laboratory samples, as mentioned in previous section. These finite element modeling is important to validate the mechanical behavior of fibers in the concrete, and to conduct the aimed comparison between stress-strain curves which are obtained from concrete laboratory samples and these finite element simulations (See Figure 2.3.1).

In all cases, these finite element simulations are prepared with mesh of quadratic finite elements. For concrete matrix, the connection is node to node by full bond and for the fiber body as well. At the interface, a sliding connection was prepared, as contact line to contact line, using different values of friction factor between the fiber and the concrete, with respect to the type of the used fiber. The scope of friction factor in case of steel fiber is 0.05 to 0.2, and in case of glass fiber from 0.1 to 0.3.



Figure 2.3.1 Finite element modeling and meshing.

# 3. Results and Discussion

From the concrete laboratory experiments, the required data of pull-out test is obtained, and using this data a stress-displacement curve can be drawn for each type of fiber (See Figures 3.1, 3.2 and 3.3).

According to the bonding type at the interface between the fiber and the concrete, each curve of stress-displacement laboratory curves can be divided into three part, first one represents the full bond stage, second represents the debonding stage, and third one represents the stage of sliding movement of the fiber inside the concrete. This last part, which is the sliding movement, can be used to find the friction factor at the interface.

From computer simulations and finite element modeling, using same dimensions and properties of all materials, exactly similar to what was used in concrete laboratory samples; it is possible to draw a stress-displacement curve for each type of fibers. The curve of computer simulations should represent only the stage of sliding movement, and then simple comparison between the drawn curves of the lab and the simulations can be done, where the curves of simulations should be prepared for different values of friction factor as mentioned before. For steel fiber the values of friction factor are (0.05, 0.1, 0.15 and 0.2) (See Figure 3.4), and for the single glass fiber (0.1, 0.2 and 0.3). From this comparison, the value of friction factor can be found (See Figures 3.5, 3.6).



**Figure 3.1** Stress-displacement curves of steel fiber embedded into concrete, obtained from laboratory pull-out test.



Figure 3.2 The average stress-displacement curve of steel fiber embedded into concrete, obtained from laboratory pull-out test.

C.





**Figure 3.3** Stress-displacement curve of single glass fiber embedded into concrete, obtained from laboratory pull-out test.



**Figure 3.4** Stress-displacement curve of straight steel fiber embedded into concrete, obtained from computer simulations.

78



**Figure 3.5** Comparison between the laboratory experiments and the computer simulations for stress-displacement curves to find the value of friction factor of straight steel fiber,  $Fri_{(f.c)} = 0.1$ .



**Figure 3.6** Comparison between the laboratory experiments and the computer simulations for stress-displacement curves to find the value of friction factor of single glass fiber,  $Fri_{(f.c)} = 0.2$ .

79

# 4. Conclusions:

This paper introduced a new method and modern technique to find the main properties at the interface of composite materials such as fiber reinforced concrete-FRC. According to this study, the friction factor between the straight steel fiber and the plain concrete was expected to be 0.1, and between the single glass fiber and the concrete was expected to be equal 0.2 as well. This method can be applied to find the value of friction factor in case of carbon fiber, aramid fiber and polymer fiber or any other type of fiber reinforced concrete composites.

The International Conference of Materials and Engineering Technology

The sliding movement of the fiber was monitored by experimental test, which is pull-out test, and the results were validated by the introduced governing equations and the analysis of computer simulations. The introduced technique in this study may give different values of friction factor according to the methods of concrete mix design and preparation, therefore the values of friction factor, which were found in this study, can be used only as a practical example, and to know the main steps of the introduced technique. Therefore, using same technique, other interfacial-properties can be found, such as Poisson's ratio and bond-strength in addition to debonding-strength.

#### **References:**

- Amjad Khabaz. (2017) Analysis of sliding mechanism of straight steel fibers in concrete and determine the effect of friction. Archives of Civil and Mechanical Engineering – Elsevier, Volume 17, Issue 3, May 2017, Pages 599-608. doi: http://dx.doi.org/10.1016/j.acme.2017.01.005
- 2. **Amjad Khabaz** (2017) Theoretical analysis and numerical simulation of development length of straight steel fiber in cementitious materials, Composite Interfaces Taylor & Francis, 24:5, 447-467, DOI: <u>http://dx.doi.org/10.1080/09276440.2016.1230999</u>
- Khabaz, A. (2015) Impact of Fiber Shape on Mechanical Behavior of Steel Fiber in Fiber Reinforced Concrete FRC. World Journal of Engineering and Physical Sciences, 3, 1-6. <u>http://wsrjournals.org/journal/wjeps/archive/january-2015-vol.-3-(1)</u>
- Khabaz, A. (2016) Monitoring of impact of hooked ends on mechanical behavior of steel fiber in concrete. Construction and Building Materials - Elsevier, Volume 113, 15 June, Pages 857-863. doi: <u>http://dx.doi.org/10.1016/j.conbuildmat.2016.03.142</u>
- 5. **Amjad Khabaz**. (2016) Performance evaluation of corrugated steel fiber in cementitious matrix. Construction and Building Materials Elsevier, Volume 128, 15 December 2016, pages 373-383. doi: <u>http://dx.doi.org/10.1016/j.conbuildmat.2016.10.094</u>
- Khabaz, A. (2014) Non-Metallic Fiber Reinforced Concrete. LAP LAMBERT Academic Publishing. ISBN 978-3-659-50914-8. <u>https://www.morebooks.de/store/gb/book/non-metallic-fiber-reinforced-concrete/isbn/978-3-659-50914-8</u>
- Krasnikovs, A., Khabaz, A., Shahmenko, G. and Lapsa, V. (2008) Glass and Carbon Fiber Concrete Micromechanical and Macromechanical Properties. Proceedings of Riga Technical University, Transport and Engineering, 28, 132-141.
- 8. **Khabaz, A**. (2015). 2D Investigation of Bonding Forces of Straight Steel Fiber in Concrete. Open Access Library Journal, 2, e1991, pp.1-8. doi: <u>http://dx.doi.org/10.4236/oalib.1101991</u>
- Hull, D. and Clyne, T.W. (1996) An Introduction to Composite Materials. 2nd Edition, Cambridge University Press, Cambridge. <u>http://dx.doi.org/10.1017/CBO9781139170130</u>
- 10. Fachvereinigung Faserbeton. (1995) Glassfibre reinforced concrete: practical design and structural analysis, Beton-Verl., e.V.
- 11. Kim, D.J., El-Tawil, S. and Naaman, A.E. (2008) Loading Rate Effect on Pullout Behavior of Deformed Fibers. ACI Materials Journal, 105, 576-584.
- 12. Tuyan, M. and Yazici, H. (2012) Pull-Out Behavior of Single Steel Fiber from SIFCON Matrix. Construction and Building Materials, 35, 571-577. http://dx.doi.org/10.1016/j.conbuildmat.2012.04.110

13. Jung Jin Kim, Dong Joo Kim, Su Tae Kang, Jang Hwa Lee. (2012) Influence of sand to coarse aggregate ratio on the interfacial bond strength of steel fibers in concrete for nuclear power plant, Nuclear Engineering and Design 252, 1–10.

The International Conference of Materials and Engineering Technology

- Krasņikovs, A., Kononova, O., Khabaz, A. and Vība, J. (2010) Fiber Concrete Non-Linear Fracture Control through Fresh Concrete Flow Numerical Simulation. Journal of Vibroengineering, 12, 149-160.
- 15. Zhandarov, S. and Mäder, E. (2014) An Alternative Method of Determining the Local Interfacial Shear Strength from Force-Displacement Curves in the Pull-Out and Microbond Tests. International Journal of Adhesion & Adhesives, 55, 37-42. <u>http://dx.doi.org/10.1016/j.ijadhadh.2014.07.006</u>
- 16. Barbosa, M.T.G. and Filho, S.S. (2013) Investigation of Bond Stress in Pull out Specimens with High Strength Concrete. Global Journal of Researches in Engineering Civil and Structural Engineering, 13.
- Koyanagi, J., Nakatani, H. and Ogihara, S. (2012) Comparison of Glass-Epoxy Interface Strengths Examined by Cruciform Specimen and Single-Fiber Pull-Out Tests under Combined Stress State. Composites: Part A, 43, 1819-1827. <u>http://dx.doi.org/10.1016/j.compositesa.2012.06.018</u>
- 18. J. Humbert, J. Baroth L. Daudeville. (2010) Probabilistic analysis of a pull-out test, Materials and Structures 43:345–355.
- 19. B. Morlin, L. M. Vas, T. Czigany. (2013) Investigation of fiber/matrix adhesion: test speed and specimen shape effects in the cylinder test, J Mater Sci 48:3185–3191.
- 20. Bilisik, K. (2011) Properties of Yarn Pull-Out in Para-Aramid Fabric Structure and Analysis by Statistical Model. Composites: Part A, 42, 1930-1942. http://dx.doi.org/10.1016/j.compositesa.2011.08.018
- 21. Mpalaskas, A.C., Vasilakos, I., Matikas, T.E., Chai, H.K. and Aggelis, D.G. (2014) Monitoring of the Fracture Mechanisms Induced by Pull-Out and Compression in Concrete. Engineering Fracture Mechanics, 128, 219-230. <u>http://dx.doi.org/10.1016/j.engfracmech.2014.07.020</u>
- 22. Alam, Md.J.I., Lo, S.R. and Karim, M.R. (2014) Pull-Out Behaviour of Steel Grid Soil Reinforcement Embedded in Silty Sand. Computers and Geotechnics, 56, 216-226. http://dx.doi.org/10.1016/j.compgeo.2013.12.004
- Beckert, W. and Lauke, B. (1996) Finite Element Calculation of Energy Release Rate for Single-Fibre Pull-Out Test. Computational Materials Science, 5, 1. <u>http://dx.doi.org/10.1016/0927-0256(95)00052-6</u>
- 24. **Khabaz, A**. (2015) Determination of Friction Coefficient between Straight Steel Fiber and the Concrete Fri (SSF.C). Advances in Materials, 4, 20-29. http://dx.doi.org/10.11648/j.am.20150402.11
- 25. **Khabaz, A**. (2014) Determination of Friction Coefficient between Glass Fiber and the Concrete Fri<sub>(GF.C)</sub>. International Journal of Materials Science and Applications. Vol. 3, No. 6, pp. 321-324. <u>http://dx.doi.org/10.11648/j.ijmsa.20140306.17</u>
- 26. W. Becker-t, B. Lauke. (1996) Finite element calculation of energy release rate for singlefibre pull-out test, Computational Materials Science 5, 1-11.
- 27. Li, Y., Liu, Y.L., Peng, X.H., Yan, C., Liu, S. and Hu, N. (2011) Pull-Out Simulations on Interfacial Properties of Carbon Nanotube-Reinforced Polymer Nanocomposites. Computational Materials Science, 50, 1854-1860. <u>http://dx.doi.org/10.1016/j.commatsci.2011.01.029</u>
- 28. Banholzer, B., Brameshuber, W. and Jung, W. (2005) Analytical Simulation of Pull-Out Tests—The Direct Problem. Cement and Concrete Composites, 27, 93-101. <u>http://dx.doi.org/10.1016/j.cemconcomp.2004.01.006</u>

81

# FABRICATION, CHARACTERIZATION, AND PERFORMANCE OF MIXED-MATRIX, WATER-TREATMENT MEMBRANES MODIFIED WITH 2-D NANOMATERIALS

The International Conference of M

aterials and Engineering Technology

# AHMED ABDALA\*1, OMNYA ABDALLA1, ZAFAR KHAN GHOURI1

<sup>1</sup>Texas A&M University at Qatar, Chemical Engineering Program, Doha, Qatar.

#### Abstract

Microfiltration and ultrafiltration membranes plays significant role in water treatment and water desalination pre-treatment. The performance of these membranes is measured by their flux, rejection of impurities and fouling resistance. Here, we develop mixed matrix polysulfone (PS) and polyether sulfone (PES) ultrafiltration membranes containing low concentrations of functionalized graphene oxide (f-GO) and Mxene (MX) 2-D nanomaterials. The membranes are fabricated by phase inversion and their hydrophilicity, morphology, porosity, and mechanical properties are investigated using contact angle measurements, SEM, AFM, and dynamic mechanical analysis. The impacts of the 2-D nanomaterial type and loading on the separation of oil-water emulsion are investigated. Regardless of the nanofiller type, there is an optimum loading that provide a balance between hydrophilicity, porosity, and pore size leads to the optimized performance in terms of flux and oil rejection. Moreover, filtering BSA solution through various membranes demonstrated better fouling resistance for f-GO membranes compared to the pristine membrane. Our results indicate that mixed matrix membranes based on 2-D nanomaterials into polysulfone/polyether sulfone membranes offer significant enhancement in the performance, mechanical properties and antifouling characteristics at very low concentration that does not affect the rheological properties of the membrane formulation and therefore requires no alteration to current commercial membrane fabrication process.

Keyword: microfiltration membranes, mixed matrix membranes, graphene, water-oil separation

#### **1. Introduction**

The demand for clean water has increased rapidly over the past years due to increase in world's population and reduced rainfall. A number of conventional and non-conventional technologies has been used to treat water and membrane based technologies are among the most energy efficient and easy to operate technologies [1–3]. Although, polymeric membranes are easy to synthesis, less costly, provide flexible configuration and require low energy, they are bounded by the tradeoff between selectivity and permeability [4,5]. One way to overcome the tradeoff and enhance the properties and performance of polymeric membranes is through the incorporation of nanomaterials [6]. The emerging 2D nanomaterials have contributed to outstanding opportunities for the development of high performance mixed matrix membranes (MMMs). Among the various 2D nanomaterials, MXenes, graphene oxide and its derivatives are undoubtedly the most intensively studied material with extraordinary properties that are more promising compared yp other nanostructured carbon allotropes.

MXenes, belongs to a family of early transition metal carbides, nitrides and carbonitrides [21] are also very attractive due to their large surface area, hydrophilicity, and good mechanical properties. MXene are prepared by selective etching of layered MAX phase and having a common formula Mn + 1XnTz (n = 1, 2, or 3), where M represents early transition metal, A are one of group IIIA



# 2. Materials and Methods

#### 2.1 Materials

Graphene oxide (GO; SE2430) and  $Ti_2AlC$  were purchased from sixth element and Kanthal, Sweden, respectively. While TiC, ethylene diamine, aspartic acid, hydrochloric acid, dimethylacetamide, polysulfone, polyethersulfone, polyvinylpyrrolidone, bovine serum albumin and sodium hydroxide were purchased from Sigma Aldrich

# 2.2 Methods

# 2.2.1 Functionalization of GO with NH<sub>2</sub> and COOH groups.

NH<sub>2</sub>-f-GO was prepared by mixing GO solution with required amount of EDA under reflux at 85° C heated oil for 4 hours. Then the reaction mixture was washed 7 times to mainitain the pH of solution. Similarly, COOH-f-GO was prepreaed by first mixing a solution of 3 g aspartic acid, 36 ml of 1M NaOH and 90 ml of DI water. The mixture was stirred at 24° C for 5 days. After that, the mixture was centrifuged at 10,000 RPM for 2 hours. The remaining solid was washed with DI water several times before mixing it with 5% HCl by 1-hour bath sonication and 5 minutes probe sonication to convert the carboxylate groups into carboxylic acid groups.

#### 2.2.2 Synthesis, exfoliation and delamination of multilayer Ti<sub>3</sub>C<sub>2</sub>Tx powder

Equal amount of Ti<sub>2</sub>AlC and TiC (Sigma-Aldrich) was ball milled and sintering at 1350°C under nitrogen flow to obatined Ti<sub>3</sub>AlC<sub>2</sub> powder with particle size less than 40  $\mu$ m. Then specific amount Ti<sub>3</sub>AlC<sub>2</sub>, was added gradually to the etching solution (LiF+ HCl). The mixture was stirred for 24 h at 35°C and centrifugation at 3500 rpm. The obtained multilayer Ti<sub>3</sub>C<sub>2</sub>Tx powder was suspended in 40 mL of DI water, degassed and sonicated under flowing argon for 1 h in an ice bath followed by centrifugation at 3500 rpm for 30 min to obtain the supernatant containing Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> flakes. Finally, supernatant containing Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> flakes were frozen for 10 min at -80 °C and freeze-dried overnight.

#### 2.2.3 Fabrication of 2D mixed matrix membranes

The mixed matrix ultrafiltration membranes were fabricated via phase inversion method. The control polymer solution was made by mixing 80% DMAc, 5% PVP and 15% PS/PES polymer. Then, the casting solution was prepared by mixing of different wt.% of GO, rGO, f-GO and Mxene to the polymeric solution. After overnight stirring, the casting solutions was degassed and sonicated for 5 min (2s on, 3s off, pulsing). Finally, the homogenious solution was casted on glass plate by a 200  $\mu$ m casting knife via membrane casting machine. After 30 s, the glass plate was immersed into the coagulation bath containing ultrapure (UP) water at ambient temperature for 5 min.

#### **3. Results and Discussion**

The surface and cross-section images morphologies of the selected membranes are compared and exhibited in Fig.1(a-e). Direct comparison of the SEM top-view images show that the addition of f-GO and MXene clearly made the surfaces more rough and porous compared to control PS (Fig.1(a)) and PES (Fig.1(d))membranes due to the presence of many types of hydrophilic groups in f-GO, and MXene which result in enhanced and rapid transfer between the solvent and non-solvent during phase



inversion. Interestingly, the GO, rGO, f-GO and MXene particles were not observed directly on surfaces while, the membrane cross-section images shows a finger-like morphology, typical of PS/PSF synthesized via phase inversion techniques.



**Figure19.** surface SEM images of (a) PS-membrane (b) NH<sub>2</sub>-f-GO-PS membrane (c) COOH-f-GO-PS membrane (d) PES-membrane (e)Mxene-PES-Membrane (top) and corresponding cross section SEM images (bottom)

The atomic compostion of GO before and after functionalization was studied through EDX analysis (table.1). The presence of nitrogen confirms the successful functionalization of GO. Figure 2 shows the Raman spectrum of GO, NH<sub>2</sub>-f-GO and COOH-f-GO. The D and G bands are considreded one of the chacatersitics of carbon based materisals in raman spectrsopscpy that represents the first-order Raman band of all  $sp^2$  hybridized carbon materials and local defect-activated band in  $sp^2$  hybridized carbon respectivly.

Sample	Composition, atomic %				
Sumple	С	0	Ν		
GO	63.15	36.85	0.00		
GO/NH <sub>2</sub>	77.58	15.07	7.35		
GO/COOH	67.44	31.41	1.15		

# Table 1:Energy-dispersive X-ray spectroscopy (EDX) results ofGO, NH2-f-GO and COOH-f-GO

Hence, the values of the ratio of intensities between  $I_G/I_D$  represents the degree of disorder in the final structure. As can be seen the ratio is almost the same in all the samples, indicating minor defect density after functionalization. One major change after functionalization of GO is the appearance of a new peak at 3200 cm<sup>-1</sup>, which may have resulted from increase in the  $NH_2$  groups, as this peak corresponds to the amine/amide groups in the Raman band correlations in literature [10]


Figure 2: Raman Spectrum of of GO, NH2-f-GO and COOH-f-GO

Hydrophilicity behavior of polymeric membrane is also very important parameter, as high hydrophilicity of polymeric membrane is of vital impacts to give high water flux and well accepted that the lower contact angle offers higher hydrophilicity and lower surface roughness. As can be seen from figure 3 and 4, the contact angle of f-GO decreased notably compared to control membrane, indicating higher hydrophilicity due to the fact that f-GO demonstrates high affinity for water during the phase inversion method, thus f-GO rapidly immigrates toward the membrane/ water interface reducing the interface energy, which derives the high hydrophilicity. On the other hand, the contact angle of pristine PES membrane is about 73.8 °, while the value decreased to 69.8° when the 0.05 % MXene is incorporated into the polymeric matrix and further decreased to 64.7 ° with the MXene loading increased to 0.1%, indicating improved hydrophilic surface to attract more water molecules (Figure.5).







This could be due to the fact that the presence of multiple hydrophilic groups (-OH, -O, or -F), the nanochannels, interlayer slits pores and negative charges on the hydrophilic MXene. Interestingly, the contact angle start increasing from 64.7 to 74.7 as the MXene loading increased further from 0.1 to 0.25% and continuously increased to 76.9 ° when the MXene content is 0.5%. The increase in hydrophilicity is directly related to pure water flux as implied by the trend in pure water fluxes for f-GO and MXene in figure 3, 4 and 6, respectively.



Figure 4: water contact angle and pure water fluxes of the COOH-f-GO mixed matrix membranes

The permeability of the membranes increased to a maximum at low loadings (0.05 - 0.2 wt. %) and then decreased at higher loadings (Fig.6). This is due to the agglomeration and irregular dispersion of nanoparticles at higher loadings, decreasing the porosity and increasing the hydrophilicity. Comparing the two sets of NH<sub>2</sub>-f-GO and COOH-f-GO exhibited higher hydrophilicity and permeability, because COOH-f-GO has more oxygen groups making the exchange between the solvent and non-solvent more rapid, hence higher porosity.



Figure 5: water contact angle of the Mxene mixed matrix membranes



Figure 6: pure water fluxes of Mxene mixed matrix membranes

The selectivity of the GO, rGO, f-GO and MXene membranes was studied by carrying out oil rejection experiments. The test was done by filitering a 100-ppm oil emlusion through a dead-end cell and collecting three permeate samples from each membrane. Figure 7 and 8 shows the percentage of oil rejection for all f-GO and MXene membranes. The percentage of oil rejection was slightly enhanced for all f-GO membranes compared to control membrane. The highest oil rejection was attained at 0.1% COOH-f-GO membrane and 0.2% NH<sub>2</sub>-f-GO membrane with 97.92 % and 95.64% rejection respectively while, the MXene membrane results showing same trend as pure water flux increased with the increase in MXene content but the highest oily water flux 407 was achieved at 0.25 % loading of MXene with a rejection of 96.5 %. The wide difference in pure and oily water flux is linked to the clogging of active pores by oil particles during the run. However, the maximum 97.9% oil rejection was achieved at 0.1 % loading.







The fouling resistance of the prepared membranes was assessed by filtering a 500-ppm BSA solution through selected f-GO membranes followed by a systemic cleaning, then checking the recovery and flux decline. The f-GO incorporated membranes demonstrated much slower BSA flux decline as shown in figure 9. This indicates that the addition of f-GO into the polymeric matrix greatly decreased the impact of BSA on the f-GO membrane's performance, as demonstrated by the smaller degree of flux decline due to higher hydrophilicity of f-GO membranes. In addition to that, the second BSA fouling cycle is done to study the flux recovery ratio. The recovery ratios notably increased for the f-GO membrane from 0.69 for the control to 0.89 and 0.9 NH<sub>2</sub>-f-GO membrane and COOH-f-GO membrane respectively. The 0.2 % COOH-f-GO membrane shows the highest recovery of ~90% consistent with having the lowest contact angle among the tested membranes.



Figure 20: Results of BSA flux vs volume for control, 0.2% GO/COOH and GO/NH2

Acknowledgments: All sources of funding of the study should be disclosed. Clearly indicate grants that you have received in support of your research work.

The International Conference of Materials and Engineering Technology

#### **References:**

- 1. M. Zahid, A. Rashid, S. Akram, Z. A. Rehan, and W. Razzaq, "A Comprehensive Review on Polymeric Nano-Composite Membranes for Water Treatment," *J. Membr. Sci. Technol.*, vol. 8, no. 1, pp. 1–20, 2018.
- 2. D. Qadir, H. Mukhtar, L. K. Keong, D. Qadir, H. Mukhtar, and L. K. Keong, "Mixed Matrix Membranes for Water Purification Applications Mixed Matrix Membranes for Water Puri fi cation Applications," vol. 2119, 2017.
- 3. L. Huang and H. Lin, "Engineering Sub-Nanometer Channels in Two-Dimensional Materials for Membrane Gas Separation," *Membranes (Basel).*, 2018.
- 4. V. Kochkodan and N. Hilal, "A comprehensive review on surface modified polymer membranes for biofouling mitigation," *Desalination*, vol. 356, pp. 187–207, 2015.
- 5. L. M. Robeson, "Correlation of separation factor versus permeability for polymeric membranes," *J. Memb. Sci.*, vol. 62, pp. 165–185, 1991.
- N. Song, X. Gao, Z. Ma, X. Wang, Y. Wei, and C. Gao, "A review of graphene-based separation membrane: Materials, characteristics, preparation and applications," *Desalination*, vol. 437, no. February, pp. 59–72, 2018.
- 7. X. M. Han, Runlin, "Preparation of a new 2D MXene/PES composite membrane with excellent hydrophilicity and high flux," *RSC Adv.*, pp. 56204–56210, 2017.
- 8. H. Huang, Y. Ying, and X. Peng, "Graphene oxide nanosheet: An emerging star material for novel separation membranes," *J. Mater. Chem. A*, vol. 2, no. 34, pp. 13772–13782, 2014.
- 9. X. Wang, Y. Zhao, E. Tian, J. Li, and Y. Ren, "Graphene Oxide-Based Polymeric Membranes for Water Treatment," *Adv. Mater. interfaces*, vol. 1701427, pp. 1–20, 2018.
- S. George, Infrared and Raman Characteristic Group Frequencies Conten: tables and charts, Third Edit. West Sussex PO19 IUD, England National: John Wiley & Sons, Incorporated, 2001.

# MATERIAL CHARACTERISTICS OF POROUS COPPER FOAMS FOR HEAT TRANSFER PERFORMANCE ASSESSMENT

#### OZKAN KIREC \*1, MELDA OZDINC CARPINLIOGLU <sup>2</sup>

<sup>1</sup> Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY
 <sup>2</sup> Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY

#### Abstract

Porous metals are cellular structural materials with a large volume fraction of pores. The characteristic features like high surface area density, low weight and ability to mix fluids have accredited metal foam as a potential choice in a variety of thermal engineering applications. Characterization of the metal foam samples in terms of their specific properties is significant for the investigations on heat transfer performance evaluation. A variety of cylindrical type Cu foam samples with a diameter of D, 25 mm and different thicknesses, t in the range of 2 mm  $\leq$  t  $\leq$  5 mm with a 90 % - 95 % porosity range were experimentally tested at a heating temperature of T<sub>H</sub> = 80 °C for a contact time of  $\Theta$  = 10 min. Scanning electron microscopy (SEM) device was used for analyzing the micro-structure and material characteristics of the porous Cu metal foams. SEM analysis results were compared with Bruker microCT software programme analysis results. In this paper, results are presented to determine the material characteristics of the porous medium influenced by t, T<sub>H</sub> and  $\Theta$ .

Keyword: Copper Foam, Heat Load, Thermal Analysis, SEM

#### **1. Introduction**

Metal foams can be made of materials such as aluminium, magnesia, tin and copper. The cell structure of metal foam is of statistical nature, where the cells and the pore sizes are more or less statistically scattered, depending on the related manufacturing process. Two major foam types can be distinguished, closed cell and open cells Open cell foams are permeable materials with metallic properties. High porosity open-cell metal foam is a relatively recent development. Open-cell metal foams can be produced with a large range of pore sizes, types and properties depending on the application.

The characteristics properties of metal foam as high surface area, light weight and porosity, make them in use in many engineering cases [1]. Thermal applications of foams consist of heat exchangers for air-cooled condenser towers, airbone equipment and heat sinks for electronic devices [2]. Many researches have investigated the characterization of metal foams for further studies in the engineering field and applications. Because of the complex geometry of the foam, correct definitions of the material are so important through the micro-structure morphology as a function of porosity, pore size, shape and other geometrical parameters.

There are a few researches on open cell Cu foam materials to define their material characteristics in relevance to thermal characteristics. In the presented study, a variety of Cu metal foams in the form of cylindrical parts as spots were tested. SEM analysis and Bruker microCT software programme analysis were referred for the micro-structure characteristics. Micro structure analysis are presented in thermal performance tests through the change of local temperatures and surface mean temperatures.



#### 2. Material and Methods

Porous metal foams feature a very homogeneous structure as can be seen from Figure 1.



Figure 1. Geometric properties of open cell metal foams (This image is taken from, [3] page 8, Figure 2.2)

As can be seen from Figure 1, pore size,  $d_{pore}$  which defines the unit cell diameter, edge thickness,  $d_{edge}$  defines the strut thickness,  $d_{face}$  defines the interior pore diameter and ligament length,  $L_L$  defines the length of edge of the pore. A certain number of pores can be achieved by both, small pores with thick struts, as well as by large pores with small struts or ligaments. Despite having the same pore density, this results in very different ratios of void volume,  $V_{Pore}$  and volume of the parent metal,  $V_{foam}$ .

Prior research in the metal foam literature has generally regarded the Kelvin unit cell as the idealized foam structure best describing metal foams [4-7]. This unit cell is relatively easy to model as a single pore described by a regular polyhedral. Some prior works have been done regarding the classification of closed-cell metal foam structures using X-rays and computed tomography (X-ray CT) [8, 9].

The common material characteristics of foams can be listed as :

Pore dependent properties as : Pore shape, pore size,  $d_{pore}$ , strut thickness,  $d_{edge}$ , interior pore diameter,  $d_{face}$  and ligament length,  $L_L$ .

Foam properties including overall foam pattern : Void volume,  $V_{pore}$ , volume of parent metal,  $V_{foam}$ , porosity,  $\epsilon$  and pore per 1nch, PPI.

Porosity,  $\epsilon$  is the ratio of the cross-sectional area of the pores, A<sub>pore</sub> and the cross-sectional area of the flow channel, A<sub>foam</sub>.  $\epsilon$  is defined in the Equation (1).

$$\varepsilon = A_{\text{pore}} / A_{\text{foam}} \tag{1}$$

PPI is usually employed to characterize the pore size of the foam metal which is defined in the Equation (2)

$$PPI = 25.4 / d_p$$
 (2)

where  $d_p$  is the pore size.



#### 2.1 SEM Analysis

A variety of cylindrical type Cu foam samples as spots which are shown in the Figure 2 with a diameter of 25 mm and different thicknesses, t in the range of 2 mm  $\leq t \leq 5$  mm were tested. The magnitude of porosity is with 90 % - 95 % porosity range and pore per inch (PPI) is 40. These porosity values were measured by using the Bruker microCT software programme [10] on SEM image views of the cylindrical Cu foams.

The International Conference

aterials and Engineering Technology



Figure 2. Cylindrical type Cu foam samples

SEM analyses of the cylindrical Cu foams of t = 2 mm, t = 3 mm and t = 5 mm were taken by using JEOL 6390LV scanning electron microscopy at the Gaziantep University Physics laboratory. SEM is a type of electron microscope that creates various images by focusing a high energy beam of electrons onto the surface of a sample and detecting signals from the interaction of the incident electrons with the sample's surface. The primary function of the SEM is an imaging platform that can image using secondary electrons (SE) or backscattered electrons (BSE).

Analysis results of the sample are shown in the Figure 3 as a case measurement applied for t = 2 mm for a variety of magnifications of 25,40 and 600.





**Figure 3**. SEM images of the t = 2 mm Cu foam sample with different scales and measured dimensions

SEM images determine pore shape, pore size, connection between the pore structures, pore distribution which are different in micro-structure. The presence of micro pores are observable for a magnification of 600 having a size range of 20 $\mu$ m. As can be seen from Figure 3 for a magnification of 40 having a size range of 500  $\mu$ m the dimensions of pores are determined as; d<sub>p</sub> are 1.47 mm and 1.52 mm and d<sub>i</sub> are 424.53  $\mu$ m and 522.23  $\mu$ m.

SEM analysis for a variety of t, at a given magnification of 40 having a size range of 500  $\mu$ m indicated that measured dimensions inside the micro-structure were different as shown in Figure 4.



t = 2 mm

t = 3 mm





Figure 4. SEM analysis results of different thickness Cu foams with measured dimensions

It is seen from Figure 4 that thickness causes variations on the pore shape, pore direction, pore structure and connections, measured dimensions changed inside the porous medium respectively.  $d_p$ ,  $d_i$  and  $L_L$  values of the samples are  $d_p$  are 1.47 mm and 1.52 mm,  $d_i$  are 424.53  $\mu$ m and 522.23  $\mu$ m,  $L_L$  are 472 mm, 588 mm and 725 mm for 2 mm.  $d_p$  are 1.42 mm and 1.71 mm,  $d_i$  are 484.66  $\mu$ m and 530.33  $\mu$ m,  $L_L$  are 537 mm, 562 mm and 770 mm for 3 mm and  $d_p$  are 1.45 mm and 1.67 mm,  $d_i$  are 577.60  $\mu$ m and 586.90  $\mu$ m,  $L_L$  are 630 mm, 656 mm and 726 mm for 5 mm.

Analysis results also showed that some micro pores are seen in the micro-structures of the samples. Sample's micro structure views which magnified by 600 and 1000 times are shown in the Figure 5 (a,b,c).



a)





c)

**Figure 5 (a,b,c)**. Micro-structure morphology of the 2 mm (a), 3 mm (b) and 5 mm (c) of the samples.

Micro-structural analysis results are stated in the Figure 5 showed that 3 mm and 5 mm samples included variety of micro pores with different size and geometric shapes. There are also some differences on the surface of the samples, for example 2 mm and 5 mm samples's surface have irregular micro fiber material structure. Surface structure of the 3 mm sample was more smooth compared with other sample surfaces. As an effect of t following dimensions are given for porous foam microstructure :

Pore diameter,  $d_p$  changed between 1.4 mm <  $d_p$  < 1.5 mm, ligament length,  $L_L$  changed between 450 mm <  $L_L$  < 750 mm for t = 2 mm, similarly 1.4 mm <  $d_p$  < 1.7 mm, 530 mm <  $L_L$  < 800 mm for t = 3 mm and 1.4 mm <  $d_p$  < 1.7 mm, 600 mm <  $L_L$  < 730 mm for t = 5 mm.

#### 2.2 Bruker microCT Analysis

CT-Analyser (CTAn) is an application for measuring quantitative parameters and constructing visual models from scanned 3D datasets obtained with SkyScan microCT instruments. It can also be used for 3D and 2D images obtained from other imaging modalities. CTAn allows real time volume rendered model viewing; surface rendered models are also created by CTAn for viewing in another program – CT-Volume ("CTVol"). Quantitative measurement is made both of densitometry (voxel attenuation coefficient or calibrated density) and of morphometry, the latter based on a segmented (black and white) image. Thresholding or "segmentation" is done by simple global or by adaptive



methods. Before analysis, adjusting of the pixel size according to magnification of the Cu foam was made. Necessary information taken from CTan user manual. Analysis results were given in the Figure 6 and compared with SEM analysis results.



Figure 6. MicroCT analysis of the t = 2 mm Cu foam sample with different scales and measured dimensions

From the Figure 6, micro-structures of the Cu foam samples can be seen. The software program enables the 2D or 3D analysis of the each point or region which needed for micro structure analysis studies which can be selected by analyser. Program gives more information about the Cu foam samples such as, tissue and bone area, area of closed and open pore, total area of pore space, closed porosity and open porosity values. Also, pore size, pore distributions and orientations can be seen in detail.

It is clear from the analysis results, if bone area decreases, total porosity,  $\varepsilon$  increases.  $\varepsilon$  change is approximately 4.5 % between Cu foam samples. Depending on the total area of pore space,  $\varepsilon$  changes.  $\varepsilon$  increases with increasing pore space area. In addition to these results, particle porosity and number of particles values can be seen from the analysis programme.

As a result of the SEM and Bruker MicroCT analysis, it is important to see the effects of the porosity on the mean ligament length,  $L_{L,m}$ , mean pore diameter,  $d_{p,m}$ , and mean inner hole diameter,  $d_{i,m}$  for this aim, comparisons are made and shown in the Figure 7 (a,b).



Figure 7 (a,b). Variations of LL,m / dp,m as a function of  $\epsilon$  (a), and variations of LL,m / di,m as a function of  $\epsilon$  (b)

It is clear from Figure 7 (a,b) depend on the porosity values, some variations are seen in the material properties,  $LL,m/d_{p,m}$  value is highest at high  $\epsilon$ . for example  $LL,m/d_{p,m}$  value is 432.688 and highest at 5 mm sample at specified  $\epsilon$  of 94.7 %. Lowest value was seen in the 3 mm sample, reasons are probably effects of the micro-structure properties of the sample by means of pore size, shape, direction and micro pores.

On the other hand,  $L_{L,m}/d_{i,m}$  values decreased with increasing t and  $\epsilon$ . This value is highest at 2 mm sample with porosity of 90.7 % and is lowest at 5 mm sample with porosity of 94.7 %.

Analysis results are compared with Bruker MicroCT analysis programme and shown in the Table 1.

Micro Structure Properties of The Cu Foam Samples						
Thickness ( mm )	Pore Diameter, dp ( mm )	Inner Hole Diameter, di ( mm )	Ligament Length, L <sub>L</sub> ( mm )	Micro Pores	Porosity, ε %	
2	1.4 - 1.5	0.4 - 0.5	(472 - 588 - 725)	-	$90.7^{*}$	
3	1.4 - 1.7	0.4 - 0.6	(537 - 562 - 770)	Available	93.5 <sup>*</sup>	
5	1.4 - 1.7	0.5 - 0.6	(630 - 656 - 726)	Available	94.7*	

**Table 1** : Measured micro structure properties of the samples

\*Porosity values calculated by using the Bruker microCT software programme for SEM image views of the cylindrical Cu foams

It is clear from the Table 1 that pore diameters  $d_p$ , inner hole diameters  $d_i$ , ligament lengths,  $L_L$  and  $\epsilon$  show some differences depending on the thickness of the Cu foam samples. It is important to note that t = 3 mm and t = 5 mm Cu foams include variety of micro pores. Analysis results also showed that porosity values increased with increasing sample thickness and depend on the increasing thickness, ligament lengths increase with increasing pore diameter and inner hole diameter.



These results show the manufacturing of the Cu foams are not same. Effects of the porosity as a function of t are shown and comparisons are also made in the following sections.

#### 3. Experimental Methodology

In this paper, an extensive experimental study [11-14] are outlined on heat transfer performance of porous Cu foams and thermal characteristictics calculations described in the previous conference papers of the authors [15,16]. In the experimental study cylindrical type Cu foam samples were located on the heater unit shown in the Figure 8 and heater temperatures (T<sub>H</sub>) were adjusted at constant magnitude of 80 °C. Thermal image views of the upper and contact surface of the samples were taken by Testo 875-2i thermal image camera periodically for a contact time;  $\Theta = 10$  min. Test samples were kept on the heater unit with a specified T<sub>H</sub> during  $\Theta = 10$  min. Thermal camera views of the test samples were taken at the end of time period from the upper surface and reversed contact surface to determine variation of local temperature, T as a function of selected and measured directions.



Figure 8. Location of the samples on heater unit at specified heating temperature,  $T_H$ 

Thermal and histogram image views of the samples are analyzed to see the heat transfer behavior as a function of t. Histogram analysis results gave the min., max. and mean temperatures values on the sample surface at specified experimental conditions. As a sample histogram picture is shown in the Figure 9.



Figure 9. Histogram view of the sample

From that figure, percentage distribution of the temperature values are seen along the sample surfaces.

#### 4. Results and Discussions

As a result of the experimental study, thermal image analyses of the samples are made and analysis results are compared as a function of sample thickness at stated  $T_H$  and  $\Theta$ . Figure 10 indicates the variation of local temperature distribution along the upper surfaces of the Cu foam samples as a function of t at a heating temperature of  $T_H = 80$  °C and contact time,  $\Theta = 10$  min.



Figure 10. Temperature profiles of Cu foam samples along the surface

It is seen and local analysis can be made from the Figure 10, upper surface, min., max. and mean temperature values of the samples are; 57.2 °C, 74.6 °C and 63 °C for t = 2 mm. 52.9 °C, 74.8 °C and 64.4 °C for t = 3 mm and 53.1 °C, 82.9 °C and 60.5 °C for t = 5 mm respectively. It is also clear that, upper surface temperature is lowest along the t = 5 mm sample which compared with other samples.



Analysis results showed the irregular distribution of the porosity inside the materials and temperature variations along the surface is highest for 5 mm sample. For example, temperature difference between min. and max.values is 29.8 °C. It is also important to note that edge temperatures of the samples are higher than other surface which temperature values can reach up to 85 °C depend on the thickness.

Effects of the porosity on the heat distribution are seen from the Figure 11 as follows;



Figure 11. Variations of upper surface mean temperatures as a function of  $\varepsilon$ 

It is seen from the Figure 11 that, depend on the t and  $\varepsilon$  values,  $T_{m,upper}$  values of the samples changed.  $T_{m,upper}$  value is 60.5 °C and lowest for 5 mm sample and 64.4 °C and highest for 3 mm sample. If comparisons are made between the  $\varepsilon$  values and  $T_{m,upper}$  values. Depending on the increasing t and  $\varepsilon$ , firstly an increase and later a decrease is seen for  $T_{m,upper}$  values of the samples according to the results. These results indicate the effects of micro-structure on t, for heat transfer performace of the

#### 5.Conclusions

material.

Experimental analysis results and effects of the porosity based on the sample thickness are summarized as;

- 1) Irregular distribution of the porosity and availability of the micro pores inside the material structure obviously effect the heat transfer performance of the samples.
- 2) If porosity values increase, ligament length, pore diameter and inner hole diameter will increase (Table 1)
- 3) Bruker microCT programme gave more sensitive results compared with manufacturing company datas.
- 4) LL,m / dp,m value is highest at high porosities, on the other hand, LL,m / di,m values decreased with increasing t and  $\epsilon$
- 5) Upper surface mean temperature values is lower at higher sample thickness.
- 6) Min. and max. temperature variations along the sample surface are higher at higher sample thicknesses.
- 7) Depending on the material's thickness, porosity values effects the upper surface mean temperature values, analysis results generally showed that, if porosity values increase, upper surface mean temperature values will decrease.

#### **References :**

1. A. Bhattacharya, V.V. Calmidi, R.L. Mahajan, Thermophysical properties of high porosity metal foams, Int. J. Heat Mass Transf. 45 (5) (2002) 1017–1031

The International Conference of Materials and Engineering Technology

- 2. K.A. Afimiwala, R.W.Mayne, R.K. Shah, Heat exchanger optimization, Proceedings of the Sixth International Heat Transfer Conference, Toronto, Canada, 4 1978, pp. 185–191.
- S.D. Schampheleire, P. D. Jaeger., H. Huisseune., B. Ameel., C. T'Joen., K.K. Kerpel., M. D. Paepe. (2013). Thermal hydraulic performance of 10 PPI aluminium foam as alternative for louvered fins in an HVAC heat exchanger, *Applied Thermal Engineering* 51 371–382.
- 4. Dai, Z., Nawaz, K., Park, Y. G., Bock, J., and Jacobi, A. M. (2010). "Correcting and extending the Boomsma-Poulikakos effective thermal conductivity model for threedimensional, fluid-saturated metal foams." International Communications in Heat and Mass Transfer, Vol. 37, pp. 575-580.
- Mahjoob, S., and Vafai, K. (2008). "A synthesis of fluid and thermal transport models for metal foam heat exchangers," International Journal of Heat and Mass Transfer, Vol. 51, pp. 3701-3711.
- 6. Krishnan, S., Murthy, J. Y., and Garimella, S. V. (2006). "Direct Simulation of Transport in Open-Cell Metal Foam," Journal of Heat Transfer, Vol. 28, pp. 793-800.
- 7. Schmierer, E.N., Razani, A., Keating, S., and Melton, T. (2004). "Characterization of high porosity open-celled metal foam using computed tomography," American Society of Mechanical Engineers, Heat Transfer Division, HTD, Vol. 375, pp. 415-424.
- Saadatfar, M., Garcia-Moreno, F., Hutzler, S., Sheppard, A. P., Knackstedt, M. A., Banhart (2009). "Imaging of metallic foams using X-ray micro-CT," Colloids and Surfaces A: Physicochemical and Engineering Aspects, Vol. 344 pp. 107-112.
- 9. Bodla, K. K., Murthy, J. Y., and Garimella, S. V. (2010). "Microtomography-based simulation of transport through open-cell metal foams," Numerical Heat Transfer, Part A: Application. Vol. 58, pp. 527-544
- 10. https://www.bruker.com/products/microtomography/micro-ct-software/3dsuite.html
- 11. O. Kirec, Heat Transfer Performance of Porous Copper Foam (M.Sc Thesis), Gaziantep University, Turkey, 2017
- M. Ozdinc Carpinlioglu., O. Kirec., A measurement practice for thermal characteristics of porous mediums, 9<sup>th</sup> International Conference on Engineering Mechanics, Structures, Engineering Geology, (EMESEG ' 16)
- M. Ozdinc Carpinlioglu., O. Kirec., Thermal image analysis of porous metal foams for material characterization, International Conference of Energy and Thermal Engineering, Istanbul 2017
- 14. O. Kirec., M. Ozdinc Carpinlioglu., An experimental study on the determination of thermal conductivity, heat capacity and thermal diffusivity of a porous metal foam, 2<sup>nd</sup> International Energy and Engineering Conference, Gaziantep University, 2017, Turkey
- 15. M. Ozdinc Carpinlioglu., O. Kirec., Thermal conductivity of a porous metal foam as a function of heat load, 4<sup>th</sup> international porous & powders materials symposium and exhibition, (PPM 2019)
- 16. O. Kirec., M. Ozdinc Carpinlioglu., Heat transfer performance analysis of porous metal foams at different heating temperatures by thermal imaging technique, 4<sup>th</sup> international porous & powders materials symposium and exhibition, (PPM 2019)

## SURFACE FINISHING OF AEROSPACE MATERIALS

The International Conference of Materials and Engineering Technology

#### OSMAN SOYDAN<sup>1</sup>, KÜRŞAD GÖV<sup>\*2</sup>, ÖMER EYERCİOĞLU<sup>3</sup>

<sup>1</sup>Gaziantep University, Faculty of Aeronautics and Astronautics, Aircraft and Aerospace Engineering Department, Gaziantep, TURKEY

<sup>2</sup> Gaziantep University, Faculty of Aeronautics and Astronautics, Aircraft and Aerospace Engineering Department, Gaziantep, TURKEY

<sup>3</sup>Gaziantep University, Faculty of Engineering, Mechanical Engineering Department, Gaziantep, TURKEY

#### Abstract

Abrasive flow machining is a non-traditional production method applied to complex surfaces. It is a very useful method to achieve good surface quality which is very important nowadays. Especially, it is desired that the surface quality of materials with high corrosion and high heat resistance used in aviation and automotive industry is good. In this paper, the effects of abrasive flow machining (AFM) technique on the surface quality of material such as Ti-6Al-4V, especially used in the aerospace industry, have been observed. Taguchi method was used to see appropriate parameters and noise factors. The surface roughness values ( $R_a$ ), material removal rate (MRR) were measured and the white layer formed by high heat during cutting was removed in the high cycle numbers. Wire electrical discharge machining (Wire EDM) process the white layer formed during cutting and corrected by the AFM technique is an undesirable layer on the material surface. Experiments were carried out by changing the abrasive concentration and abrasive mesh size in this abrasive media. The number of cycles was increased, and experiments were continued with up to 50 cycles. The best surface quality values were achieved in experiments using 180 abrasive grain size and 60% abrasive concentration. The surface roughness ( $R_a$ ) values, material removal rate (MRR) were measured and the white layer formed by high heat during cutting was removed in the high process cycles.

Keyword: Abrasive flow machining, aerospace materials, finishing process, surface roughness, material removal

#### 1. Introduction

UWE Bristol

Surface finishing operations are the most costly operations in the manufacturing sector. Besides being costly, it is also a long time-consuming process. In addition, the parts used in industry have taken an increasingly complex shape, and traditional methods of processing the surfaces of these parts are insufficient. New methods are being developed to save time and reduce costs. Abrasive Flow Machining (AFM) is one of these newly developed methods.

Abrasive Flow Machining was developed by Extrude Hone Corporation in the 1960s [1]. Nowadays, needs have changed with the developing technology. Especially in the medical, automotive, and aerospace industries, the surface quality of the increasingly complex parts is desired to be excellent. The AFM method is a suitable method for improving the surface quality of complicated shaped workpieces [2]. It is a very convenient method to use to improve the quality of hard-to-reach surfaces [3]. Surface quality of aerospace components is important for fuel savings [4].

Kumar and Hiremath [5] applied the AFM method to complex shaped components with different abrasive types. The parameters used in the AFM method and the surface of the part used affect the decrease in surface roughness values ( $R_a$ ) [6]. Rhoades [7] also stated that the previous surface quality of the workpiece was important while achieving the desired surface quality.

Abrasive media is the most important parameter in the AFM process [8]. it is stated that it is difficult to determine parameters such as the type and amount of abrasives to be used in the media prior to application in the AFM process [9]. AFM processing has been applied to parts used especially in automotive and aviation, with different abrasive ratios and mesh sizes and variable cycle numbers

[10]. When the AFM parameters were examined, the improvement in surface roughness values decreased as the abrasive concentration decreased [11].

The International Conference of Materials and Engineering Technology

During the Electrical Discharge Machining (EDM) process, a very hard layer forms on the surface of the part being studied [12]. This unproductive layer is called the white layer [13]. The amount of material reattached to the material surface during the EDM process affects the  $R_a$  values [14]. The type of cooling water used in the EDM process also affects the  $R_a$  value and it is stated that de-ionized water gives the best results [15].

The AFM method has been proposed to improve the surface quality of workpieces prepared with Wire EDM [16]. Göv and Eyercioğlu [17] applied the AFM method to the Ti-6Al-4V workpiece produced by the EDM method with abrasive media prepared using SiC and stated that the inefficient layer had been removed. Göv and Eyercioğlu [18] also applied different types of abrasives to the surfaces of samples prepared with the Wire EDM method and stated that the undesirable layer was removed more quickly using SiC and B<sub>4</sub>C (Boron carbide).

The AFM method uses various methods to determine the parameters, and the Taguchi method is one of these methods [19]. Ibrahim [20] used the Minitab program to estimate the amount of material removal.

The analysis programs, which have become widespread with the developing technology, are also used for the AFM method. Seifu et al. used ANSYS FLUENT to work on the amount of material removal in the AFM method [21].

In this study, improvement of surface quality by applying the AFM method to Ti-6Al-4V workpiece commonly used in aviation sector was studied experimentally. However, the process parameters; abrasive mesh size, abrasive concentration, number of cycles and changing the type of abrasive, output parameters; material removal rate, surface roughness value and SEM images, were studied experimentally and the results of the effects outlined.

#### 2. Material and Methods

#### 2.1. Abrasive Flow Machine

In this study, two-way AFM machine was used. The machine has three units; the hydraulic system, the workpiece holder, and the electronic controller. The hydraulic unit provides sufficient pressure. The number of cycles is determined by the control screen. In the two-way AFM machine, a cycle starts with the upward movement of the lower and upper pistons at the lowest level, and one cycle is obtained by returning to their former position. Depending on the speed of the pistons, the time is measured as two minutes for one cycle in this experiment. There is also a holder for placing work pieces to be used in experiments. With this holder, the desired surface of the workpiece can be processed. Figure 1 shows the Two-way AFM machine used in the studies and located within the University of Gaziantep.



aterials and Engineering Technology

Figure 1. Two-way abrasive flow machine

#### 2.2. Workpiece Material

Ti-6Al-4V sample to be used in the experiment were cut by using Wire EDM of 5x10x20 dimensions. The R<sub>a</sub> values of each samples to be used were measured and all of weights were recorded. So, the MRR was calculated. Figure 2 shows a picture of the workpieces used in the studies.



Figure 2. Workpiece Ti-6Al-4V

#### 2.3.Abrasive Media

400, 240, and 180 mesh sizes of SiC and 20%, 40%, and 60% polymer based abrasive media is prepared for use and 1,3,5,10,20, and 50 cycle numbers have been studied in this study. One of the prepared abrasive medias is shown in Figure 3.



Figure 3. Polymer-based abrasive media

104



#### 2.4. Experimental Design

We decided on the parameters to use in the experiments and used the Taguchi method to experiment with different combinations of parameters. We determined the number of cycles, abrasive mesh size, abrasive concentration as parameters and created the necessary combinations in the Taguchi method using Minitab and shown in Table 1.

Α	В	С	D	Number of	Number	Abrasive	Abrasive
				Experiments	of Cycle	Mesh Size	Concentration
1	1	1	1	1	1	400	20
2	1	2	2	2	1	240	40
3	1	3	3	3	1	180	60
4	2	1	1	4	3	400	20
5	2	2	2	5	3	240	40
6	2	3	3	6	3	180	60
7	3	1	2	7	5	400	40
8	3	2	3	8	5	240	60
9	3	3	1	9	5	180	20
10	4	1	3	10	10	400	60
11	4	2	1	11	10	240	20
12	4	3	2	12	10	180	40
13	5	1	2	13	20	400	40
14	5	2	3	14	20	240	60
15	5	3	1	15	20	180	20
16	6	1	3	16	50	400	60
17	6	2	1	17	50	240	20
18	6	3	2	18	50	180	40

#### Table 1. Determination of parameters by Taguchi method

#### 2.5. Experimental Procedure

The experiments were performed on surface of Ti-6Al-4V. The workpieces were placed in a suitable size holder. One side of the holder has an openness to influence the workpiece surface of the abrasive media. Hence, we can observe the effect of the abrasive media flowing from the surface of the workpieces and the previous version of the workpieces. Each sample was operated with the calculated concentration and mesh size of abrasive. Pre-weighed samples are weighed once more after the process by running Shimadzu Aux220 balance belonging to the University of Gaziantep. The surface roughness value was measured by using Mitutoyo SJ 401 machine belonging to the University of Gaziantep. The SEM images of the surfaces were taken by using Zeiss GeminiSEM 300.

#### 3. Results and Discussion

#### **3.1. Material Removal Rate**

Each workpiece whose weights were previously measured with precision scales was also measured after AFM. In experiments where a cycle number lasted two minutes, post-processing numbers and weights of the samples executed in the desired cycles were recorded. So, we had all the values needed to determine the amount of material removed in the unit time shown in Equation 1 and were able to calculate the MRR. In Figure 4, graphs of experiments with media prepared in the size of 400 abrasive grains are given. 20%, 40%, 60% abrasive concentrations of three different media prepared by the results of the experiments were shown.



Figure 4. MRR of Ti-6Al-4V in 400 abrasive mesh size

The maximum material removal rate in the experiments using the Ti-6Al-4V sample was after the experiment using media with an abrasive concentration of 60%. A fast MRR has been observed from the twentieth to the fiftieth cycle.

Figure 5 shows the work done with the media prepared in 240 abrasive grain sizes. A graph of the values obtained from three different concentrations of abrasives is shown.



Figure 5. MRR of Ti-6Al-4V in 240 abrasive mesh size

MRR increased in studies with 240 abrasive grain sizes. For Ti-6Al-4V, up to 50 cycles of MRR at 60% abrasive concentration were observed to follow rapidly increasing course.

Figure 6 shows a graph of values taken in 180 abrasive grain sizes and three different abrasive concentrations.



Figure 6. MRR of Ti- 6Al-4V in 180 abrasive mesh size

After experiments with 180 abrasive mesh size, the maximum MRR was observed in this abrasive mesh size.

Experiments with media containing 60% abrasive for Ti-6Al-4V samples showed a fast MRR compared to other abrasive concentrations.

#### **3.2. Surface Observations**

Three  $R_a$  values were taken from the surface of Ti-6Al-4V samples. Three of these values were measured in the direction perpendicular to the flow and their averages were taken. Surface roughness graphics are formed with these obtained values.

As a result of the studies, the best surface quality of different media types was obtained with the media prepared with 180 abrasive mesh size and 60% abrasive concentration. Surface quality improves as values get closer to zero.

SEM images of samples used in the studies were taken. In the images taken, an inefficient layer called the white layer was observed after the EDM process. In experiments with different abrasive mesh sizes and abrasive concentrations, it was observed whether the white layer was removed.

#### 3.2.1. Processed Surfaces with 400 Abrasive Mesh Size

Surface roughness values obtained after experiments with media prepared with three different abrasive concentrations and 400 abrasive mesh size are shown in Figure 7. The samples were run in cycle numbers 1, 3, 5, 10, 20, 50.



Figure 7. R<sub>a</sub> values which were measured perpendicular to flow of surfaces of Ti-6Al-4V



Surface roughness values decreased as a result of the values taken. Furthermore, the decrease in values relative to the different abrasive concentration followed a different course.

#### 3.2.2. Processed Surfaces with 240 Abrasive Mesh Size

The graphic obtained from experiments with media prepared with 240 mesh size is shown in Figure 8.



Figure 8. R<sub>a</sub> values which were measured perpendicular to flow of surfaces of Ti-6Al-4V

Better surface roughness values were obtained compared to experiments with 240 abrasive mesh size and 400 abrasive mesh size. Decreased  $R_a$  values were observed.

#### 3.2.3. Processed Surfaces with 180 Abrasive Mesh Size

Figure 9 shows a graphic of  $R_a$  values taken from Ti-6Al-4V surfaces, where experiments were carried out with 180 abrasive mesh size.



Figure 9. Ra values which were measured perpendicular to flow of surfaces of Ti-6Al-4V

A rapid reduction in surface roughness values were observed as a result of the measurements. The best results were achieved in this abrasive mesh size compared to other abrasive mesh sizes. A rapid decrease in  $R_a$  values was observed in the first cycles at a rate of 60% abrasive. The lowest  $R_a$  values



were measured. As with other abrasive mesh sizes,  $R_a$  values decreased with the increase in abrasive concentration and number of cycles.

In figure 10, SEM images taken from sample surfaces after experiments with media prepared with 180 abrasive mesh size are given.



**Figure 12.** 180 abrasive mesh size; 20%, 40%, 60% abrasive concentration SEM images of Ti-6Al-4V after AFM

After experiments with the media prepared in 180 abrasive mesh size, the white layer on the surface of Ti-6Al-4V was considerably reduced. The unproductive layer was removed from the first cycle by increasing the abrasive concentration.20% abrasive concentration is insufficient for Ti-6Al-4V in this abrasive mesh size. However, at 40% and 60% abrasive concentrations, the white layer has been completely removed. In fact, traces of abrasives have been observed on the surface along with the increasing number of cycles.

109

#### 4. Conclusions

Ti-6Al-4V, type of material frequently used in the aerospace industry, was produced by wire EDM method while the inefficient layer formed on the surface was removed by applying the AFM method. In short, if we explain the matter:

1. As the number of cycles increases in the AFM process, the MRR increases, thus the inefficient layer is removed, the  $R_a$  value decreases, and consequently the surface quality increases.

The International Conference of Materials and Engineering Technology

- 2. As the abrasive concentration of the media used in the studies increased, the white layer was removed more quickly from workpiece surfaces.
- 3. With the reduction in abrasive mesh size, the MRR increased. The inefficient layer is removed in fewer cycle numbers.
- 4. The white layer was removed in the first cycles, both by increasing the abrasive concentration and by decreasing the abrasive mesh size.

#### References

- 1. Ravi Sankar, M., V. Jain, and J. Ramkumar, Abrasive Flow Machining (AFM): An Overview. 2011.
- Duval-Chaneac, M.S., et al., Experimental Study on Finishing of Internal Laser Melting (SLM) Surface with Abrasive Flow Machining (AFM). Precision Engineering, 2018, 54: p. 1-6.
- 3. Przyklenk, K., Abrasive Flow Machining: A Process for Surface Finishing and Deburring of Workpieces with a Complicated Shape. **1987**. p. 123-138.
- 4. Sato, T., S. Wan, and Y. Ang, Study of Process Characteristics of Abrasive Flow Machining (AFM) for Ti-6Al-4V and Validation with Process Model. **2013**, Vol. 797. 411-416.
- 5. Kumar, S.S. and S.S. Hiremath, A Review on Abrasive Flow Machining (AFM). Procedia Technology, **2016**, 25: p. 1297-1304.
- 6. Uhlmann, E. and S. Roßkamp, Surface Integrity and Chip Formation in Abrasive Flow Machining. Procedia CIRP, **2018**, 71: p. 446-452.
- 7. Rhoades, L., Abrasive Flow Machining: A Case Study. Journal of Materials Processing Technology, **1991**, 28(1): p. 107-116.
- 8. Ali, P., et al., Hybrid Abrasive Flow Machining for Nano Finishing A Review. Materials Today: Proceedings, **2017**, 4(8): p. 7208-7218.
- 9. Williams, R.E., Investigation of the Abrasive Flow Machining Process and Development of a Monitoring Strategy using Acoustic Emission. **1993**.
- 10.Yadav, S., E.M. Singh, and P.B.R. Singh, Effect of Unconventional Machining on Surface Roughness of Metal: Aluminum and Brass-A Case Study of Abrasive Flow. **2015**, Vol. 2.
- 11.Cherian, J., D.J.I.J.o.E.T. Issac, and A. Engineering, Effect of Process Variables in Abrasive Flow Machining. **2013**, 3(2): p. 554-557.
- 12.Göv, K., Ö. Eyercioğlu, and M.V. Çakır, Hardness Effects on Abrasive Flow Machining. **2013**, Vol. 10. 626-631.
- 13.Kozak, J., K.P. Rajurkar, and N. Chandarana, Machining of Low Electrical Conductive Materials by Wire Electrical Discharge Machining (WEDM). Journal of Materials Processing Technology, 2004, 149(1): p. 266-271.



The International Conference of Materials and Engineering Technology

- 15.Göv, K., Havacılık ve Uzay Malzemelerinde Soğutma Sıvısının Elektro Erozyonla Delinen Deliklerin Performans Parametrelerine Etkisinin Deneysel İncelenmesi. Gazi Üniversitesi Mühendislik-Mimarlık Fakültesi Dergisi, **2017**, 32(1).
- 16.Göv, K., Ö. Eyercioğlu, and M.V. Çakır. Aşındırıcı Akışkan ile İşleme (AFM) Parametrelerinin Tel Erozyonla Kesilmiş Kalıp Yüzeyine Etkisinin İncelenmesi. TMMOB Makina Mühendisleri Odası Konya Subesi VI. Makina Tasarım ve Imalat Teknolojileri Kongresi. Konya, 2011.
- 17.Göv, K. and Ö. Eyercioğlu. Abrasive Flow Machining of TI-6AL-4V. in International Science and Technology Conference. **2017**.
- 18.Göv, K. and Ö. Eyercioğlu, Effects of Abrasive Types on The Surface Integrity of Abrasive-flow-machined Surfaces. **2018**, 232(6): p. 1044-1053.
- 19.Butola, R., et al., Optimization to the Parameters of Abrasive Flow Machining by Taguchi Method. **2018**, Vol. 5. 4720-4729.
- 20.Ibrahim, A., Studying Material Removal in Abrasive Flow Machining by using SiC. **2014**, Vol. 34204.
- 21.Seifu, Y., S.S. Kumar, and S.S. Hiremath, Modeling and Simulation: Machining of Mild Steel using Indigenously Developed Abrasive Flow Machine. Procedia Technology, **2016**, 25: p. 1312-1319.

### MICRO HOLE DRILLING OF AEROSPACE MATERIALS AND REGRESSION ANALYSIS

The International Conference

aterials and Engineering Technology

#### AYKUT AKBULUT<sup>1</sup> KURSAD GOV\*<sup>2</sup>

 <sup>1</sup>Gaziantep University, Faculty of Aeronautics and Astronautics, Aircraft and Aerospace Engineering Department, Gaziantep, TURKEY
 <sup>2</sup>Gaziantep University, Faculty of Aeronautics and Astronautics, Aircraft and Aerospace Engineering Department, Gaziantep, TURKEY

#### Abstract

In this paper, Electrical Discharge Micro Hole Drilling process was applied to specific aerospace material of Ti-6Al-4V. Hole-EDM drilling is a different type of EDM processes, i.e. Wire-EDM and Sink-EDM. Although Hole-EDM drilling uses the same principles as other EDM methods, a rotated hollow electrode and pumping of dielectric coolant fluid through the electrode tube are the two distinct features. This process has been alternatively used for producing fuel injectors, holes in turbine blades, cutting tool coolant holes, medical equipment, plastic mould vent holes, hardened punch ejectors and Wire-EDM starter points. In this experimental study, surface observation and the effects of machining parameters of arc on-time (Ton), arc off-time (Toff) and current (I) on performance parameters were studied. Experimental works were applied with single channel, cylindrical, brass, tabular electrode. Deionized water was used for coolant where was pumping inside the electrode at high pressure. The performance parameters were chosen as surface roughness (Ra), material removal rate (MRR), electrode wear ratio (EWR) which were occurred under the influence of the machining parameters were analysed. For the purpose of analysing, Taguchi method was consulted to design of experiments. Regression mathematical model was developed and analysis of variance (ANOVA) was implemented to figure out of the reliability of mathematical model results. The experimental results reveal that the 500µm brass electrode has comparatively better MRR and lower EW. According to ANOVA reliable mathematical models were developed with 0,9643, 0,9827 and 0,9201 R-sq% for Ø500µm successfully.

Keyword: Aerospace materials, Electrical discharge hole drilling, Electrical discharge machining

#### **1. Introduction**

Electrical discharge machining (EDM) is one of the methods where requires to process micro size holes on hard metals like titanium alloys[1-3] or would be hard to drill with conventional methods. These holes generally located on complex surfaces and bodies of parts which are widely used at Aerospace industries like turbine blades, also some specific automotive parts like diesel ejectors and dies [4, 5]. However, although wants to use EDM on every material, there is a criterion related validation. This reality requires that the materials used in the EDM must be electrically conductive. Because EDM uses a thermoelectric force to wear material from parts. This force produced via spark's heat energy. Sparks causing evaporation and melting on part local area [6]. At that point temperature raise to 8000-12000°C approximately [7]. At the same time wreckages are transferred to the outside from hole perimeter by using high pressure water. These sparks are also the main cause of some problems such as electrode removal rate, material removal rate and surface roughness which is focused on this article. The character of hole is easily affected from sparks. The spark behaviour is not controlled easily and it causes irregularity on part. According to studies on EDM, these problems are linked with a lot of parameters such as discharge voltage, current, turn speed, pulse span[1-3, 8], electrode [9-11], workpiece characteristics. On the other hand, EDM has an advantage



the effects of peak current, pulse on time and rotational speed of the tool electrode as optimization of machining parameters. Workpiece has chosen as Ti-6Al-4V with brass electrode and found that higher peak current leads to higher discharge energy and raise of MRR. Also recognized that with rotating electrode higher MRR is reached. Eyercioglu et al [13] evaluated the effect of Ø2 mm electrode which has single hole and brass material on the cold working tool steel DIN 1.2080 and results show us responses were implemented such as dimensional accuracy, better finish and reasonable machining time. Gov [14] researched the effect of dielectric liquid temperature on hole geometries and reached the better surface roughness was linked with decrease of liquid temperature also improved hole accuracy. Gov [15] approached from different ways to observe the effects of different types coolant liquids on EDM responses and reached deionized water is better for Ti-6Al-4V compound, the other hand kerosene and normal water results better for Inconel 718 when compared with deionized water. Gov [16] studied on the different amount of dissolved oxygen and focused on the surface roughness and machining time to increase parameters. And experiments show increase of dissolved oxygen in the coolant improved these parameters as low surface roughness and fast machining time. Some other EDM hole drilling studies were studied by Gov et.al.[17-19] and the effects of EDM parameters on hole drilling process were carried out.

#### 2. Experimental Setup

Experiments were implemented with JS EDM AD-20 (Figure 1) type hole electrical discharge machine which is available in Mechanical Engineering Department of Gaziantep University.



Figure 1. Electrical Discharge Machine

The sample was chosen as Ti-6Al-4V (Table 1) due to widespread application on aerospace and

<b>Table 1.</b> Chemical Compositions of Materials (wt. %)						
Ti-6Al-4V						
Ti	89,464	0	0,18			
Al	6,08	С	0,02			
V	4,02	Ν	0,01			
Fe	0,22	Н	0,0053			



medical sectors. Materials prepared as 5x10x20 mm blocks (Figure 2) which were cut by wire electro discharge machining. The faces which are opposite of each other on samples were ground by using 320 to 1000 size emery papers gradually and polished by using 1  $\mu$ m diamond



Figure 2. 3D Workpiece Illustration

suspension before drilling. Single channel electrodes with different sizes as  $\emptyset 300 \mu m$ ,  $\emptyset 400 \mu m$  and  $\emptyset 500 \mu m$  (Figure 3) were used for making holes which were positioned on the centre of the



Figure 3. 300 and 400  $\mu$ m Diameter Brass Single Channel Electrodes

opposite polished faces vertically and drilled with depth of 10 mm. After process was completed, workpieces were appear as shown in Figure 4.



Figure 4. (a) Top and (b) side views of workpiece

The electrode properties are given in (table 2). As coolant liquid deionized water was chosen and implemented to all experiments regularly. The operations were made considering the results of preliminary experiments.

Electrode Material	Brass
Melting point (°C)	900-940
Electrical resistivity (ohm-cm)	25659
Thermal conductivity (W/m-°K)	159
Specific heat capacity (J/g-°C)	0.380

Table 2. Property	ties of The	Electrode	Material
-------------------	-------------	-----------	----------

These experiments were performed with setting the parameters (Table 3) as current (I), pulse on time (T<sub>on</sub>), pulse off time (T<sub>off</sub>) and remained constant the other parameters to observe the changes on the output parameters. Output parameters were focused on surface roughness, material removal rate and electrode wear ratio.

Table 3. Machining Cond	ditions
Discharge current (I)	3,4-8,8 A
Pulse-on time (Ton)	35-44 µs
Pulse-off time (Toff)	20-26 µs
Capacitance	104 µF
Voltage	29 V
Coolant liquid	deionized water
Dielectric flushing pressure	100 bar
Electrode rotation	200 rpm
Polarity of tool electrode	Negative (-)

#### 2.1 Design of Experiments

UWE Bristol

With the DOE method, the amount of test required to be done normally, is reduced considerably, thus eliminating a large workload, which can lead to more test results in a short time. In this context, it was decided to do experiment design with Taguchi method (Table 5) and applied as machining parameters they were selected that Current, Pulse on time and Pulse of time as shown in table 4 with 3 factor, 3 level experiment parameters A, B, C and level 1, 2, 3 respectively.

	Table 4. Proc	ess Parameters	5
	А	В	С
levels	I (ampere)	Ton (µs)	$T_{off}(\mu s)$
1	3,4	35	20
2	8,2	38	23
3	8,8	44	26

Experiment no	L9 Taguchi Design		Machine Parameters		Parameter Values				
	А	В	С	Ι	Ton	$T_{\rm off}$	I(A)	$T_{on}(\mu s)$	$T_{\text{off}}(\mu s)$
1	1	1	1	4	7	7	3,4	35	20
2	1	2	2	4	8	8	3,4	38	23
3	1	3	3	4	9	9	3,4	44	26
4	2	1	2	5	7	8	8,2	35	23
5	2	2	3	5	8	9	8,2	38	26
6	2	3	1	5	9	7	8,2	44	20
7	3	1	3	6	7	9	8,8	35	26
8	3	2	1	6	8	7	8,8	38	20
9	3	3	2	6	9	8	8,8	44	23

Table 5. L9 Taguchi Experiment Design for One Electrode Dimension
---

This method is one type of design methods. Taguchi method focus on design the development of superior performance, designs to deliver quality in comparison with statistical process control, which tries to control the factors that unfavourably affect the quality of product. Table 5 repeated for all electrode dimension separately. With each electrode, were implemented 9 experiments and consequently 27 experiments were implemented in this article.

e International Conference

Materials and Engineering Technology

#### **2.2 Surface Roughness Measurements**

Different types of fluids pass through the created holes according to their working condition. Each fluid is significantly affected by the roughness of the surface through which passes inside. At this point, if the application is to be developed, the surface roughness should be considered. Minimizing the surface roughness will turn into a benefit on fluent characteristics which flows inside holes like air, fuel etc. Machining parameters are playing active role on the surface roughness. The experiment surfaces were measured with Mitutoyo SJ 401 stylus type surface roughness measuring machine, evaluation length was chosen as 4 mm respectively and all measurements applied with 3 repetitions.



Figure 5. Roughness Measurement

#### 2.3 Material Removal Rate

Material removal rate was calculated with weighing of workpieces. Workpieces were weighted before drilling operations and after drilling operation regularly. Each part of workpiece's weight recorded separately then weights were summed to understand how much weight loses. In order to understand effect of parameters, weights were rated per minute (Equation 1). All weight measurements were performed with SHIMADZU AUX220 respectively and electronic timer were used to measure drilling time.

$$MRR\left(\frac{mg}{min}\right) = \frac{\text{Initial weight of material - final weight of material}}{\text{Machining Time}}$$
(1)

#### 2.4 Electrode Wear Ratio

During the EDM operation, spark which occurred on head of electrode reached to high temperatures. These sparks melt the workpiece's surface and separate material via pressurized water. While operating this process, electrode also melts and remove tiny parts from head of electrode at the same time. This situation named as electrode wear. It's important for improve the EDM process performance. Electrode wear effects the performance of EDM with inverse ratio. Small electrode wear ratio means low electrode cost and low manufacturing cost also. To understand that, electrode weights were measured before and after operation. Obtained data used to calculate EWR as shown in equation (2).



(2)

# 3. Experimental Results3.1 Effects on Surface Roughness

Surface roughness can be described as deformation on surface under working condition of machining. These conditions occur with the result of sparks which were producing on the tip of electrode. These sparks melt the material and remove them by coolant liquid. The molten materials create some cavities on the surface behind them. These geometric irregularities are unwelcome characteristic results of EDM. According to the result of surface roughness inspection (Figure 6-8), when current increases roughness decreases also.



Figure 6. Surface Roughness (Ra) vs I

The worst roughness values observed when current parameter chosen as 3,4 A and best result obtained at 8,8 A. If we only observed  $Ø500 \mu m$  holes, we can say the effect of current is not significant on roughness. Pulse-on time also not significant on surface roughness even if change the diameters, as shown in (figure 7). Only for  $Ø300 \mu m$  was small change occurred between 8,2 A and 8,8 A.



Figure 7. Surface Roughness (Ra) vs Ton

When we observed the pulse-off time effect (Figure 8), general view occurred irregular and independent from each other. Besides, we reached Pulse-off time is not significant on roughness also.



Figure 8 Surface Roughness (Ra) vs Toff

#### 3.2 Effects on MRR

EDM process works with the principle of melting the workpiece's contact surface with electrode and remove the molten material from the workpiece. The amount of molten material is figure out MRR. Generally, MRR shows parallel behaviour with discharge current. When discharge current increase, it means the energy of spark increase and it causes more melting and vaporizing on the workpiece. According to figure 9, there was a slight increase in the MRR for  $\emptyset$ 300 µm and considerable rise at  $\emptyset$ 500 µm and linear increasing observed at  $\emptyset$ 400 µm. Ampere highly effected on MRR considerably.



Figure 9. MRR vs I

The effect of pulse on-time ( $T_{on}$ ) on MRR was shown in Figure 10. The increment of  $T_{on}$  caused some changes on MRR for all electrode diameter. While increasing  $T_{on}$  from 35 µs to 38 µs, MRR decreased for Ø500 µm. At the same time, MRR increased for Ø400 µm. That means there is no logical relation between  $T_{on}$  and MRR.



Figure 10. MRR vs Ton

The effect of Pulse off-time ( $T_{off}$ ) on MRR was shown in Figure 11. MRR decreased slightly when the  $T_{off}$  was increased from 23 µs to 26 µs for Ø500 µm. Also increment of  $T_{off}$  didn't affected on MRR for Ø400 µm and Ø300 µm. Results keep going on horizontally. So, the increase in  $T_{off}$  not significant on MRR as shown in figure 11.



Figure 11. MRR vs Toff

#### 3.3 Effects on EWR

Sparks on the tip of electrode erodes the workpiece and remove parts from materials. At the end of that there is occurs hole on the material. Beside melting workpiece, electrode is consumed by melting due to high volume energy expand on electrode tip. Electrode wear is related with the electrical parameters of EDM and electrode materials [20]. In this article capacitance (C) and voltage (V) were taken constant and the effects of current, pulse on time and pulse off time on the electrode wear were investigated. For all different diameters, ampere(I) had significant effects on EWR obviously (Figure 7). For  $\emptyset$ 500 µm, EWR was dramatically increase at the range of 3,4 A-8,2 A and continuous with slight rise. The other side,  $\emptyset$ 400 µm was increase with linear and constant slope. For  $\emptyset$ 300 µm, firstly increase the EWR with rising ampere, but after reach the maximum value, EWR decreased again.



Figure 12. EWR vs I

When the effect of  $T_{on}$  was observed, for all diameter pulse-on time at 38 µs was same mean values, but when it was increase, slightly down EWR except Ø500 µm.



Figure 13. EWR vs Ton

 $T_{off}$  effects EWR slightly for all diameters. While drilling with Ø500 µm hole, changing the pulseoff time had small effect on EWR response. Between 20 µs and 26 µs EWR decreased slightly. Ø300 µm slope was increased and decreased again. So EWR and pulse-off time not proportional at Ø300 µm. Unlike others, Ø400 µm slightly increase with increasing pulse-off time. Even Ø400 µm has bigger dimensions according to Ø300 µm, general electrode consumptions less than Ø300 µm.


Figure 14. EWR vs Toff

## 3.4 Regression analysis

Regression analysis was applied to construct a model to predict experiment results. The model fit is usually linear, sometime non-linear models such as log linear models are also constructed. In this work multiple regression analysis was implemented for better understanding of the effects of the input parameters on the response parameters and a linear regression equation (3) is also figure out for the prediction of the output parameters. Therefore, we used MINITAB software and followed the consequent process. First, a linear model was developed on MINITAB to analyse the data of  $\emptyset$ 500 µm as demonstration. The other experiments  $\emptyset$ 400 µm and  $\emptyset$ 300 µm which were experimented not shown here with details. Just  $\emptyset$ 500 µm experiment results revealed. The fitness characteristic is shown by the following as:

$$Response \ Parameter = b_0 + b_1 A + b_2 B + b_3 C + (\varepsilon) \tag{3}$$

where  $b_1$ ,  $b_2$ , and  $b_3$  were estimate of the process parameters, and  $\varepsilon$  is error. An empirical equation is then derived to illustrate the functional correlation between response parameters and process parameters. A, B, and C as follows:



Figure 15. Comparison of mathematical model and experiment results of MRR for Ø500µm

---MRR(Exp)

•MRR(Regression)

 $EWR = -2,85 + 0,7951 \ I + 0,0839 \ T_{on} - 0,1151 \ T_{off} + (\epsilon) \\ R\text{-sq} \ 98,27\%$ 

(5)



Figure 16. Comparison of mathematical model and experiment results of EWR for  $Ø500\mu m$ Ra = 20,56 - 1,707 I + 0,071 T<sub>on</sub> - 0,113 T<sub>off</sub> + ( $\epsilon$ ) (6) R-sq 92,01%



Figure 17 Comparison of mathematical model and experiment results of Ra for Ø500µm

First, the analysis of variance (ANOVA) of the regression model is considered for the reliability of the formulation and is considered to find the relationship between the variables within the statistical model. The experimental data were used to develop linear models, and analysis of the models was figure out through ANOVA.

Ø500µm		Ø400µm		Ø300µm	
MRR	96,43	MRR	98,17	MRR	97,94
EWR	98,27	EWR	98,64	EWR	91,90
Ra	92,01	Ra	88,24	Ra	91,44

Table 6. R-sq(%) for  $Ø500/400/Ø300 \ \mu m$  ANOVA results

As a consequential parameter  $R^2$ , which is called R-sq, is the correlation coefficient and should fall between 0.8 and 1. In this study,  $R^2$  was found to be 0,9643, 0,9827 and 0,9201 for Ø500 µm holes MRR, EWR and R<sub>a</sub> respectively. As a result, the multiple regression model for response parameters matches very well with the experimental data.

# 4. Conclusions

In this article, micro hole EDM process investigated under condition which are designed with Taguchi method and output's reliability has been proven with Analysis of Variance. The surface roughness, MRR and EWR are chosen as experiment responds. Input parameters, which were combined with Taguchi, were current, pulse on time, pulse off time respectively. From the analysis, the following may be concluded.



The International Conference of Materials and Engineering Technology

- Even tough minimal pulse off-time is required to increase MRR, a sufficient amount of pulse off-time is necessary to remove the molten metal between the electrode and the workpiece by the pressurized water.
- The logical response function designed by regression analysis and analysed with ANOVA, experiment results proved as reliable with these results for  $\emptyset$ 500/400/300 µm electrode dimension respectively 0,9643, 0,9827 and 0,9201 terms of R<sup>2</sup>.

#### **References:**

- 1. Lin, M.Y., et al., Use of the grey-Taguchi method to optimise the micro-electrical discharge machining (micro-EDM) of Ti-6Al-4V alloy. Vol. 28. 2014. 1-8.
- 2. Meena, V. and M. Azad, Grey Relational Analysis of Micro-EDM Machining of Ti6Al4V Alloy. Vol. 27. 2012. 973-977.
- 3. Yadav, U. and V. Yadava, Experimental Investigation on Electrical Discharge Drilling of Ti-6Al-4V Alloy. Vol. 19. 2015. 515-535.
- 4. Diver, C., Micro-EDM drilling of tapered holes for industrial applications. Vol. 149. 2004.
- 5. Y. Li, Z., et al., State-Of-Art, Challenges, and Outlook on Manufacturing of Cooling Holes for Turbine Blades. Vol. 19. 2015.
- 6. Ho Huang, S., F. Yuan Huang, and Y. Biing Hwa, Fracture strength analysis of micro WC-shaft manufactured by micro-electro-discharge machining. Vol. 26. 2005. 68-77.
- 7. Ho, K.H. and S. Newman, State of the art electrical discharge machining (EDM). Int J Mach Tools Manuf Des Res Appl. Vol. 43. 2003. 1287-1300.
- Mohri, N., et al., Electrode Wear Process in Electrical Discharge Machinings. Vol. 44. 1995. 165-168.
- 9. Amorim, F. and W. Weingaertner, The influence of generator actuation mode and process parameters on the performance of finish EDM of a tool steel. Vol. 166. 2005. 411-416.
- 10.Kurnia, W., et al., Analytical approximation of the erosion rate and electrode wear in micro electrical discharge machining. Vol. 18. 2008. 085011.
- 11.T Richardson, M. and Y. B Gianchandani, Achieving precision in high density batch mode micro-electro-discharge machining. Vol. 18. 2008.
- 12.Pradhan, B., et al., EDM with rotational electrode for machining micro-holes in Ti6Al4V. Vol. 89. 2009. 1-8.
- 13.Eyercioglu, O., M. V. Cakir, and K. Göv, Influence of machining parameters on the surface integrity in small-hole electrical discharge machining. Vol. 228. 2013. 51-61.
- 14.Göv, K., The effects of the dielectric liquid temperature on the hole geometries drilled by electro erosion. Vol. 92. 2017.
- 15.Göv, K., Havacılık ve Uzay Malzemelerinde Soğutma Sıvısının Elektro Erozyonla Delinen Deliklerin Performans Parametrelerine Etkisinin Deneysel İncelenmesi. Vol. 32. 2017.
- 16.Göv, K., Investigation of the effects of the dissolved oxygen in the coolant on the hole geometries drilled by electro erosion. Vol. 31. 2016. 231-239.
- 17.Gov, K., Experimental Investigation of the Effects of Electrodes on EDM Hole Drilling Process. Journal of Polytechnic, 2017. 20(2).
- 18.Gov, K., Influence of the Coolant on Performance of Electro Discharge Hole Drilling. Journal of Polytechnic, 2017. 20(1): p. 191-196.



- 19.Göv, K., A Comparative Investigation of the Effects of Brass And Copper Electrodes in Hole-EDM Process, in 3th International Conference on Advanced Technology & Sciences. 2016: konya.
- 20.Hasçalık, A. and U. Çaydaş, Electrical discharge machining of titanium alloy (Ti-6Al-4V). Vol. 253. 2007. 9007-9016.



# USING NANO FIBERS FOR ACOUSTIC INSULATION IN AEROSPACE PARTS

# BİRKAN BAĞLAMA<sup>1</sup>, KÜRŞAD GÖV\*<sup>1</sup>, EYÜP YETER<sup>1</sup>

<sup>1</sup>Gaziantep University, Aeronautics and Aerospace Faculty, Aircraft and Aerospace Engineering Department, Gaziantep, TÜRKİYE.

#### Abstract

Nanotechnology refers to a field whose theme is the control of matter on an atomic and molecular scale. Generally, nanotechnology deals with structures of the size 100 nanometres or smaller and involves developing materials or devices within that size. Nanofibers are defined as fibers with diameters approximately 100 nanometres. They can be produced by interfacial polymerization and electrospinning. Electrospinning is a straightforward and simple method to produce one-dimensional (1D) novel structures with tuneable fiber diameter on nanoscale. It can transfer numerous conventional materials into 1D nanostructure in an easy way. Both bare polymers and polymer composites with metal salt precursors are possible to be made into 1D nanofibers. The Electrospinning technique can also be scaled up for a wide range of applications including filtration, energy storage and regenerative medical treatment due to the interesting properties of 1D nanostructure. In this paper, the production techniques of the Nano materials will be discussed. The application areas of high performances Nano materials will be explained especially in the acoustic insulator application for aerospace components.

Keywords: Nano materials, Electrospinning, Acoustic insulation, Aerospace materials

# **1. Introduction**

Nanotechnology is the management issue on a nuclear, sub-atomic, and supramolecular scale. the foremost prompt, extensive depiction of technology alluded to the particular mechanical objective of specifically dominant iotas and particles for the creation of macroscale things, to boot currently alluded to as atomic technology [1].

Indeed, even before the term applied science was used by logical circles, the thoughts and ideas with regard thereto were a bit of a discussion entitled "There's many areas at the Bottom" by scientist Feynman. This discussion was given at associate yank Physical Society meeting that was command at the Calif. Institute of Technology on Gregorian calendar month twenty-nine, 1959. In his discourse, Richard Phillips Feynman mentioned the method that individual particles and atoms would be controlled and used by researchers. regarding 10 years once the very fact, another teacher named Norio Taniguchi begat the term applied science, and this, as it were, prompted the development of the checking burrowing magnifying instrument through that researchers may watch and management singular iotas, prompting the start of current applied science [2].

To begin with, nanotechnology can be depicted as science, building, and innovation that is directed with the nanoscale, and nanoscience and nanotechnology, applying to a great degree little things, can be profited in a wide range of research zones including science, material science, and materials science [2].

Various preparing methods, for example, drawing, layout amalgamation, stage partition, self-get together, electrospinning, and so forth have been utilized to plan polymer nanofibers in recent years [3]. It is conceivable to create nanofibers with measurements going from a couple of nanometres to a couple of hundred manometers because of the most recent advancements in electrospinning. The method can without much of a stretch be utilized in the research facility and can be scaled up to a mechanical procedure. Electrospinning of nanofibers from polymer arrangements or melts has been a focal point of enthusiasm as they have numerous potential applications [4].

#### 2. Materials and Methods

Nanofibers are very thin solid fibers with a diameter of less than 100 nm, big surface subject per mass unit and small pore size. Because of the normal houses of the electrospinning method, which will control the positioning of polymeric fibers on an intention, intricate and regular third-dimensional nanofibers, can also be fashioned. Very small-scale composite fibers can be made by way of electrospinning from a combo of inflexible rod and flexible polymer. Electro spun nanofibers can be used to build very small buildings in special homes akin to nanotubes and nanowires. In any other case, depending on the polymer resolution used durability, weight, porosity and surface functionality, and so on. A vast range of fabric homes will also be bought. Electro (fiber-reinforced) bending process presents the capacity to lock together polymers, fibers and particles in different constructions to provide very skinny layers. Minor particles that are insoluble in solution can be added and protected in dry nanofibers [5, 6].



Figure 21 Electrospun Nanofibers [7]

Not like conventional fiber bending approaches that may produce polymer fibers at diameters up to the micrometre range, a process can produce polymer fibers in the nanometre range of diameters using electrostatic bending or 'electrospinning' methods. Electrospinning is the method used to combine fibers with diameters smaller than 100 nm, to produce new and efficient fibers. This electrodeposition approach makes use of an excessive voltage electric subject to form dry fibers from the polymeric solution circulation, which is carried out through an awfully small-scale syringe [5].

Technology



Figure 22 Electrospun nanofibers on a polyester substrate

Despite the fact that the electrospinning method has not been identified for many years, the first step used to be given to Formhals [8] in 1934. Polymeric nanofibers acquired via electrospinning method have begun to be used so much in recent years. In 1990, Reneker and Chun reinterpreted this technological interest, and in 1996, it was possible to electro spin a large group of polymer solutions. Larrondo and Manley conducted a study on polymer melt in 1981. In general, electron spinning can also be applied to a large group of polymers, electrical conductivity and photonic polymer solutions. The polymers such as biological polymers, electrical conductivity and photonic polymer solutions. The excessive specified floor discipline and the small pore measurement of those fibers make them available for a broad range of applications. Through instance, 100 nm nanofibers have a geometric floor discipline ratio of about one hundred m2/g. One more effect of the usage of electro spin fibers is that the fibers can distribute or hold electrostatic costs relying on the electrical homes of the polymer options. Bills will also be neutralized via electric fields and the electrical forces of the fibers are affected by the polarity of the utilized voltage. Nanofibers have additionally served as a hyperlink between the nanoscale and the macroscale world due to the fact that their diameters are within the nanometre style and their lengths are kilometres [5, 9].

Electrospinning is the transfer of the solution onto the collector by spraying an area with an electric current to form fine fibers. The measurements of these fibers will be in nanometre size and have wide application areas such as filtration, composite materials, medical sheet and so on [10]. The electric spinning method is schematically shown in figure 1. Electro spinning has emerged from research on liquids in the field of electricity. First, William Gilbert made observations of a liquid that interacted under electric current in 1600. He discovered that when a drop of water is held at a suitable distance on a dry surface, a base is pulled. William Gilbert defined that the electrostatic forces towards the outside anxiety of the liquid brought about it to form on the drop surface of the electrical discipline. After this finding, first 1745 was electro-sprayed to produce a spray [11].

The electrospinning gadget contains a polymer resolution or solution, an excessive-voltage energy provides, a capillary tube (needle or pipette), a pump, and a collector. In a solution-centered electrobending system, a polymer solution is utilized to the tip of the tube. If the electric drive just is not utilized, the drop from the top drops to gravity considering the fact that it is going to be uncovered to gravity. If excessive electric force is utilized to the polymer solution, a high voltage difference will arise between the solution within the pumping and a grounded collector. This voltage change can intent the solution to turn out to be a conical protrusion. That is called the Taylor theme. The increase in utilized electrical energy, jet initiation, electrostatic drive, occurs right away after the skin tension of the polymer solution exceeds the skin anxiety of the Taylor. On this case. The answer will enter the jet electrical area. After step one, the jet will emerge as unstable and field itself to an excellent-pierced pushing force and bending motion. After the solution is evaporated, the remaining dry or semi-nanofibers are fashioned and deposited on the grounded electrode. The elemental scheme of electrospinning is shown in Fig 1 [12].

The International Conference of Materials and Engineering Technology



Figure 23 Basic electrospinning apparatus [12]

Nanofibers; they have many different morphologies such as porous, core-sheath and side-by-side structures. Extremely thin electro spun nanofibers are very useful in a wide variety of applications and many studies have been done on nanofibers. However, new applications for nanofibers are constantly being investigated. The main application areas of electro spun nanofibers are shown in Table 1 [13].

Table	2.Applications	of Electro	spun	Nanofibers

Filtrations
Affinity membranes and recovery of metal ions
Tissue engineering scaffolds
Wound healing
Release control
Catalyst and enzyme carriers
Sensors
Energy storage

Filters are broadly used for navy, home and industrial purposes to separate air, liquid or liquid resources. The electro spun nanofiber membrane supplies a tremendous broaden in filtration effectivity in comparison with other products in permeability. In comparison with the nylon electro spun membrane (thickness a hundred  $\mu$ m, pore dimension 0.24  $\mu$ m) and thicker and extra effective high efficiency particulate air filtration (HEPA) (thickness 500  $\mu$ m, pore measurement 1.7  $\mu$ m), the high-quality nanofiber membrane exhibited a better filtration efficiency than the HEPA filter were observed [14]. The nanofibers are a scaffold used as a support for ultrafiltration to separate water/oil emulsions. Other experiences regarding filtration applications comprise the results of electrospinning process parameters on filtering tactics, fiber morphology, and pore constitution [15].

Chemical precipitation, ion alternate, membrane separation, electrochemical cure, adsorption and many others. More than a few techniques were used for the removing and restoration of heavy metals from aqueous options similar to water. Functionalized nanofibers can collect a solution of metal ions or small molecules. The electrospinning technique, an easy and versatile approach, has been generally applied to the creation of nanofibers. Electro spun nanofibers are known to have properties such as higher porosity, smaller pore size and, most importantly, very large specific surface compared to conventional pores. Nanofibers are widely applied as tissue engineering, drug delivery, sensor construction, protective clothing, fine filtration processes. High precise floor nanofibers make better adsorbents with better swelling charges and capacity than other substances akin to resins, foams and natural fibers [16].

The International Conference of Materials and Engineering Technology

UWE

Tissue engineering involves the use of remodelled living cells. This effort consists essentially of three core technologies: Cell technology, scaffolding technology and integration technology. [17, 18].

At wound treatment, additional physique fluids come out from the wound area; cure features decontaminate the exogenous microorganism, beef up the appearance and many times speed up the medication method. For this service, a wound dressing material grants a bodily barrier for a wound, but have to be permeable to moisture and oxygen. For a full-thickness dermal harm, the adhesion and integration of a "synthetic dermal layer" inclusive of a 3D tissue scaffold with excellent-cultured dermal fibroblasts will vastly support re-epithelization. The electro spun nanofiber membrane is an efficient candidate for wound dressing for that reason of its certain houses: the vastly porous membrane structure and good-interconnected pores are notably predominant for the wound to leak out of the fluid; The small pores and really high distinctive surface subject now not most mighty ward off the invasion of the exogenous microorganisms, however moreover support to control fluid drainage; in addition, the electrospinning process presents an easy system so to add drugs to nanofibers for any feasible clinical medicine and antibacterial intent [13].

Controlled release technique is an effective procedure for the administration of medicines in medical treatment. The toxicity and side effects that can be caused by controlled release medicines can be minimized and patient comfort can be increased [19]. Electro spun nonporous; a skills drug provider has gained many advantages on this regard. It is as a substitute handy to apply drug loading using electrospinning, and the high voltage used within the electrospinning approach has little outcomes on drug endeavour. The high-targeted surface discipline and the fast diffusion barrier dimension provide the nanofiber drug approach a larger entire liberate fee than the majority material. The release profile will also be precisely controlled with the support of the modulation of nanofiber morphology, porosity, and composition [13].

Nanofibers are used in chemistry and biology as a carrier for the catalyst, to maintain high catalytic activity, to improve the compatibility and lifetime of the catalyst, and to simplify the reaction process. Nano-fibers having a high surface area and high permeability to action can be suitable materials for effective catalyst carriers due to their structure being an inert porous material. Electro spun nanofibers can provide a wide and active surface area when used as a catalyst carrier seal, thus enhancing catalytic activity [13].

Sensors made using nanofibers are widely used in many areas such as environmental, industrial, and medical and defence applications. Sensors should have high sensitivity, selectivity and reliability, as well as a small size, low production cost and multiple functions [20]. The electro spun nanofiber is very effective in meeting the requirements such as the sensitivity, porosity, etc. that the sensors need in terms of their properties [13].

Nano-fiber materials are the most common name for use in component parts of energy conversion and storage devices. Recently, much effort has been made to develop nanostructured materials synthesized by different methods including electrospinning [21]. Polyvinylidene fluoride (PVdF) electro-spun nanocomposite membranes have been specifically investigated for the application of lithium batteries [22, 23].

129

Electro spun nanofibers have been studied in the strengthening of polymers. Electro spun nonporous mats can cause mechanical closure between nanofibers due to their active and effective properties [24]. Other studies have shown that the nanofibers have an excellent effect on sound absorption. Elmarco, a renowned nanofiber technology company, has patented an electro spun Nano fiber material with more effective and powerful sound absorbing properties, with just one third of the load of usual sound insulation substances. It has been seen that in a giant frequency range, especially in low frequency sounds under a thousand Hz, the sounds are absorbed [13].

The International Conference of Materials and Engineering Technology

Aircraft noise are produced by aircraft during runway, taxi, take off, flyover, or landing, including aircraft propeller and jet engine [25]. Noise studies on general aviation plane show that the noise stage alterations with aircraft and motor forms. Noise discount is a set of tactics for reducing unwanted noise/noise in the aircraft. It is known that any fabric that allows the passage of air will function as a sound/sound absorber. The rubbing of an acoustic vigour passing by way of an absorber reduces the volume by using transferring sound to the warmth vigour. Thicker substances in most cases have greater porosity and surface areas, which can be immune to air molecules and hence stronger in absorbing, sound. Nonetheless, thicker absorbers will increase the total weight of the aircraft and slash gas efficiency [25-29]

The noise influences the flight wellness (eg, hearing impairment, high blood stress, dizziness, headache, etc.), of the crew and the passengers and threatens flight performance and safeguard ultimately. Therefore, a multidisciplinary study strategy is required to receive appropriate noise levels in airplanes and other transport vehicles [27-29].

Most often the soundest absorbers used to control the aircraft within noise are porous, membrane, hollow, perforated panel and composite absorbers within the sort of open cell phone foam, fiberglass, mineral fiber wool, acoustic roof tiles and wooden fibers. Porous absorbers comprise mineral wool, chipboard, plastic foam, cloth, felt, carpet, cotton and certain acoustic plaster. Kevlar can be utilized in the development of composite add-ons for very powerful porous fibers and puncture resistant panels, cloths and bulletproof vests in the form of ropes or fabrics. Nomex is both in fiber type (50-a hundred  $\mu$ m) and in sheet form. It is also used as a fabric (eg, frame overlay) anywhere warmness and flame resistance is required. Both of these aramids are viewed as noise abatement for aircraft [25].

#### 3. Results and Discussion

In this study, nano fiber production methods were investigated to be used in sound permeability. There are multiple nano fiber production methods. Melting, interface polymerization and electrospinning. In the electro-spinning method, the physical structure of the material to be produced by increasing the distance between the collector and the needle can be changed, while in other methods, the system needs to be restructured for physical changes. In addition, high production speed and low cost make electro spinning the most effective production method in nano fiber production. When the sound absorption coefficients of the nanofibers produced by electro spinning method were measured with the help of impedance tube, it was seen that the results were much higher than the other composite materials.

**4. Conclusions:** The theoretical and practical benefits of using nano fibers in composite filter media were investigated and a method for nano fiber production was discussed. Nano fibers can provide an improvement in filter efficiency and a significant increase in noise insulation without a significant increase in filter pressure drop. It has been proven that sound insulation materials used in flying vehicles can be produced at a cheaper and at a faster time.

#### **References:**

1. Hall, J.S. An electroid switching model for reversible computer architectures. in Proceedings of Physics of Computation Workshop, Dallas Texas. 1992. Citeseer.

The International Conference of Materials and Engineering Technology

2. <u>https://www.nano.gov/nanotech-101/what/definition</u>.

- 3. Wang, H., et al., Insulin-like growth factor binding protein 2 enhances glioblastoma invasion by activating invasion-enhancing genes. 2003. 63(15): p. 4315-4321.
- 4. Karakaş, H.J.I.T.U., Textile Technologies and D. Faculty, Electrospinning of Nanofibers and There Applications. 2015.
- 5. Reneker, D.H. and I.J.N. Chun, Nanometre diameter fibres of polymer, produced by electrospinning. 1996. 7(3): p. 216.
- 6. Asst. Prof. Dr.Kürşad Göv, A.P.D.I.b.G., Asst. Prof. Dr. Eyüp Yeter, Birkan Bağlama. Production of Nano Materials for Acoustic Insulation for Aerospace Components. in II. Uluslararası Multidisipliner Çalışmaları Kongresi. 2018. Adana: Adana 2018.
- 7. Graham, K., et al. Polymeric nanofibers in air filtration applications. in 5th annual technical conference & expo of the American Filtration & Separations Society, Galveston, Texas. 2002.
- 8. Anton, F., Process and apparatus for preparing artificial threads. 1934, Google Patents.
- Larrondo, L. and R.J.J.o.P.S.P.P.E. St. John Manley, Electrostatic fiber spinning from polymer melts. I. Experimental observations on fiber formation and properties. 1981. 19(6): p. 909-920.
- 10.Mirjalili, M. and S.J.J.o.N.i.C. Zohoori, Review for application of electrospinning and electrospun nanofibers technology in textile industry. 2016. 6(3): p. 207-213.
- 11.Gilbert, W., De magnete. 1958: Courier Corporation.
- 12.Wang, Z.L.J.A.n., Triboelectric nanogenerators as new energy technology for self-powered systems and as active mechanical and chemical sensors. 2013. 7(11): p. 9533-9557.
- 13.Fang, J., et al., Applications of electrospun nanofibers. 2008. 53(15): p. 2265.
- 14.Huang, Z.-M., et al., A review on polymer nanofibers by electrospinning and their applications in nanocomposites. 2003. 63(15): p. 2223-2253.
- 15.Lala, N.L., et al., Fabrication of nanofibers with antimicrobial functionality used as filters: protection against bacterial contaminants. 2007. 97(6): p. 1357-1365.
- 16.Hu, X., et al., Electrospinning of polymeric nanofibers for drug delivery applications. 2014.185: p. 12-21.
- 17.Nerem, R.M. and A.J.T.e. Sambanis, Tissue engineering: from biology to biological substitutes. 1995. 1(1): p. 3-13.
- 18.Katta, P., et al., Continuous electrospinning of aligned polymer nanofibers onto a wire drum collector. 2004. 4(11): p. 2215-2218.
- 19.Yih, T. and M.J.J.o.c.b. Al-Fandi, Engineered nanoparticles as precise drug delivery systems. 2006. 97(6): p. 1184-1190.
- 20.Mohr, G.J., et al., Development of chromogenic reactands for optical sensing of alcohols. 1998. 49(3): p. 226-234.
- 21.Lee, J.D., et al., Exact post-selection inference, with application to the lasso. 2016. 44(3): p. 907-927.
- 22.Lee, J.S., et al., Role of molecular weight of atactic poly (vinyl alcohol)(PVA) in the structure and properties of PVA nanofabric prepared by electrospinning. 2004. 93(4): p. 1638-1646.
- 23.Gao, K., et al., Crystal structures of electrospun PVDF membranes and its separator application for rechargeable lithium metal cells. 2006. 131(1-3): p. 100-105.



- 24.Bergshoef, M.M. and G.J.J.A.m. Vancso, Transparent nanocomposites with ultrathin, electrospun nylon-4, 6 fiber reinforcement. 1999. 11(16): p. 1362-1365.
- 25.Asmatulu, R., W.S. Khan, and M.B. Yildirim, Acoustical properties of electrospun nanofibers for aircraft interior noise reduction. 2009.
- 26.Astley, R.J., et al. Finite element models for predicting the propagation and radiation of fan noise from turbofan engines. in INTER-NOISE and NOISE-CON Congress and Conference Proceedings. 2006. Institute of Noise Control Engineering.
- 27.Manneville, A., D. Pilczer, and Z.S.J.J.o.a. Spakovszky, Preliminary evaluation of noise reduction approaches for a functionally silent aircraft. 2006. 43(3): p. 836-840.
- 28.Hileman, J., et al. Development of approach procedures for silent aircraft. in 45th AIAA Aerospace Sciences Meeting and Exhibit. 2007.
- 29.Plas, A., et al. Performance of a boundary layer ingesting (BLI) propulsion system. in 45th AIAA aerospace sciences meeting and exhibit. 2007.



# CONTROLLING THE STRUCTURE AND PERFORMANCE OF GRAPHENE OXIDE MEMBRANES

# ISHA ABBASI, NAFIA TANSEEM, ZAFAR GHOURI, KHALED ELSAYED, AND AHMED ABDALA

<sup>1</sup>Chemical Engineering Program, Texas A&M University at Qatar, Doha, Qatar.

#### Abstract

Advanced membranes fabricated of multilayer/laminated graphene oxide (GO) are promising in water treatment membrane applications as they provide very high flux and good rejection of various water pollutants. However, these membranes are quasi stable and suffer from stability and swelling due to the hydrophilic nature of graphene oxide. In this presentation, we improved the stability and performance of laminated GO membranes via functionalization with different amine containing molecules. The membranes are fabricated via vacuum and pressure filtration and their structure is characterized using SEM, AFM, and mechanical testing. Pillaring the GO layers using diamine resulted in improved membrane stability and durability and increased water flux. Our results indicate that proper functionalization of GO and GO membranes provides roadmap for potential commercialization of such advanced membranes in water treatment application.

Keyword: Graphene oxide, membranes, d-spacing, functionalization

#### Introduction

Seawater and brackish water desalination is the main source of water for many countries including the Arabian Gulf countries gulf. ONn the other hand, treatment of domestic and industrial wastewater can provide alternative water sources for industrial applications, landscaping, or irrigation. Moreover, wastewater associated with the oil, gas, and petrochemical industry such as produced and process water requires proper treatment to allow its reuse, reinjection into depleted oil and gas reservoirs, aquifer recharge, or sea disposal. Although the type and concentration of pollutants in process and produced water vary significantly based on their source, salts and hydrocarbons are commonly present. Advanced treatment methods based on the use of organic and inorganic membranes is gaining increased importance recently, yet these membranes suffer from fouling and permeability/selectivity trade off.

#### **Graphene based Membranes**

Recently, advanced membranes based on nanomaterials are gaining significant interest. For example, using graphene and its derivatives such as graphene oxide (GO) and reduced graphene oxide to develop new graphene based-membranes or enhance the performance of commercial wastewater treatment membranes is a hot research subject. Graphene based membranes are classified in three categories[1]. The first category is based on single or few-layer graphene membranes with engineered nano-size pores and are characterized by very high flux and excellent salt rejection but we expect such membranes to remain research curiosity for the next decade(s) due to the major challenges associated with the ability to fabricate large and defect free membranes with large fraction of non-overlapping nono-size pores. [2-5] The second type of membranes is based on multilayer/laminated GO structure, which are fabricated using vacuum filtration or self-assembly techniques, but these GO membranes are quasi stable and suffer from stability and swelling due to the hydrophilic nature



of graphene oxide[6-9]. The third approach relies on the use of composites of graphene or graphene derivatives with different commercial membrane polymers to enhance the permeability, rejection, and/or antifouling characteristic of the commercial membrane [10-12].



**Figure 24:** Nanoporous graphene membrane (left)[13], graphene oxide support and freestanding membrane, SEM image of GO membrane cross section, and water touristy path (middle)[13], and polysulfone- graphene mixed-matrix membrane (right) (A. Abdala, unpublished results).

#### **Graphene Oxide membranes**

Graphene oxide is single layer of carbon atoms decorated with various oxygen containing functional groups such as hydroxyl, carbonyl, carboxyl and epoxy groups[14].



Figure 25: Left: Surface and cross-section SEM image of GO membrane [15].

GO membranes are candidates for the next generation membranes as they offer near 100% ion selectivity coupled with permeation rates that is 2-3 orders of magnitude higher than that of current commercial membranes in addition to other attractive characteristics such as thermal, chemical, and antifouling characteristics as well as ease of fabrication [16-21]. GO membranes are fabricated by vacuum filtration, spin-coating, or layer-by-layer assembly of GO suspension. They have been extensively studied experimentally for separation of organic molecules and ions from water streams and the dynamics of the water transport through GO membranes and the rejection of organic molecules and ions have been extensively studied using MD simulation [16, 17, 22, 23].

The effect of the inter layer spacing on the performance of GO membranes was investigated by molecular dynamic simulation and the results inidctaed that the water flux increases with the interlayer spacing [24].



**Figure 3:** tortuous pathfor water molecules through double layer GO membrane layer and effect of the d-spacing on water permeability [24].

The hydrophilicity of the GO membranes and the spacing between the GO layers is dictated by the concentration of the oxygen functional groups [25].



**Figure 4:** Mechanism of formation of GO membranes [26], effect of oxygen content on the interlayer spacing [25].

Despite the promising characteristics of GO membranes, they commercialization is currently hindered due to the limited stability and durability associated with the swelling and delamination of the GO layers due to the hydrophilic nature of GO [27, 28]. Zhang *et al.* observed an increase in the d-spacing of GO membranes upon soaking in water due to swelling leading not only to impacting the membrane integrity but also its ability to reject ions [27].



Figure 5: Increase of GO membrane d-spacing upon soaking in water for different time intervals[27]

Therefore, enhancing the stability of GO membranes is necessary to transform such promising membranes to the commercialization stage and there already several attempts to stabilize the structure of laminated GO membranes[15, 29-31].

# Experimental

# 3.1 Materials

Natural flake graphite (-10 mesh, 99.9%, Alfa Aesar), sulfuric acid (95–97%, J.T. Bakers), phosphoric acid (Sigma Aldrich), hydrogen peroxide (30% solution, BDH), potassium permanganate (Fisher Scientific), dioctanol amine (Alfa-Aesar), 1,8 dioctane amine (Alfa Aesar).

# 3.2 Experimental Work

Synthesis of GO was carried via the Tour method [25] by adding 1 g of graphite to a 9:1 of  $H_2SO_4/H_3PO_4$  mixture while stirring. Then, 5 g of KMnO4 was added to the solution and the reaction temperature was maintained at 20° C for three days. The mixture was centrifuged at 5000 rpm for 30 min and then spent sulfuric acid was decanted. Finally, GO with washed water six times, and collected by centrifuging at 5000 rpm for 60 min and dried at 80° C. *Functionalization with diaminooctane (DAO)* was carried by dissolving 1,8-diaminooctane is in ethanol and adding it dropwise to a suspension of GO in water under stirring and the reaction is continued for 24 h at room temperature. The functionalized GO is collected by centrifugation and washed with ethanol/water mixture before drying at 80° C. *Fabrication of GO and GO-DOA membranes* were carried by vacuum filtration of 150 ml of 3 mg/ml of GO/f-GO suspension over 0.2 µm polysulfone membrane (4.7 cm). After filtration, the membrane is left to dry in air. *The chemical structure of GO and GO-DOA* is analyzed using FTIR and XPS, while the morphology and the stacking of GO layers is measured by X-ray diffraction and the morphology of GO, GO,-DOA and the fabricated membranes is characterized using SEM. *The permeability of water through GO membranes* is measured at 1 bar using 300 ml deadend membrane cell with active membrane area of 4.63 cm<sup>2</sup>.



## **Results and Discussion**

The composition of the prepared GO is analuyzed by FTIR and XPS and the results shown in Figure 6 confirm the presence of hydroxy, epoxy, and carbonyl groups.



Figure 6: FTIR and C1s XPS spectra for GO.

Moreover, the functionalization of GO with DAO is also confimed by presnece of N1s peak in the XPS spectrum as shown in Figure 7. Moreover, the qunitative analysis indicates that the composition of GO. The FTIR spectrum reveal the atomic percentage of N/C/H/O has changed from 0/40/29/31 for GO to 2.3/37/39/22 for GO-DOA confirming the successful attachment of the DOA to the oxygen containing groups on the surface of GO. More importantly, XRD has confirmed the pillaring of the GO layer as can be deduced from the increase in the d-spacing from 8.1 to 10.2 Å. This crosslinking/pillaring the GO layers with DOA would stabilize the laminate structure and control the interlayer spacing.



**Figure 7:** Schematic representation of GO functionalization using DOA, XPS surve spectrum of GO and GO-DOA, and XRD patterns of GO and GO0-DOA.



Table	1:	XPS	elemental	analysis	(atomic%)	of	GO	and	GO	-DO	A
-------	----	-----	-----------	----------	-----------	----	----	-----	----	-----	---

Sample	Ν	С	H	0
GO	-	40.0	29.2	30.7
GO-DOA	2.3	37.3	38.8	21.6

The water flux through GO and GO-DOA membrane is measured at 2 bar and the results are shown in Figure 8. It is clearly evident that the increase d-spring upon functionalization of GO with DOA gas resulted in significant flux enhancement as the flux increase form ~30 LMH to ~ 3000 LMH corresponding to 100 time increase in water flux.



Figure 8: Water flux versus time for Go and GO-DOA membranes.

#### Conclusions

The functionalization of GO with DOA was successfully accomplished and analyzed. The pillaring of the GO layers with DOA has increased the inter-layer spacing of GO to 10.2 Å. This increase in the d-spacing has led to significant increase in water flux.

Acknowledgments: This research was supported by Qatar National Research Fund (QNRF) through Undergraduate Research Experience Program (UREP) project UREP23-056-2-028.

#### **References:**

- 1. Liu, G., W. Jin, and N. Xu, Graphene-based membranes. Chemical Society Reviews, 2015. 44(15): p. 5016-5030.
- 2. Cohen-Tanugi, D. and J.C. Grossman, Water desalination across nanoporous graphene. Nano letters, 2012. 12(7): p. 3602-3608.
- 3. Surwade, S.P., et al., Water desalination using nanoporous single-layer graphene. Nature nanotechnology, 2015. 10(5): p. 459-464.
- 4. Cohen-Tanugi, D. and J.C. Grossman, Water permeability of nanoporous graphene at realistic pressures for reverse osmosis desalination. The Journal of chemical physics, 2014. 141(7): p. 074704.
- 5. Sun, C., B. Wen, and B. Bai, Recent advances in nanoporous graphene membrane for gas separation and water purification. Science bulletin, 2015. 60(21): p. 1807-1823.
- 6. Dave, S.H., et al., Graphene oxide membranes. 2017, Massachusetts Institute of Technology, USA . p. 84pp.
- 7. Hibbs, M., et al. Graphene oxide-based desalination membranes. 2018. American Chemical Society.
- 8. Hu, M. and B. Mi, Enabling graphene oxide nanosheets as water separation membranes. Environmental science & technology, 2013. 47(8): p. 3715-3723.
- 9. Chen, C., et al., Self-Assembled Free-Standing Graphite Oxide Membrane. Advanced materials, 2009. 21(29): p. 3007-3011.

138

10. Hwang, T., et al., Ultrafiltration using graphene oxide surface-embedded polysulfone membranes. Sep. Purif. Technol., 2016. 166: p. 41-47.

MET

The International Conference of Materials and Engineering Technology

UWE Stitle

- 11. Ganesh, B., A.M. Isloor, and A.F. Ismail, Enhanced hydrophilicity and salt rejection study of graphene oxide-polysulfone mixed matrix membrane. Desalination, 2013. 313: p. 199-207.
- 12. Zinadini, S., et al., Preparation of a novel antifouling mixed matrix PES membrane by embedding graphene oxide nanoplates. Journal of Membrane Science, 2014. 453: p. 292-301.
- 13. Aghigh, A., et al., Recent advances in utilization of graphene for filtration and desalination of water: a review. Desalination, 2015. 365: p. 389-397.
- 14. He, H., et al., Solid-state NMR studies of the structure of graphite oxide. The Journal of physical chemistry, 1996. 100(51): p. 19954-19958.
- 15. Xi, Y.-H., et al., Graphene Oxide Membranes with Strong Stability in Aqueous Solutions and Controllable Lamellar Spacing. ACS Appl. Mater. Interfaces, 2016. 8(24): p. 15557-15566.
- 16. Willcox, J.A.L. and H.J. Kim, Molecular dynamics study of water flow across multiple layers of graphene oxide: Effect of graphene oxide layer-to-layer distance. J. Phys. Chem. C, 2017. 121(42): p. 23659-23668.
- 17. Willcox, J.A.L. and H.J. Kim, Molecular Dynamics Study of Water Flow across Multiple Layers of Pristine, Oxidized, and Mixed Regions of Graphene Oxide. ACS Nano, 2017. 11(2): p. 2187-2193.
- 18. Chen, B., et al., Molecular Insight into Water Desalination across Multilayer Graphene Oxide Membranes. ACS Appl. Mater. Interfaces, 2017. 9(27): p. 22826-22836.
- 19. Jiao, S. and Z. Xu, Non-Continuum Intercalated Water Diffusion Explains Fast Permeation through Graphene Oxide Membranes. ACS Nano, 2017. 11(11): p. 11152-11161.
- 20. Kholmanov, I., et al. Permeation of metal ions through graphene-based membranes. 2013. American Chemical Society.
- 21. Song, K., et al., Theoretical investigations of transport properties of organic solvents in cationfunctionalized graphene oxide membranes: Implications for drug delivery. Nano Res., 2018. 11(1): p. 254-263.
- 22. Lee, C.S., et al., Facilitated Water Transport through Graphene Oxide Membranes Functionalized with Aquaporin-Mimicking Peptides. Adv. Mater. (Weinheim, Ger.), 2018. 30(14): p. n/a.
- 23. Wei, N., X. Peng, and Z. Xu, Understanding water permeation in graphene oxide membranes. ACS Appl Mater Interfaces, 2014. 6(8): p. 5877-83.
- 24. Safaei, S. and R. Tavakoli, On the design of graphene oxide nanosheets membranes for water desalination. Desalination, 2017. 422: p. 83-90.
- 25. Amadei, C.A., et al., Role of oxygen functionalities in graphene oxide architectural laminate subnanometer spacing and water transport. Environmental Science & Technology, 2017. 51(8): p. 4280-4288.
- 26. Talyzin, A.V., et al., The structure of graphene oxide membranes in liquid water, ethanol and water-ethanol mixtures. Nanoscale, 2014. 6(1): p. 272-281.
- 27. Zheng, S., et al., Swelling of Graphene Oxide Membranes in Aqueous Solution: Characterization of Interlayer Spacing and Insight into Water Transport Mechanisms. ACS Nano, 2017. 11(6): p. 6440-6450.
- 28. Yeh, C.-N., et al., On the origin of the stability of graphene oxide membranes in water. Nat. Chem., 2015. 7(2): p. 166-170.
- 29. Hu, J.-Q., et al. Graphene oxide membranes with strong stability in aqueous solutions and controllable lamellar spacing. 2017. American Chemical Society.
- 30. Sun, S., et al., The mechanism for the stability of graphene oxide membranes in a sodium sulfate solution. Chem. Phys. Lett., 2013. 561-562: p. 166-169.
- 31. Nam, Y.T., et al., Enhanced Stability of Laminated Graphene Oxide Membranes for Nanofiltration via Interstitial Amide Bonding. ACS Appl. Mater. Interfaces, 2016. 8(40): p. 27376-27382.

# KINEMATIC ANALYSIS OF 3 DOF FLIGHT SIMULATOR

The International Conference

aterials and Engineering Technology

# EDIP OZTURK<sup>1</sup> KURSAD GOV<sup>2</sup>

 <sup>1</sup>Gaziantep University, Aeronautics and Aerospace Faculty, Aircraft and Aerospace Engineering Department, Gaziantep, TURKEY.
 <sup>2</sup>Gaziantep University, Aeronautics and Aerospace Faculty, Aircraft and Aerospace Engineering Department, Gaziantep, TURKEY.

#### Abstract

Real flight trainings have many disadvantages. Flight simulators are used to eliminate these disadvantages and provide safety flight training conditions. Every vehicle has its own simulator in order to simulate vehicle's motion. The main aim of the simulator usage is to make pilot feel a realistic perception of flight. Human body feels motion by means of its vestibular system and eyes. Therefore, an acceptable flight simulator has ability to stimulate human motion perception organs as realistic as possible. An algorithm called motion cue algorithm is used to convert real motion parameters to vestibular system parameters. Since, pilot is located in flight simulator, defining flight simulator kinematic parameters is obligatory. Flight simulator is consist of a base and three ring shaped structure. Three nested rings form the simulator, all degree of freedom of flight simulator are rotation type. However, only motion can be performed by simulator is rotation, still linear motion feeling can be generated in simulator. This study presents Kinematic analysis of flight simulator. Rotation matrices is used to obtain kinematic parameters of the flight simulator. Firstly, Kinematic analysis is performed analytically. Then, three-dimensional flight simulator model is generated in engineering modeling software and numerical validation is done by means of computer aided analysis software.

Keywords: Flight Simulator, Kinematic Analysis, Rotation Matrices, Coordinate Transformation.

#### **1. Introduction**

Flight simulator has great importance for both military and educational purpose. In literature, many papers are available about flight simulators. Since, designed flight simulator has similar structure with gyro, relevant studies should be considered. Usubamatov, studied mathematical model of gyroscope and derived its motion behavior. After deriving analytical model of gyroscope, he compared analytical results with practical results [1]. Volkaner et al. studied motion cueing algorithm for flight simulators. Since, using machines as flight simulators have finite moving ranges but real aircrafts have no limit to move, real flight data should be mapped. Volkaner et al. applied washout filter to data to obtain limited data for flight simulator [2]. Kinematic analysis case rotational matrix method easy to apply and solve. Day et al. shows how to extract Euler angles from rotation matrix [3]. Gov et al. designed and manufactured a flight simulator which has structure as human gyroscope. They used servo motors to drive the flight simulator [4]. Gov and Oguz, compared electric motors and they selected suitable electric motors to drive the human gyroscope shaped flight simulator [5].

In this study, kinematic analysis of flight simulator is performed. Firstly, kinematic equations are derived analytically and these equations are tested with a case study. Numerical analysis of flight simulator is done and correctness of numerical results are validated by analytical results.

ne International Conference

Materials and Engineering Technology

#### 2. Materials and Methods

#### 2.1. Kinematics

Kinematics is a branch of mechanics which deals with shape or geometry of motion. In kinematic analysis case, forces and moments are not involved, it means that kinematics doesn't consider causes of motion, it only considers motion itself. Since flight simulator is a dynamic machine which means that its parts move continuously relative to each other. Every part of machine effects the other parts motion, therefore absolute motion of specified point should be defined clearly. Specified point for flight simulator is location of the pilot's head. Because main purpose of the flight simulator is artificially creates real flight perception. Therefore, while performing kinematic analysis, a fixed coordinate frame should be attached to a base and relative coordinate frames should be attached to every moving part of the flight simulator. Relations between relative coordinate frames and fixed frame are should be defined clearly.

#### **2.1.1. Coordinate transformation – Pure Rotation**

Point P is arbitrarily located in figure 1. Frame 0 is attached to fix point so it is inertial coordinate frame and frame 1 is relative coordinate frame. When frame 1 is rotated about Z axis of frame 0, relation between location of point P according to frame 0 and location of point P according to frame 1 is called Pure rotation coordinate transformation.



Figure 1. Pure rotation of coordinate frame.



UWE of the Bristol Engla

$x_o = r \cos \alpha$	(1)
$y_o = r \sin \alpha$	(2)
Location of point P according to frame 1 are written as follows;	
$x_1 = r \cos(\alpha - \theta)$	(3)
$y_1 = r\sin(\alpha - \theta)$	(4)
$x_1 = r \cos \alpha \cos \theta + r \sin \alpha \sin \theta$	(5)
$y_1 = r \sin \alpha \cos \theta - r \cos \alpha \sin \theta$	(6)
If equation 1 and 2 is substituted to equation 5 and 6, equations become;	
$x_1 = x_0  \cos\theta +  y_0 \sin\theta$	(7)
$y_1 = -x_0 \sin\theta + y_0 \cos\theta$	(8)
Equation 7 and 8 in matrix form;	
$\begin{bmatrix} X_1 \end{bmatrix} \begin{bmatrix} \cos \theta & \sin \theta \end{bmatrix} \begin{bmatrix} X_0 \end{bmatrix}$	

$$\begin{bmatrix} x_1 \\ y_1 \end{bmatrix} = \begin{bmatrix} \cos\theta & \sin\theta \\ -\sin\theta & \cos\theta \end{bmatrix} \begin{bmatrix} x_0 \\ y_0 \end{bmatrix}$$
(9)

The matrix which consists of trigonometric terms is called rotation matrix and it shows rotation about Z axis amount of  $\theta$ .

In a same manner, if we write rotation matrices for three dimensional case;

Rotation about Z:

$$[R]_{z} = \begin{bmatrix} \cos\psi & -\sin\psi & 0\\ \sin\psi & \cos\psi & 0\\ 0 & 0 & 1 \end{bmatrix}$$
(10)

About X:

$$[R]_{\chi} = \begin{bmatrix} 1 & 0 & 0 \\ 0 & \cos\alpha & -\sin\alpha \\ 0 & \sin\alpha & \cos\alpha \end{bmatrix}$$
(11)

About Y:

$$[R]_{y} = \begin{bmatrix} \cos\beta & 0 & \sin\beta \\ 0 & 1 & 0 \\ -\sin\beta & 0 & \cos\beta \end{bmatrix}$$
(12)



## **2.1.2.** Coordinate transformation – Pure Translation

Point P is arbitrarily located in figure 2. Frame 0 is attached to fix point so it is inertial coordinate frame and frame 1 is relative coordinate frame. When frame 1 is translated from frame 0, relation between location of point P according to frame 0 and location of point P according to frame 1 is called Pure translation coordinate transformation.



Figure 2. Pure translation of coordinate frame.

$$x_o = x_1 + \Delta x \tag{13}$$

$$y_o = y_1 + \Delta y \tag{14}$$

## 2.1.3. Homogeneous transformation

Rotation and translation matrices can be expressed as single matrix. This single matrix is called homogeneous transformation matrix.

Pure Rotation:

$$R = \begin{bmatrix} \cos\gamma & -\sin\gamma & 0 & 0\\ \sin\gamma & \cos\gamma & 0 & 0\\ 0 & 0 & 1 & 0\\ 0 & 0 & 0 & 1 \end{bmatrix}$$
(15)

Pure Translation:

$$T = \begin{bmatrix} 1 & 0 & 0 & x \\ 0 & 1 & 0 & y \\ 0 & 0 & 1 & z \\ 0 & 0 & 0 & 1 \end{bmatrix}$$
(16)





## Homogeneous Transformation:

	cosγ	−sinγ	0	x
H =	sinγ	cosγ	0	y
	0	0	1	Z
	0	0	0	1

(17)

## 2.1.4. Flight Simulator Model



Figure 3. Flight simulator model.

The flight simulator consists of three nested rings and a base which supports three rings (Fig.3). The base part only supports the moving parts and it is fixed. Therefore, inertial coordinate frame can be attached to base (Fig.4).



Figure 4. Base part and inertial coordinate frame.



Coordinate frame 1 is attached to center of outer ring (Fig.5) and outer ring makes only rotational motion about its axis Z. Distance between origin of frame 0 and origin of frame 1 is called as d1.



Figure 5. Outer ring coordinate frame.

Flight simulator makes pitch rotation by means of outer ring and pitch angle is defined as gamma (Fig.6.).



Figure 6. Pitch angle gamma.

Homogeneous transformation from frame 1 to 0, in other words outer ring coordinate frame to base coordinate frame becomes as follows;

$${}_{1}^{0}T = \begin{bmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & d_{1} \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{bmatrix} \begin{bmatrix} \cos\gamma & -\sin\gamma & 0 & 0 \\ \sin\gamma & \cos\gamma & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{bmatrix}$$
(18)



Coordinate frame 2 is attached to center of middle ring (Fig.7) and outer ring makes only rotational motion about its axis Z. Origins of coordinate frames are coincident.



Figure 7. Outer ring coordinate frame.

Flight simulator makes yaw rotation by means of middle ring and yaw angle is defined as psi (Fig.8.).



Figure 8. Yaw angle psi.

Homogeneous transformation from frame 2 to 1, in other words middle ring coordinate frame to outer ring coordinate frame becomes as follows;

$${}_{2}^{1}T = \begin{bmatrix} 1 & 0 & 0 & 0 \\ 0 & \cos(-90) & -\sin(-90) & 0 \\ 0 & \sin(-90) & \cos(-90) & 0 \\ 0 & 0 & 0 & 1 \end{bmatrix} \begin{bmatrix} 1 & 0 & 0 & 0 \\ 0 & \cos\psi & -\sin\psi & 0 \\ 0 & \sin\psi & \cos\psi & 0 \\ 0 & 0 & 0 & 1 \end{bmatrix}$$
(19)

Coordinate frame 3 is attached to center of inner ring (Fig.9) and inner ring makes only rotational motion about its axis Z. Origins of coordinate frames are coincident.



Figure 9. Inner ring coordinate frame.

Flight simulator makes roll rotation by means of inner ring and roll angle is defined as theta (Fig.10.).



Figure 10. Roll angle theta.

Homogeneous transformation from frame 3 to 2, in other words inner ring coordinate frame to middle ring coordinate frame becomes as follows;

$${}_{3}^{2}T = \begin{bmatrix} \cos(-90) & 0 & \sin(-90) & 0 \\ 0 & 1 & 0 & 0 \\ -\sin(-90) & 0 & \cos(-90) & 0 \\ 0 & 0 & 0 & 1 \end{bmatrix} \begin{bmatrix} \cos\theta & 0 & \sin\theta & 0 \\ 0 & 1 & 0 & 0 \\ -\sin\theta & 0 & \cos\theta & 0 \\ 0 & 0 & 0 & 1 \end{bmatrix}$$
(20)



Overall homogeneous transformation matrix from frame 3 to 0 can be defined as simply multiplication of three homogeneous transformation matrices.

$${}^{0}_{3}T = {}^{0}_{1}T{}^{1}_{2}T {}^{2}_{3}T$$
(21)

And overall homogeneous transformation matrix becomes;

	(
--	---

Where:

$$r_{11} = \sin(\theta) \cos(\gamma) - \sin(\gamma) \cos(\psi) \cos(\theta)$$
  

$$r_{12} = -\sin(\psi) \sin(\gamma)$$
  

$$r_{13} = -\cos(\theta) \cos(\gamma) - \sin(\gamma) \cos(\psi) \sin(\theta)$$
  

$$r_{21} = \sin(\theta) \sin(\gamma) + \cos(\gamma) \cos(\psi) \cos(\theta)$$
  

$$r_{22} = \sin(\psi) \cos(\gamma)$$
  

$$r_{23} = -\cos(\theta) \sin(\gamma) + \cos(\gamma) \cos(\psi) \sin(\theta)$$
  

$$r_{31} = -\sin(\psi) \cos(\theta)$$
  

$$r_{32} = \cos(\psi)$$
  

$$r_{33} = -\sin(\psi) \sin(\theta)$$

#### 2.1.5. Case Study

After deriving homogeneous transformation equations, using a simple numerical example for testing the correctness of the equations is better.

$$P_0 = {}^3_0 T * P_3 \tag{23}$$

Where  $P_0$  is point in coordinate frame 0, and  $P_3$  is point in coordinate frame 3.

For 
$$\gamma = 30^{\circ}, \theta = 0^{\circ}$$
 and  $\psi = 0^{\circ}$  For  $P_3 = \begin{bmatrix} 200\\0\\0\\1 \end{bmatrix} \quad {}_{0}^{3}T = \begin{bmatrix} -0.5 & 0 & -0.866 & 0\\0.866 & 0 & -.05 & 600\\0 & -1 & 0 & 0\\0 & 0 & 0 & 1 \end{bmatrix}$ 

The Result is:

$$P_0 = \begin{bmatrix} -100\\773.2\\0\\1 \end{bmatrix}$$
 Point coordinates at frame 0, x= -100 mm, y= 773.2 mm and z= 0 mm.

In addition to analytical calculation, numerical analysis is performed by using Msc Adams which is dynamic analysis software.



#### Figure 11. Flight simulator model Adams view.

For Msc Adams dynamic analysis software, geometry is imported from Solidworks three dimensional computer aided engineering modelling software. Revolute joints are assigned between parts in order to model rotations. 30° rotation applied to outer ring in order to simulate pitch rotation. Simulation time is selected as one second and simulation is performed successfully.



Figure 12. Flight simulator rotated view.

#### 3. Results and Discussion

As a result of analytical calculations,

For 
$$\gamma = 30^{\circ}, \theta = 0^{\circ}$$
 and  $\psi = 0^{\circ}$  For  $P_3 = \begin{bmatrix} 200\\0\\0\\1 \end{bmatrix} \quad {}_{0}^{3}T = \begin{bmatrix} -0.5 & 0 & -0.866 & 0\\0.866 & 0 & -.05 & 600\\0 & -1 & 0 & 0\\0 & 0 & 0 & 1 \end{bmatrix}$ 

The result is obtained as;

Point coordinates at frame 0, x = -100 mm, y = 773.2 mm and z = 0 mm.





Numerical analysis is performed during one second and pitch angle is assigned as 30° as analytical calculation (fig.13).



During simulation time, it is clearly seen that X coordinate of point at frame 0 is -100 mm (fig.14).



In a same manner, Y coordinate of point at frame 0 is 773.2 mm (fig.15).



Finally, Z coordinate of point at frame 0 remains 0 mm during simulation time.



# 4. Conclusions

In this study, kinematic analysis of a 3 dof flight simulator is performed. Firstly, Analytical equations are derived and used for simple case study to check their correctness. Next, flight simulator solid model is created by using engineering modelling software Solidworks and model is exported as parasolid file format. Exported parasolid file is imported to Msc Adams dynamic analysis software to run numerical analysis. Finally, numerical result and analytical result are compared and it is observed that numerical result is good agreement with analytical result. Therefore, kinematic model can be used for further studies and its correctness is validated.

The International Conference

of Materials and Engineering Technology

## **References:**

- 1. Usubamatov, R., Mathematical Model for Gyroscope's Gimbal Motions.
- Volkaner, B., et al. Experimental Motion Cueing Studies Employing Desktop Flight Simulation System. In Proceedings of the 14th International Conference on Circuits, Systems, Electronics, Control & Signal Processing (CSECS '15). 2015.
- 3. Day, M. and I. Games, Extracting Euler angles from a rotation matrix. Insomniac Games R&D. Available online at: http://www. insomniacgames. com/mike-day-extracting-euler-angles-from-a-rotation-matrix, 2012.
- 4. Gov et al. Servo Motor Driven Human Gyroscope Structured Flight Simulator. In 5<sup>th</sup> International Conference on Advanced Technology&Sciences.2012.Istanbul.
- 5. Gov. K. Oguz. M. Human Gyroscope Control. In International Mechanical Engineering and Technologies Conference. 2016. Istanbul.

# **ELECTROSPINNING OF PAN/ZEOLITE COMPOSITE NANOFIBERS**

he International Conference of Materials and Engineering

Technology

# FUNDA CENGİZ ÇALLIOĞLU<sup>\*1</sup> and HÜLYA KESİCİ GÜLER<sup>1</sup>

<sup>1</sup> Süleyman Demirel University, Engineering Faculty, Textile Engineering Department, Isparta, TURKEY.

#### Abstract

In this study, it was aimed to achieve production and characterization of polyacrylonitrile (PAN)/zeolite nanofibers via electrospinning method. Firstly, polymer solutions were prepared with various concentrations of zeolite. Then, solution properties were determined such as; viscosity, conductivity and surface tension. Nanofiber production was carried out with electrospinning under the optimum process parameters (voltage, distance between electrodes, feed rate and atmospheric conditions). Lastly, nanofibers were morphologically characterized with SEM-EDS. According to the results; solution viscosity and surface tension increased while conductivity decreased with zeolite concentration increasement. Also, during the spinning process, it was observed that spinning performance decreased and average fiber diameter increased dramatically with zeolite concentration especially for 10 wt % PAN/10 wt % zeolite. Moreover, zeolite particles held on to the nanofibrous structure successfully even at minimum and maximum concentration.

Keyword: Polyacrylonitrile, Zeolite, Nanofiber, Electrospinning

#### **1. Introduction**

Zeolite is a natural raw material which has superior functional properties such as environmental pollution control, filtration, energy applications (storage of solar energy, purification of natural gas, deodorizing of malodor), antibacterial property, absorption of radioactive waste [1-7].

Electrospun nanofibers have superior properties than conventional fibers such as; high specific surface area  $(m^2/g)$ , high porosity, small and controllable pore size, and very small fiber diameter [8–10]. Similarly, polyacrylonitrile (PAN) which is used widely for electrospinning method was chosen as a polymer in terms of high spinning performance and absorbent properties [3, 11].

In literature, there are limited study about electrospinning of nanofibers with zeolite [1, 3, 6-7]. For this aim, in this study, effect of zeolite concentration on the solution properties and fiber morphology was investigated. It is thought that this nanofibrous composite material has an important end-use potential as a filtration application or absorption material.

#### 2. Materials and Methods

In this study, PAN was used as a polymer, dimethylformamide (DMF) was used as a solvent and zeolite was used as an additive. PAN and DMF were purchased Corporation (St. Louis, MO, USA), zeolite was supplied Rota Madencilik A.Ş. (<40  $\mu$ m particle size). All solutions were prepared with 10 wt % PAN polymer concentration in consider of our previous studies (in terms of high spinning performance and bead-free fiber structure). PAN/DMF polymer solutions were prepared with various zeolite concentrations (Table 1).



Table 1. PAIN sample codes with vario			bus concentration of ze
		PAN	Zeolite
	Sample Codes	Concentration	Concentration
		(wt %)	(wt %)
	ZEO-0	10	0
	ZEO-2	10	2
	ZEO-4	10	4
	ZEO-6	10	6
	ZEO-8	10	8
	ZEO-10	10	10

After polymer solutions prepared, solution properties were determined such as; conductivity, viscosity (shear rate 5 s<sup>-1</sup>) and surface tension with Wilhelmy Plate method. Then, nanofiber production was carried out with electrospinning system under the optimum process parameters (Table 2). All nanofibers were produced for 20 minutes and collected on aluminum foil.

		Table 2. Process parameters of the electrospinning process						
Voltage (kV)	Distance Between Electrodes (cm)	Feed Rate (mL/h)	Humidity (%)	Temperature (°C)				
23.4	18.2	0.7	54	25.4				

Lastly, nanofiber morphology and inorganic substance were analyzed with SEM-EDS on a FEI Quanta 250 FEG model instrument. And also, histogram curves were created with SPSS statistical program to analyze fiber diameter distribution.

#### **3. Results and Discussion**

According to the solution properties results; viscosity and surface tension increase and conductivity decreases with zeolite concentration increasement (Table 3 and Figure 1).

Table 3. Solution properties of PAN polymer solutions							
Solution Codes	Conductivity (µS/cm)	Viscosity (Pa.s) (shear rate 5s <sup>-1</sup> )	Surface Tension (mN/m)				
ZEO-0	95.8	0.65	21.7				
ZEO-2	52.6	0.92	36.6				
ZEO-4	49.8	1.35	42.8				
ZEO-6	46.2	1.78	54.4				
ZEO-8	45.4	2.88	58.8				
ZEO-10	44.7	4.12	62.1				

# olite



Figure 1. a) Viscosity and conductivity of PAN/zeolite solutions b) Surface tension of PAN/zeolite solutions

SEM images and fiber diameter histograms of PAN nanofibers with various concentrations of zeolite are given in Figure 2.



ZEO-2



Figure 2. SEM images (1.000x and 5.000x) and histograms of PAN nanofibers samples with various concentrations of zeolite

155



According to the fiber morphology analyzes, it is seen clearly that, average fiber diameter increased with zeolite concentration increasement. It is possible to say that nanofibers were obtained with ZEO-0 and ZEO-2 while microfibers (higher than 1000 nm) were obtained with ZEO-4, 6, 8, 10. Moreover, spinnability reduced with zeolite concentration significantly. Generally, nanofiber samples had unimodal histogram curves. In addition, solution properties and fiber morphology results are compatible with each other. It is possible to say, zeolite particles hold on to the fiber surface. According to the Figure 3, the most uniform nanofibers were obtained with ZEO-2 (1.008).



Figure 3. Average fiber diameter and diameter uniformity coefficient of PAN/zeolite nanofibers

SEM-EDS results are given in Figure 4. and Table 4.
















### ZEO-10

Figure 4. SEM-EDS Spectroscopy of PAN nanofibers with various concentrations of zeolite

Sample	С	Ν	0	Al	Si	S	Cl	K	Ca	Fe
Codes	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
ZEO-2	66.55	28.11	4.56	0.32	1.24	0.10	-	0.06	0.06	-
ZEO-4	90.93	-	5.66	0.89	2.13	0.17	-	-	0.22	-
ZEO-6	64.70	-	3.94	28.98	1.34	0.08	0.07	0.16	0.22	0.52
ZEO-8	56.37	15.69	8.74	16.25	2.63	-	-	0.13	0.19	-
ZEO-10	66.92	-	6.72	22.91	2.56	-	-	0.18	0.24	0.45

Table 4. SEM-EDS	results	of PAN/ze	eolite 1	nanofiber	samples
------------------	---------	-----------	----------	-----------	---------

Table 5. SEM-EDS results of pure zeolite raw material											
Sample	С	Ν	0	Mg	Al	Si	S	Cl	K	Ca	Fe
Codes	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
Sample-1	13.65	-	40.96	0.42	6.32	30.73	-	-	2.69	3.62	1.62
Sample-2	7.07	-	57.16	0.98	6.10	25.03	-	-	1.23	1.71	0.71
Sample-3	8.59	_	45.38	2.06	14.00	20.49	_	_	2.96	_	1.52

Generally, zeolite consists of Carbon (C), Oxygen (O), Aluminum (Al) and Silicium (Si). Elemental structure can be show difference from each other regionally due to natural structure of zeolite (Table 5). For this reason, elemental analysis results of zeolite nanofibers showed differences between each other.

### 4. Conclusions

This study was carried out production and characterization of PAN nanofibers with various concentrations of zeolite. PAN concentration was adjusted at 10 wt % constantly and zeolite concentrations were applied 2, 4, 6, 8 and 10 wt %. Firstly, solution properties were determined and then nanofiber production was achieved with optimum process parameters. Lastly, PAN nanofibers include various zeolite concentrations were analyzed with SEM-EDS. According to the results; zeolite concentration has a significant effect on the solution properties. Viscosity and surface tension increases while conductivity decreased with zeolite concentration increasement. It was observed from the SEM-EDS analyzes, average fiber diameter increased and spinnability decreased with zeolite concentrations. However, fiber diameter histograms showed that fiber diameter distributions were unimodal. In conclusion; it is possible to say that zeolite particles hold on to the fibers surface successfully. It is expected that results of this study provide basic information for the application of



### References

- 1. Agarwal, S.R., Subramanian, S. and Seeram, R. Functionalized cellulose:PET polymer fibers with zeolites for detoxification against nerve agents. Journal of Inorganic Materials, **2012**, 27(3):332-336.
- 2. Bilgin, Ö. and Koç, E. Doğal zeolitlerin çevre kirliliği kontrolünde kullanımı. Türk Bilimsel Derlemeler Dergisi, **2013**, 6(1):169-174.
- 3. Faghihian, H., Iravani, M., Moayed, M. and Ghannadi-Maragheh, M. A novel polyacrylonitrile-zeolite nanocomposite to clean Cs and Sr from radioactive waste. Environmental Chemistry Letters, **2013**, 11:277-282.
- 4. Khatri, L., Ramsier, R.D. and Chase, G.G. High temperature zeolite nanofibers, <u>http://www.slidefinder.net/2/20oct03\_20zeolite\_20nanofibers/9oct03zeolitenanofibers/2779</u> <u>1040</u>
- 5. Moattar, F. and Hayeripour, S. Application of chitin and zeolite adsorbents for treatment of low level radioactive liquid wastes. International Journal of Environmental Science&Technology, **2004**, 1(1):45-50.
- 6. Namekawa, K., Schreiber, M. T., Aoyagi, T. and Ebara, M. Fabrication of zeolite-polymer composite nanofibers for removal of uremic toxins from kidney patients. Biomaterials Science, **2013**, doi:10.1039/c3bm60263j.
- Nawawi, M.S., Ahmad, M.R., Affandi, N.D.N, Sekak, K.A. and Ahmad, W.Y.W. Effect of zeolite presence and voltage variance on the fiber diameter of microporous PVA/zeolite nanofibrous membrane. IEEE Business Engineering and Industrial Applications Colloquium (BEIAC), 2013, 344-348.
- 8. Bhardwaj, N., and Kundu, S.C. Electrospinning: a fascinating fiber fabrication technique.
- 9. Biotechnol. Adv., **2010**, 28(3):325–347.
- 10. Haghi, A.K. Advances in Nanofibre Research, Smithers Rapra, Shawbury, Shrewsbury, Shropshire, United Kingdom, **2011**,
- 11. He, J.-H. et al. Electrospun nanofibres and their applications, ISmithers, Shawbury, UK, 2008.
- 12. Chen, M., Wang, C., Fang, W., Wang, J., Zhang, W., Jin, G. and Diao, G. Electrospinning of calixarene-functionalized polyacrylonitrile nanofiber membranes and application as an adsorbent and catalyst support. Langmuir, **2013**, 29(38):11858-11867.



# DETERMINATION OF THE CRITICAL POINT ON SNIPER BARREL IN TERMS OF THE STRESS AND DEFORMATION CONCEPTS

# MEHMET HANİFİ DOĞRU<sup>1</sup>

<sup>1</sup>Gaziantep University, Aeronautics and Aerospace Faculty, Pilotage Department, Gaziantep, TURKEY.

### Abstract

Impact performance of the material is critical property in the weapon industry. In particular, pressure distribution concept in barrel has revealed the need to work on this issue. Therefore, the behavior of the barrel was investigated, when the explosion pressure was realized in the barrel. In this study, different pressure values were applied to the inside of the barrel to obtain critical stress region. The barrel of the new generation long range sniper weapon of the American Barret company, which is legendary with the M82A series, was used. 3D barrel model was created in SOLIDWORKS program. ANSYS workbench static structural toolbox was used to perform the proposed study. Within this study, the critical points in sniper weapon barrel were determined in terms of the stress and deformation concepts.

Keywords: Barrel, Numerical analyses, Pressure distribution, Deformation and stress values.

### **1. Introduction**

The importance of the impact performance of the material is great. Especially, these concepts have been studied in the weapon industry for along time. Penetration equations using analytical dependence between impact velocity and depth of penetration was derived by Ben-Dor et al. [1].

A model of high-speed penetration into ductile targets were universalized by Ben-Dor et al. [2]. Correlation between ballistic and residual velocities and impact were compared by Ben-Dor et al. [3] and Lambert-Jonas [4]. For this comparison Recht and Ipson [5] formulations were used by the authors.

Propellent geometry was designed using mathematical optimization method by Yıldırım [6]. Barrel velocity and pressure values were determined using PRODAS software. Optimum web thickness value was calculated with non-linear regression method by using MATLAB optimization tool by using these values.

A new model has been developed which gives the pressure distribution within the barrel and the velocity of the projectile by Işık [7]. To confirm the model, several firing tests were performed with a 7.62 mm diameter barrel and ballistic parameters were measured.

The barrels are considered as thin-walled circular tubes with special use. They are constantly exposed to high temperatures and pressures during operation. Therefore, the dynamic temperature data of the barrel and the stress values under continuous loading were examined using FEM software by Changwei et al. [8].



A trigger mechanism for a 20 x 102 mm caliber sniper rifle was designed by Gullerova [9]. Kinematic and dynamic quantities of the sniper rifle trigger mechanism were analyzed.

Two analytical methods and one numerical method are considered to determined pressure profile by 10. Micković et al. [10]. The analytical methods of proportionate expansion and two-phase mixture were studied. Pressure profiles were computed numerically

The CFD model was formulated to illustrate the details of the flow field produced by the revolving barrel gun firing by Yu and Zhang [11]. Two different algorithms, which are second-order monotone upstream-centered schemes approach and the advection upstream splitting method solver was used to simulate the high-pressure muzzle flow field.

At the end of the literature review, it was determined that stress distribution has critical importance. So, critical stress and deformation values were investigated according to the different explosion pressures in the barrel.

## 2. Materials and Methods

### **2.1. Numerical Analyses**

The barrel of the new generation long range sniper weapon of the American Barret company, which is legendary with the M82A series, was used. 3D barrel model was created in SOLIDWORKS program as shown in Figure 1.



Figure 1. 3D barrel model

Mesh accuracy was performed to check the reliability of the meshing procedure. Mesh structure were shown in Figure 2. After mesh accuracy (Figure 3) procedure, it was determined that the 90000-element number is suitable for these analyses.



Figure 2. Mesh view of barrel



Figure 3. Mesh accuracy

ANSYS workbench static structural toolbox was used to perform the proposed study. 200 Mpa pressure was applied to the barrel for this analysis as shown in Figure 4.



Figure 4. Applied pressure



Figure 5. Support point and position

Fixed support was applied to the system to perform the study as shown in Figure 5.



Figure 6. von-Mises stress result

Von-Mises stress distribution and deformation results were obtained according to the applied pressure as shown in Figure 6 and Figure 7.



Figure 7. Deformation result



Figure 8. Fatigue life

At the end of the analyses, fatigue life of the barrel was determined according to the applied pressure.

## 2.2. Theoretical Validation of the Analyses Results

When the geometry of the M82A gun barrel was investigated, it was clearly seen that the system behavior is thick walled cylinder (in Figure 9). According to the Equation 1, if the r/t ration is bigger than 10, the system calls as thin walled cylinder.



Figure 9. Thick walled cylinder

$$\begin{split} r_{i} &= 6,35 \text{ mm} \\ r_{0} &= 15,5 \text{ mm} \\ t &= 9,15 \text{ mm} \\ p_{0} &= 0 \text{ Mpa} \\ p_{i} &= 200 \text{ Mpa} \\ \\ \frac{r_{i}}{t} &> 10 \\ \sigma_{h} &= \frac{p_{i}r_{i}^{2} - p_{0}r_{0}^{2}}{r_{0}^{2} - r_{i}^{2}} + \frac{r_{i}^{2}r_{0}^{2}(p_{i} - p_{0})}{r^{2}(r_{0}^{2} - r_{i}^{2})} \end{split}$$

(1)

(2)



When the  $p_i$  was applied to the barrel and equation (2) was used, it was observed that the results of the theoretical and the analysis results overlapped.

### **3. Results and Discussion**

Stress, deformation and fatigue life of barrel obtained according to different pressure values were determined by analysis as given in Table 1. ANSYS workbench static structural toolbox was used to perform the proposed study.

Table 1. Analyses results.						
Pressure	Stress	Deformation	Life			
(Mpa)	(Mpa)	( <b>mm</b> )	(cycle)			
25	56,94,8	0,01104	1000000			
50	113,9	0,02208	200105			
100	227,79	0,04417	16148			
150	341,69	0,06626	4400			
200	455,59	0,08834	1827			
250	569,48	0,11043	1028			
350	683,38	0,13253	640			

Generally, barrels are made of heat-treated special materials. Therefore, these materials can withstand these stresses when subjected to high stress values.



Figure 10. Pressure-Stress curve

At the end of the study, critical points in sniper weapon barrel were determined in terms of the stress (in Figure 10) and deformation (in Figure 11) concepts. Maximum von-Mises stress was obtained as 683 Mpa (in Figure 10), when the 350 Mpa pressure was applied.



Figure 11. Pressure-Deformation curve

Maximum deformation value was obtained as 0,13253 mm at tip of barrel (in Figure 11), when the 350 Mpa pressure was applied.



Figure 12. Pressure-Fatigue life curve

Also, Fatigue life was determined for the barrel according to applied different pressure values (in Figure 12). Minimum fatigue life of barrel was achieved as 640 cycles, due to applied pressure value was high.

# 4. Conclusions

The behavior of the barrel was investigated, when the explosion pressure was realized in the barrel. In this study, different pressure values were applied to the inside of the barrel to obtain critical stress region. It was seen that the pressure formed inside the barrel decreases from inside to outside. therefore, it was considered that it would be appropriate to produce them using functional graded material method while producing barrels. By means of this method, both lighter and more durable



barrels will be produced since the inside of the barrel having critical stress value will have higher mechanical properties. In addition, the fatigue life of the barrel will also be increased.

### References

- 1. Ben-Dor, G., A. Dubinsky, and T. Elperin, Engineering approach to penetration modeling. Engineering Fracture Mechanics, 2008. 75(14): 4279-4282.
- 2. Ben-Dor, G., A. Dubinsky, and T. Elperin, A model of high speed penetration into ductile targets. Theoretical and applied fracture mechanics, 1998. 28(3):237-239.
- 3. Ben-Dor, G., A. Dubinsky, and T. Elperin, On the Lambert–Jonas approximation for ballistic impact. Mechanics Research Communications, 2002. 29(2):137-139.
- 4. Lambert, J.P., Jonas, G.H., Report. 1976, Ballistic Res. Lab.
- Recht, R. and T. Ipson, Ballistic perforation dynamics. Journal of Applied Mechanics, 1963. 30(3):384-390.
- 6. Yıldırım, F., The Effect of Geometric Changes of Propellants which Used in Large Caliber Weapons on Barrel Pressure and Muzzle Velocity, Ankara University Graduate School of Natural and Applied Sciences Department of Engineering Physics, 2013, M.Sc Thesis.
- 7. Işık, H., Modeling the Ballistic Parameters in a Barrel, The Journal of Defense Sciences, 2016, 15(2): 157-177.
- Chang-wie, W., Yong-hai, W., Qin-man, F., Analysis of Temperature and Stress of a Thin-Walled Cylinder based on FEM, Applied Mechanics and Materials Vols 373-375 2013:12-15
- 9. Gullerova, M., Design of a Trigger Mechanism for a Sniper Rifle, American International Journal of Contemporary Research, 2012, 2(7):106-117
- Micković, D., Jaramaz, S., Elek, P., Jaramaz, D., & Micković, D., Determination of pressure profiles behind projectiles during interior ballistic cycle. Journal of Applied Mechanics, 2013, 80(3): 031402.
- 11. Yu, W., Zhang, X., Numerical Simulation and Analysis of the Muzzle Flow During the Revolving Barrel Gun Firing, Journal of Applied Mechanics, 2013, Vol. 80: 031602-1

# INVESTIGATION OF THE BULLET IMPACT ENERGY PERFORMANCE ACCORDING TO VARIABLE TIP GEOMETRY

The International Conference of Materials and Engineering Technology

# Murat SUBAȘI<sup>1</sup>, Mehmet Hanifi DOĞRU<sup>\*2</sup>, Eyüp YETER<sup>3</sup> and Necip Fazıl YILMAZ<sup>4</sup>

<sup>1</sup>Gaziantep University, Aeronautics and Aerospace Faculty, Aircraft and Aerospace Engineering Department, Gaziantep, TURKEY.

<sup>2</sup>AssGaziantep University, Aeronautics and Aerospace Faculty, Pilotage Department, Gaziantep, TURKEY.

<sup>3</sup>Gaziantep University, Aeronautics and Aerospace Faculty, Aircraft and Aerospace Engineering Department, Gaziantep, TURKEY.

<sup>4</sup>Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY.

### Abstract

Penetration is the most critical concept in ballistic studies. Penetration is defined as the ability to penetrate the target. Penetrator tip geometry and velocity are the most important factors in terms of the penetration. There are many limitations in increasing the velocity of the penetrator. Therefore, projectile geometry can be studied in this field. So, in this study, six different tip geometry of bullet are investigated in terms of the ballistic impact performance. Numerical simulations are performed in ANSYS program, which is the finite element program. Explicit dynamics toolbox is used to perform the numerical study. Six different tip geometry of bullet was modeled in 3D modeling program. The geometries are transferred to the ANSY workbench program. Initial velocity is accepted as 300 m/s for all conditions. Within this study, the impact energy performance of six different bullet tip geometry was obtained. In addition, stress and deformation results on the target were also compared.

Keywords: Bullet, Impact performance, Nose geometry, Numerical analyses.

### **1. Introduction**

Penetration is the most critical concept in ballistic studies. Penetrator tip geometry and velocity are the most important factors in terms of the penetration. So, some studies were reviewed about the penetrator tip.

Wen et al. [1-2] formulated analytical equation to figure penetration quantity using different penetrators' tip shapes o puncturing target material.

Jordan has made ballistic investigations and studied residual velocity of penetrators impacting on target plate and viewed that the energy absorbed on the target material changes in different types of the bullets [3].

Jeng et al. [4] estimated the ballistic limit based upon law of conservation of energy for GFRP. Ballistic limit, damage evolution, penetration mechanism was analyzed by Ulven. Four particular penetrators were used in his study; hemispherical, conical, flat tip, fragment simulating [5]. Muslim Ansari studied experimental and finite element analyses of penetration of target plate, different projectile nose shapes. Also, result of experiment and series of analysis were compared in his study [6].

Muslim Ansari et al. concerned numerical an experimental of perforation of laminated composite. The effect of inclined impact on the ballistic rendition of target plate has been investigated by datate four impact angels is conducted with gas gun [7].



Eser SÖZEN at al. [9] presented that another factor affecting ballistic resistance as well as tip geometry of the penetrators is the weight and dimensions of the projectile.

The values obtained from the model and experimental data were compared in order to verify the model which provides the calculation of the distribution of pressure and bullet velocity in the barrel by Halil ISIK [10].

At the end of the literature review, it was obtained that penetrator tip shape is important parameter in terms of the penetration. So, bullet impact energy performance according to variable tip geometry was investigated.

### 2. Materials and Methods

There are many limitations in increasing the velocity of the penetrator. Therefore, projectile geometry can be studied in this field. So, in this study, six different tip geometry of bullet are investigated in terms of the ballistic impact performance. The geometries of the pellets were created by using SOLIDWORSK program as shown in Figure 1.



Figure 1. Pellets tip shape.

Pellets materials were assigned as lead material. In the analyses, initial velocity was accepted as 300 m/s for all pellets. Mesh independency was performed to validate the obtained mesh structure as shown in Figure2. After mesh accuracy operation, it was determined that the 18000-element number is suitable for these analyses.

Technology



Figure 2. Mesh İndependency

Mesh view was shown as Figure 3 after validation process for the pellet (a).



Figure 3. Sample mesh view for pellet (a)

ANSYS workbench explicit dynamic toolbox was used to perform the proposed study as shown in Figure 4. Pellet was accepted as rigid and plate was accepted as deformable.



Figure 4. Ansys workbench analysis model

Pellet velocity was accepted as 300 m/s for each pellet as shown in Figure 5.



Figure 5. Pellet velocity

Support type, which is fixed support, was applied to the model as shown in Figure 6.



Figure 6. Fixed support view

When the analysis was performed for the pellet (a) with 300 m/s initial velocity, exit velocity was obtained as maximum 259,7 m/s as shown in Figure 7.

F: pellet a	
Directional Velocity 2	
Type: Directional Velocity(Y Axis)	
Unit: mm/s	
Global Coordinate System	
Time: 8,3252e-005	
Cycle Number: 6075	
8.10.2019 14:52	
2,5976e5 Max 2,5975e5 2,5974e5 2,5973e5 2,5972e5 2,5971e5	
2,597e5 2,5969e5 2,5968e5	

**Figure 7.** Exit velocity of the pellet (a)

Exit velocity of the pellet (b) was found as 261,66 m/s after the analysis as shown in Figure 8.





Exit velocity of the pellet (c) was found as 278,81 m/s after the analysis as shown in Figure 9.



**Figure 9.** Exit velocity of the pellet (c)

Exit velocity of the pellet (d) was found as 285,21 m/s after the analysis as shown in Figure 10.



Figure 10. Exit velocity of the pellet (d)

Exit velocity of the pellet (e) was found as 274,73 m/s after the analysis as shown in Figure 11.



**Figure 11.** Exit velocity of the pellet (e)

Exit velocity of the pellet (f) was found as 277,75 m/s after the analysis as shown in Figure 12.



Figure 12. Exit velocity of the pellet (f)

At the end of the analyses, exit velocities of the pellets were determined according to the applied initial velocity.

### 3. Results and Discussion

Penetration concept is important in many ballistic studies. Penetration is defined as the ability to penetrate the target. Penetrator tip geometry and velocity are the most important factors in terms of the penetration. Exit velocity of the different tip shape pellets were given in Table 1.

Table 1. Exit velocity of the pellet						
Pellet Type	V <sub>e</sub> (m/s)					
Pellet (a)	259,76					
Pellet (b)	261,66					
Pellet (c)	278,81					
Pellet (d)	285,21					
Pellet (e)	274,73					
Pellet (f)	277,7					

Impact energy was calculated according to the initial velocity  $V_i = 300 \text{ m/s}$  by using energy equation (1).

$$E = \frac{1}{2}m(V_i^2 - V_e^2) \tag{1}$$

Calculated energy was shown in Table 2.



Figure 13. Comparison of the exit velocity according to tip shapes

When the Figure 13 was investigated, it was clearly seen that sharpen tip geometry (pellet (d)) has lowest velocity decrease due to geometrical properties. Maximum velocity decrease was found in Pellet (a) due to flat tip shape.

			<u> </u>	5/
Pellet Type	V <sub>i</sub> (m/s)	$V_e(m/s)$	Weight (gr)	Energy (J)
Pellet (a)	300	259,76	0,46	5,180691
Pellet (b)	300	261,66	0,38	4,091468
Pellet (c)	300	278,81	0,41	2,514322
Pellet (d)	300	285,21	0,57	2,466748
Pellet (e)	300	274,73	0,79	5,736754
Pellet (f)	300	277,7	0,64	4,122467

**Table 2.** Calculated energy according to the tip geometry

When the weight of the pellet was taken into account, maximum energy absorption was found in pellet (e) due to weight of the pellet. When the stress and deformation results were investigated, there is no meaningful result due to high velocity impact process.

# 4. Conclusions

Penetrator tip geometry and velocity are the most important factors in terms of the penetration. In this study, six different tip geometry of bullet are investigated in terms of the ballistic impact performance. The effect of the shape of the projectile on penetration was examined and the projectiles were considered rigid. Pellet (d) has minimum velocity decrease due to geometrical properties. So, pellet (d) needs minimum energy to penetrate the plate in terms of the ballistic impact performance.

### References

1. Wen HM. Predicting the penetration and perforation of FRP laminates struck normally by projectiles with different nose shapes. Compos Struck 2000;49:321-9

The International Conference of Materials and Engineering Technology

- 2. Wen HM. Penetration and perforation of thick FRP laminates. Compos Sci Technol 2001;61:1163-72.
- 3. Jordan, J.B. and Naito, C.J. (2014), "An experimental investigation of the effect of nose shape on fragments penetrating GFRP", Int. J. Impact Eng., 63, 63-71.
- 4. Jenq, S. T., Jing, H.-S., & Chung, C. (1994). Predicting the ballistic limit for plain woven glass/epoxy composite laminate. International Journal of Impact Engineering, 15(4), 451–464.
- Ulven, C., Vaidya, U. ., & Hosur, M. . (2003). Effect of projectile shape during ballistic perforation of VARTM carbon/epoxy composite panels. Composite Structures, 61(1-2), 143– 150.
- Ansari, M. M., & Chakrabarti, A. (2017). Influence of projectile nose shape and incidence angle on the ballistic perforation of laminated glass fiber composite plate. Composites Science and Technology, 142, 107–116. doi:10.1016/j.compscitech.2016.12.033
- Ansari, M. M., & Chakrabarti, A. (2017). Ballistic Performance of Unidirectional Glass Fiber Laminated Composite Plate under Normal and Oblique Impact. Procedia Engineering, 173, 161–168.
- 8. Nilakantan, G., Wetzel, E. D., Bogetti, T. A. and Gillespie, J. W. 2013. A Deterministic Finite Element Analysis of The Effects of Projectile Characteristics on The Impact Response of Fully Clamped Flexible Woven Fabrics. Composite Structures, 95, 191-201.
- Sozen, E, Gunduz, G, Imren, E. (2016). Balistik Panel ve Koruyucu Zırh Üretiminde Kullanılan Lif ve Kompozit Malzemeler. Bartın Orman Fakültesi Dergisi, 18 (2), 194-204. DOI: 10.24011/barofd.267304
- 10. Işık, H., Modeling the Ballistic Parameters in a Barrel, The Journal of Defense Sciences, 2016, 15(2): 157-177.



# INVESTIGATION OF FRICTION PROPERTIES BETWEEN PLASMA SPRAY CERAMIC COATED DISC-PAD INTERACTIONS

# RECEP AKYÜZ<sup>\*1</sup>, EKREM ALTUNCU<sup>2</sup>, ZEKERİYA KOLBASAR<sup>2</sup>, OZAN DEMİRDALMIŞ<sup>3</sup>

<sup>1</sup> TOFAŞ Turkish Automotive Company R&D Center, Bursa, TURKEY.
<sup>2</sup> Sakarya & App. Sci. Uni., Dept. Metallurgy and Materials Eng. Sakarya, TURKEY.
<sup>3</sup> Kale Balata R&D Center, Kocaeli, TURKEY.

## Abstract

In today's automotive sector; as a result of the increasing interest in electric vehicles, changes in brake systems are observed. The new trend is the need for long-lasting coating in regenerative cast iron brake disc systems. In order to increase disc life, thermal spray coating methods with higher wear resistance and longer corrosion life are obtained. The plasma spray process produces high quality coatings by a combination a high temperature, high energy heat source and high particle velocities. In this study, the cast iron brake discs (GG20) are coated with 250  $\mu$ m thick Alumina-Titania (Al2O3-TiO2) based ceramic powder by optimized plasma spray (F4 MB gun) parameters. The ceramic coated discs put on the test bench and disc-pad interaction characteristics (wear thickness loss, temperature- friction coefficient, torque, brake energy) was investigated during the dynamometer testing. In the investigations, it is understood that ceramic coatings can be used effectively in automotive brake systems. The friction coefficient between the disc and the pad is within the expected target value range ( $\mu$ :0.3-0.6). The friction coefficient remains stable with increasing temperature between two surfaces. When compared with discs with conventional paintbased corrosion protection, ceramic coated discs ensure better resistance against corrosion and wear performance.

Keyword: Thermal spray coating, brake disc, pad, friction coefficient

### 1. Introduction

International standards and environmental regulations force the automotive industry to use both vehicle weight reduction, energy efficient and environmentally friendly systems. Therefore, manufacturers have begun to be interested in wear-resistant coatings or surface treatments on discs. High thermal and mechanical stresses occur on the brake discs. Increasing stresses are converted to temperature rise and thermal gradients on the disc. For effective braking, the increased heat due to friction between the pad and the disc must be safely removed from the disc body. The ability to control the friction coefficient between the disc and the friction material within a certain limit range is crucial for brake safety. In order to reduce disc wear and prevent the release of brake dust impurities to the environment, variety of coatings are applied to the disc surfaces [1-2]. Thermal spray methods (plasma or flame spraying) for brake discs are versatile and very convenient coating technology. Metallic, ceramic and carbide-based coatings can be easily applied to disc surfaces. A wide variety of coating materials (as powder or wire form) and high energy thermal spray methods can be used to increase disc service life and reduce wear. Carbide based coatings which sprayed with HVOF technique includes WC-Co, WC-Co-Cr, WC-NiCr, NiCr-Cr2C3 could be extensively used in automotive industry for engine components: rods, gears, brake pistons, brake discs.



The cost-benefit feasibility is very important for OEM parts where there is intense competition under mass production conditions. In this context, both the coating method and the coating material costs are expected to be feasible. It is also expected to be technically satisfactory [3-5]. In this direction, Alumina- Titania based ceramic coatings were applied to brake discs surface by plasma spray method. Alumina based coatings have superior abrasion, corrosion resistance and high hardness [6]. The hardness and fracture toughness of the coating can be varied with the addition of titania content. In this case, another important point is the effect of ceramic coating on the heat distribution of the disc surface and the change of coefficient of friction against temperature increase. Therefore, the friction behavior of the disc coated with Al2O3-TiO2 was investigated at different temperatures.

## 2. Materials and Methods

Ceramic coatings were sprayed using a Sulzer–Metco F4 plasma torch in atmospheric conditions (APS) onto gray cast iron (DIN 1561: GG20) disc. The chemical components of GG20 grade include, C: 3.10-3.40%, Si: 1.90-2.30%, Mn: 0.60-0.90%, P  $\leq$  0.15%, S  $\leq$  0.15%. CE: 3.90-4.15. Plasma spraying parameters were kept identical for ceramic powders. Ceramic feedstock Al<sub>2</sub>O<sub>3</sub>–10-15% wtTiO<sub>2</sub> was used as the main coating and (Ni20Cr) powder was used as bond coating with an average of about 50 µm thick bond layer on the surface of the substrates to obtain better performance of the plasma sprayed Al<sub>2</sub>O<sub>3</sub>–TiO<sub>2</sub>. They are reported in Table 1.

Before spraying, discs were grit blasted with alumina to improve the mechanical adhesion of coatings. The resulting average surface roughness was about 5-8  $\mu$ m Ra as sprayed condition. Roughness value is Ra < 1  $\mu$ m after finishing coated surfaces. During the coating process, cooling was applied to avoid disc temperature exceeding 200 °C. Cooling during spraying was performed using either: compressed air, distributed by two air jets disposed on each side of the plasma gun, the air pressure being maintained at 6 bar. The coating was carried out at 75-125  $\mu$ m per pass. The total number of passes varies between 4-6, until the desired total coating thickness (300±50 $\mu$ m) is reached.

The ceramic coated disc was subjected to standard metallographic sample preparation and examined under a microscope. Coating thickness was measured by image analysis. The surface and cross-section microstructures of the coating are shown in Fig.1. Micro hardness measurements of the coating were carried out by applying 300 gr load and 10 sec. by Vickers method. Ceramic coated brake disc was tested in a dynamometer regarded procedure with a standard OEM brake pad. The friction behavior of coated discs was tested at different temperatures with dynamometer tests. The change of friction coefficient was observed. The factors affecting the braking performance were examined.





**Table 1.** Plasma Spray Parameters and disc – pads surface views

# 3. Results and Discussion

Plasma spray method was able to deposit ceramic based coating layer on cast iron brake discs with targeted thickness and surface quality. The parameters of spraying have an influence on mechanical properties like porosity, microhardness, and the microstructure. Microstructural characterization was carried out with scanning electron microscopy (SEM-EDX). From the cross-sectional microstructures, it can be seen that coatings consist of the lamellar built up from the molten droplets impinging on the disc surface.

The coating has a layered microstructure (Fig. 1), typical of plasma sprayed coatings, which is the result of full melting of the ceramic feedstock powder and its solidification as "splats" on the cast iron substrate. Technically, optimum spray parameters is important to ensure good adherence of coating bonding. Too short spraying distance will produce lower adherence due to overheating and resulting internal stress inside the coating. In contrast, too long spraying distance will decrease the adherence bonding due to cooling and deceleration of the particles flying in the plasma jet. Adhesion strength, porosity, micro hardness varied depending on the process parameters setting. In general, the design of experimental approaches in the optimization of plasma spray processes is highly beneficial. In this direction, optimum parameters can be obtained and reproducible quality coating



properties can be achieved. The important point to note here is that the coating properties may vary depending on the properties of the spray gun and coating material.



Figure 1. SEM micrograph of Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> coating a. top surface b. cross section

In the electron microscopy image, dark areas are alumina based and light areas are titania based. The average thickness of the top coating is  $300\pm15 \ \mu m$ , bond coating is  $50\pm10\ \mu m$ . The micro hardness of the coatings applied with optimum spray parameters is about  $900\pm50$  HV0.3. The titanium oxide content in the coating composition changes the micro hardness and fracture toughness of the coating. Micro hardness and adhesion properties decreases as titanium oxide content increases. The obtained micro hardness values are in good agreement with literature (APS:850 -950 HV0.3) and confirm the state-of-the-art quality of the coatings. The performance test of the ceramic coated brake disc and brake pad pair was performed in a special dynamometer which was established for developing friction material and testing the brake bench tests. The test results show that, the ceramic coated brake disc and brake pads exhibit quite good performance in lower temperatures in terms of wear. By increasing temperature where in the intersection of the brake disc and brake pads, not only increase the material loss but also reduces the friction coefficient (Fig. 2 a, b). In theory, the reduced friction coefficient might be caused brake fading when it decreases under 0,3. Therefore, it is accepted the safety zone as above 0,3 by some brake pad producers.



Figure 2. Ceramic coated brake disc and brake pad performance test **a.** friction coefficient trend vs temperature **b.** weight loss of brake pads vs temperature





Figure 3. Braking torque analysis interactions in dynamometer test a. braking torque trend vs friction coefficient **b**. effect of the increasing temperature and reducing  $\mu$  on braking torque

### 4. Conclusions

Alumina-Titania coatings are deposited on cast iron brake discs with an intermediate bond coat of NiCr by atmospheric plasma spraying and these coatings exhibit desirable coating characteristics. The parameters setting such as powder flow rate, plasma gas flow rates, current, and stand-offdistance has provided evidence to directly influence the properties and performance of Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub> coating. Brake discs were coated with optimized coating parameters and examined within the scope of dynamometer tests. The variation of the disc friction coefficient with the original pad varies depending on the disc temperature. Friction coefficient up to 400 °C is within the target limits. Above 500 °C, the coefficient of friction decreases dramatically. This decline is due to the degradation of the friction material. The coefficient of the friction should be relatively high but most importantly stable. The disc brake performance sensitivity against the dynamic changes in the pressure, speed and temperature. However, it should be noted that these test conditions are based on extreme braking conditions. Under normal vehicle driving conditions, discs do not remain for long periods of time at peak temperatures. The plasma spray coated discs exhibited the desired friction behavior and passed the performance tests successfully.

This study shows that, in condition of using such type of coating on iron brake disc it is required to develop a suitable friction material in order to prevent the sudden decrease of friction coefficient during high temperature braking.

### References

1. Sebastian Gramstat, Technological Measures for Brake Wear Emission Reduction: Possible Improvement in Compositions and Technological Remediation: Cost Efficiency, Non-Exhaust Emissions, (2018), 205-227.

Technology



- 2. M.R.W. Brakea, A.C. Halla, J.D. Madiso, Designing energy dissipation properties via thermal spray coatings, Surface & Coatings Technology 310 (**2017**) 70–78.
- 3. Matteo Federici, Cinzia Menapace, Alessandro Moscatelli, Stefano Gialanella, Giovanni Straffelini, Effect of roughness on the wear behavior of HVOF coatings dry sliding against a friction material, Wear, Volumes 368–369, (**2016**), 326-334
- 4. A. Demir, R. Samur, I. Kılıçaslan, Investigation of the coatings applied onto brake discs on disc-brake pad pair, Metalurgija 48 (**2009**) 3, 161-166
- 5. G. Sundararajan, SV. Joshi, L. Rama Krishna, Engineered surfaces for automotive engine and power train components, Current Opinion in Chemical Engineering, Vol. 11, (**2016**), 1-6.
- 6. B.Normanda, V. Fervela, C.Coddet, V.Nikitine, Tribological properties of plasma sprayed alumina–titania coatings: role and control of the microstructure, Surface and Coatings Technology, Vol. 123, Issues 2–3, (**2000**), 278-287.
- 7. V. Cirovic, D. Aleksendric, D. Mladenovic, Braking torque control using recurrent neural networks, Proc IMechE Part D: J Automobile Engineering Vol 226, Issue 6, (**2012**), 1-13.



aterials and Engineering Technology

## CENGIZ BOZADA<sup>\*1</sup>, MİKAİL ASLAN<sup>2</sup>

<sup>1</sup>Gaziantep Üniversitesi, Mühendislik Fakültesi, Fizik Mühendisliği, Gaziantep, Türkiye <sup>2</sup>Gaziantep Üniversitesi, Mühendislik Fakültesi, Metalürji ve Malzeme Mühendisliği, Gaziantep, Türkiye

### ÖZET

Nadir toprak hekzaborürlerin (REB<sub>6</sub>) mekaniksel;elastik, kafes-dinamik ve sertlikler, optiksel; iş fonksiyonu, elektronik yapıları ve termodinamik özellikleri,yoğunluk fonksiyon yöntemine (DFT) dayalı temel prensiplerden (Ab initio) yararlanılarak hesaplanabilir. DFT ile ayrıca bant yapıları, durum yoğunlukları, yük analizleri hesaplanabilir. Bu hesaplamalara göre, malzemenin yalıtkan, yarı iletken ve iletken özelliklerden birini gösterip göstermediği bulunabilir. Mekanik özellikleri araştırmak açısından kafes yapıları incelenerek malzemenin kırılgan veya yumuşak bir malzeme olup olmadığı bulunabilir. Ayrıca DFT yöntemini REB<sub>6</sub>'ların bazı optiksel özelliklerini (iş fonksiyonu gibi) bulmak için kullanılabilir. Örneğin, Sm katkılı LaB<sub>6</sub>'nın optik özelliklerini yorumlamak için çalışmalar mevcuttur. Bu çalışmalarda, Sm 4f durumlarının, Sm katkısından sonra LaB<sub>6</sub>'nın Fermi yüzeyindeki DOS'u değiştirdiğini ve iletken elektronların sayısının azaldığını gösterdiler. Biz bu çalışmada DFT yönteminin REB<sub>6</sub> malzemelerin mekaniksel, optiksel ve elektronik özelliklerini kullanımı üzerine odaklandık.

Anahtar Kelimeler: Nadir Toprak Hekzaborürler, DFT, Ab initio, Hesaplamalı Malzeme Bilimi, Malzeme Modelleme ve Simulasyon, Elekronik, Optiksel, Mekanik ve Termal Özellikler

#### 1.Giriş

Metal borürler, genel olarak, yüksek sertlik, yüksek ergime noktası, yüksek termal iletkenlik, yüksek elektriksel iletkenlik, düşük yoğunluk ve yüksek kimyasal kararlılık gibi özelliklere sahiptirler. Bu özellikler, ileri teknoloji alanlarında kullanım yeri bulmalarına neden olmaktadır. Metalurjiden elektronik sektörüne kadar çok geniş bir alanda kullanım yerleri bulunmaktadır.

Nadir toprak metallerinden oluşan metal hekzaborürler (REB<sub>6</sub>), düşük elektronik iş fonksiyonu, düşük elektriksel direnç ve termal genleşme katsayısı (bazı sıcaklık aralıklarında) ile birlikte yüksek sertlik ve rijitlik, yüksek kimyasal ve termal kararlılık ve erime noktaları gibi özelliklerinden dolayı endüstride geniş kullanım alanlarına sahiptir. Yüksek çözünürlüklü optik sistemler, kaynak teknolojisi, dedektörler, yüksek voltaja ve sıcaklığa dayanan metalik kaplamalar, termiyonik malzemeler, elektron mikroskoplar, X-ışını tüpleri, nükleer alanda koruyucu malzemeler bu alanlardan bazılarıdır.

DFT, genellikle gaz halindeki yalıtılmış moleküller, kristal ile nanoparçacıklı yapılar, periyodik ile arayüzeyler gibi yapıların araştırılmasında ve niteliklerin incelenmesinde kullanılan hesaplamalı biri metottur. DFT'nin çeşitli ilimsel problemlere çözümler üretmesi ve deneyler ile elde edilmeyen mühim bilgilerin elde edilebilmesi bu metotun tercih edilmesinin iki önemli faktörüdür[1].REB<sub>6</sub>, yüksek erime noktası, sertlik, kimyasal stabilite, düşük iş fonksiyonu, yüksek sıcaklıklarda düşük uçuculuk, süper iletkenlik, manyetik özellikler, verimlilik, termiyonik emisyon ve dar bant yarı iletkenliği gibi özelliklere sahiptir. Ayrıca fotonik ve elektronik uygulamalarda REB<sub>6</sub> nanoyapıları kullanılır[2].



## 2.Sonuç Ve Tartışma

REB<sub>6</sub>'nin mekanik, optiksel özellikleri, elektronik yapıları ve sertlikleri, temel prensip yönteminden biri olan DFT'den yararlanılarak hesaplanılabilir[3]. Lihua Xiao ve diğerleri, SmB<sub>6</sub>'nın teorik sertliğini, mekanik ile optik özellikleri ve elektronik yapıları DFT kullanarak temel prensiplerden hesapladı. Hesaplanan sonuçların önceden bildirilen deneyler ve teori ile mükemmel uyum içinde olduğu görüldü.SmB<sub>6</sub>'nın bant yapıları bu malzemenin en az boşluklu yarı iletken özelliklere sahip olduğunu gösterdi.Hesaplanan sonuçlar SmB<sub>6</sub>'nın kırılgan bir malzeme olduğunu gösterdi[4]. Farklı bir çalışmada Qi ve diğerleri, nanokristal Ca katkılı CeB<sub>6</sub>'nın ayarlanabilir optik özelliklerini açıklamada; bant yapının, durum yoğunluğunun ve optik özelliklerinin analizlerini yapmak içinDFT çerçevesinde temel prensip hesaplamayı kullandılar. Hesaplamada, 2 × 2 × 2 supercell ( süper hücre ) ile Ce<sub>0.875</sub>Ca<sub>0.125</sub>B<sub>6</sub> seçildi ve bant yapısı hesaplama sonuçları, Fermi seviyesinin enerji ölçeğinin sıfır konumu olarak seçildiği Şekil-1 'de gösterildi. İletken bandın Fermi seviyesinin üzerinden geçtiği ve bu malzemenin tipik bir iletken davranış gösterdiği Şekil- 1'de gösterilmiştir[5].

aterials and Engineering Technology



**Şekil-1:**Ce<sub>0.875</sub>Ca<sub>0.125</sub>B<sub>6</sub>'nın bant yapısının elektronik yapısı Adopted from[5]Copyright © 2017 Elsevier B.V.

Diğer bir çalışmada ise Luomeng Chao ve diğerleri, DFT'yi Sm-katkılı LaB<sub>6</sub>'nın optik özelliklerini yorumlamak için kullandılar ve Sm 4f durumlarının, Sm dopinginden sonra LaB<sub>6</sub>'nın Fermi yüzeyindeki DOS'u değiştirdiğini ve iletken elektronların sayısının azaldığını gösterdiler[6].

Başka bir çalışmada ise Guo-Liang ve diğerleri, LaB<sub>6</sub>'nın elastik ve termal özelliklerini, DFT çerçevesinde, yarı harmonik Debye modeliyle araştırdılar[7]. Ayrı bir çalışmada ise Tanju Gürel ve diğerleri, titreşimlerin anharmonikliğini (bir sistemin harmonik osilatör olmaktan sapmasıdır) hesaba katan yarı harmonik yaklaşımı kullanarak LaB<sub>6</sub> ve CeB<sub>6</sub>'in elastik, kafes-dinamik ve termodinamik özelliklerinin araştırmalarını yaptılar. Bu çalışmada DFT ve perturbasyon teorisini (tam olarak çözümlenmeyen bir problemin, bu probleme bağlı farklı bir problemden yola çıkarak yaklaşık bir çözüm elde etmek için matematiksel metotlar içeren teori) kullandılar[8]. Ayrı bir çalışmada ise Schmidt ve diğerleri, LaB<sub>6</sub> için moleküler dinamik ve DFT hesaplamalarından faydalanarak geliştirilmiş interatomik (atomlar arası) potansiyelleri literatüre sundular. Bu çalışmada, DFT teorisi, çeşitli konfigürasyonlarda ve ortamlarda atomların enerjik ve dinamik verilerini elde etmek için kullanıldı. Bağlanma enerjisi şekil 2 'de gösterilmiştir [9].



**Şekil-2:** DFT'den LaB<sub>6</sub> için bağlanma enerjileri Adopted from[9]Copyright© 2015 American Chemical Society.

Farklı bir çalışmada ise Guo-Liang ve diğerleri, yarı-harmonik Debye modeli ve DFT çerçevesinde genelleştirilmiş gradyan yaklaşımını kullanan düzlemsel dalga psödopotansiyel metodunu (birçok problemi basite indirger), LaB<sub>6</sub> kristalinin kütle modülüne, 1s1l genleşmesine ve 1s1 kapasitesi çalışmasına uyguladılar[7]. DFT[11] temelli temel prensip hesaplamalarını Cambridge üniversitesi tarafından geliştirilen CASTEP kodunu[10] kullanarak yapılmıştır. Ayrıca DFT'ye dayanarak REB6'nın elektronik yapılarını, elastik, fonon ve termodinamik özelliklerini incelemek için temel prensip hesaplamaları Huang ve diğerleri tarafından yapıldı. Elde edilen bant boşlukların, kafes sabitinin değerlerine hassas bir şekilde bağlı olduğunu belirlediler[12].Son yıllarda, enerji-gerilme iliskilerini elde etmek için bir kristalli sisteme uygulanan bir dizi homojen deformasyon kullanılarak, tek kristallerin ikinci dereceden elastik sabitlerini (SOEC), üçünü dereceden elastik sabitlerini (TOEC) ve daha yüksek dereceli elastik sabitlerini başarılı bir şekilde belirlemek için temel ilkeler hesaplamalarını Viyana Ab initio simülasyon paketi (VASP) kodu kullanılarak yapılmıştır [13-14]. Zeng ve diğerleri, LaB<sub>6</sub> ve CeB<sub>6</sub>'nın SOEC'leri ve TOEC'lerini hesapladılar. Hesaplanan kafes parametreleri, SOEC'ler ve kütle modülleri, mevcut deneysel ve teorik değerlerle uyumlu bulundu. Hesaplanan elastik sabitlerden, etkili SOEC'lerin basınç türevleri, yapısal stabilite ( kararlılık), mekanik özellikler ve elastik anizotropi (eşyönsüzlük) üzerindeki basınç etkileri ile birlikte incelendi.Şekil-3'de gösterilen SOEC'lerin ve TOEC'lerin belirlenmesi için gerilme parametresine karşı gerilme parametresi eğrileri ve çeşitli gerilmelerin altındaki eğri parametreleri gösterilmiştir. Ayrık noktalar ve düz çizgiler, temel prensip hesaplamalarından elde edilen sonuçları gösterir. Gerilme enerjisinin gerginlik üzerindeki bu bağımlı eğrileri, sonlu gerginlik elastik deformasyonu altında beklenen davranış olan asimetrinin özelliklerine sahiptir. Negatif gerilmelere sahip gerilme enerjileri her zaman pozitif gerilmelere sahip gerilme enerjilerden daha büyüktür ve dolayısıyla TOEC'leri tipik olarak negatiftir. Bu durum, kafes titreşimlerine katkılarının en büyük olduğunu ve aynı zamanda anizotropilerinin en yüksek olduğunu gösterir[15].



**Şekil-3:**(a) CeB<sub>6</sub> ve (b) LaB<sub>6</sub>için Gerilme-enerji ilişkileri Adopted from[15] Copyright©2017 by the authors. Licensee MDPI, Basel, Switzerland.

Ayrı bir çalışmada Wills ve diğerleri, deneysel verilerin basınç etkilerini analiz etmek için EuB<sub>6</sub> ve  $GdB_6$ 'nın hacme bağlı bant yapılarını,  $EuB_6$  ve  $GdB_6$ 'nın paramanyetik (PM), ferromanyetik (FM) ve antiferromanyetik (AF) fazlarını hesapladılar[16]. Diğer bir temel prensip çalışmasında Bai ve diğerleri, CeB<sub>6</sub> ve EuB<sub>6</sub>'nın yapısal özelliklerini ve kimyasal bağ özelliklerini EXCITING[17] kodu ile araştırdılar. Hesapladıkları kafes sabitlerini ve bulk modüllerini deneysel sonuçlarla iyi bir uyum içinde olduklarını buldular[18].Optiksel özelliklerin araştırılmasıyla ile ilgili bir çalışmada ise Chao ve diğerleri, Yb katkılı LaB<sub>6</sub> optik özelliklerini DFT çerçevesinde temel ilke hesaplamaları ile araştırdılar.Araştırmanın sonuçlarına göre, Yb 4f'nin, yakın Fermi yüzeyindeki durumların optik özelliklerini etkilediğini ve Yb-dopingin, LaB6'nın plazmon (plazma salınımı) enerjisinde bir düşüşe yol açtığını, yani görünür-yakın kızılötesi bölgede iletim tepe pozisyonunun kırmızıya kaydığını tespit ettiler[19].Ayrı bir çalışmada ise Lui ve diğerleri,CeB<sub>6</sub>/Al arayüzünü eriterek eğirme ile hazırlanan bir Al-Ce-B alaşımındaki mikro yapısını taramalı elektron mikroskobu (SEM) ve transmisyon elektron mikroskobu (TEM) ile incelediler. Erimiş Aluminyumda bulunan sentetik CeB6 partikülünün, yüzeyin (220) düzlem olan kübik morfolojiyi gösterdiğini belirlediler[20].Ning ve diğerleri,  $LnB_6$  (001) (Ln = Pr, Nd ve Gd) yüzeylerinin iş fonksiyonlarını sistematik olarak hesapladılar. LnB<sub>6</sub>'nın (100) düzlemindeki çalışma fonksiyonunda  $\Phi(PrB_6) \leq \Phi(NdB_6) \leq \Phi(GdB_6)$ ilişkisine uyduğunu gösterdiler. Tek kristallerin kristal kalitesi ve emisyon özelliklerini, dönen hedef X ışını kırınımı, X ışını tek kristal kırınımı, X ışını Laue kırınımı ve termal elektron emisyon ölçümleri ile karakterize ettiler. Buna göre LnB6( Ln=Pr, Nd ve Gd ) (001) yüzeyindeki iş fonksiyonları ise Şekil- 4'de gösterilmektedir. Yapısal gevsemeden önce siyah noktalarla gösterilen PrB<sub>6</sub>, NdB<sub>6</sub> ve GdB<sub>6</sub>'nın çalışma işlevleri sırasıyla 2.84 eV, 2.87 eV ve 2.96 eV'dir. Geometri optimizasyonundan sonra, kırmızı lekeler ile gösterilen iş fonksiyonları sırasıyla 2.38 eV, 2.47 eV ve 2.71 eV'ye düşer[21].



Şekil-4: HesaplananLnB<sub>6</sub> (Ln=Pr,Nd ve Gd) (001) yüzeyindeki iş fonksiyonları Adopted from[21]Copyright© 2019 WILEY-VCH Verlag GmbH&Co. KGaA, Weinheim

Farklı bir çalışmada ise Liu ve diğerleri ikili ve üçlü REB6'nın (LaB6, CeB6, GdB6, NdB6, La<sub>0.75</sub>Ce<sub>0.25</sub>B<sub>6</sub>, La<sub>0.75</sub>Nd<sub>0.25</sub>B<sub>6</sub>, La<sub>0.75</sub>Gd<sub>0.25</sub>B<sub>6</sub>, Ce<sub>0.75</sub>Gd<sub>0.25</sub>B<sub>6</sub>) kristal elektronik yapısını (bant yapısı ve durum yoğunluğu) DFT'yi temel alarak temel prensip hesaplamaları ile yaptılar. DOS içeren REB6'nin metalik özelliğe sahip olduğunu ve büyük DOS nadir toprak elementleri ile sağlandığı sonucuna vardılar. Fermi enerji seviyesi, büyük DOS ve Fermi enerji seviyesine yakın RE atomlarının aktif valans elektronunun konumu, REB6'nın elektron emisyon performansını belirlediğini tespit ettiler. REB<sub>6</sub> tek kristallerinin termiyonik emisyon testinin sonuçları, gerçek performansın temelde hesaplanan değere uygun olduğunu gösterdiler[22].NdB<sub>6</sub> ve LaB<sub>6</sub>'nın temel durum kristal yapıları, DFT teorisi çerçevesinde Kohn ve Sham tarafından optimize edilmiştir. Kendiyle tutarlı toplam enerji, temel ilkeler hesaplamaları ile gerçekleştirdiler[10]. Ayrı bir çalışmada ise Liu ve diğerleri, tek kristalli LaB6 tipik kristal yüzeylerin kristal elektronik yapısını ve çalışma fonksiyonlarını DFT'ye dayanan temel prensip hesaplamaları ile LaB<sub>6</sub>'nın kristal elektronik yapısını (bantların yapısı ve durumlarının yoğunluğu), Malzeme Stüdyosu'ndaki[11] CASTEP (Cambridge Seri Toplam Enerji Paketi) hesaplama koduyla gerçekleştirilmiştir) sistematik olarak incelediler. Elektronik yapı hesaplamalarına göre, LaB6'nın metalik bir özelliğe sahip olduğunu ve LaB6'nın Fermi seviyesine yakın yüksek durum yoğunluğunun temel olarak La 5d, 6s ve B2p'den oluştuğunu gösterdiler. Tipik (100), (110), (111), (210), (211), (310) yüzey hesaplamasının iş fonksiyonları, en yüksek La konsantrasyonlu (100) yüzeyin düzenli simetrik yüzey yapısına sahip olduğunu gösterdiler.Şekil-5(a) ve (b), LaB<sub>6</sub> için hesaplanan durumların toplam ve kısmi yoğunluğunu gösterir. LaB<sub>6</sub> için hesaplanan enerji bandı yapısı, Şekil-5(c)'de gösterilmiştir. Fermi seviyesi 0 eV olarak ayarlanır, diğer enerji seviyeleri Fermi seviyesine göre belirlenir.LaB6'nin kristal yapısı Şekil-5(d)'da gösterildi. [23].



Şekil-5:LaB<sub>6</sub>'nın (a) kısmi DOS'u, (b) DOS, (c) Elektronik bant yapısıve (d) Kristal Yapısı Adopted from[23] Copyright © 2017 Elsevier Ltd.

# 3.Sonuç

REB<sub>6</sub>'nın mekanik ile optik özellikleri, sertlikleri ve elektronik yapıları, elastik, kafes-dinamik ve termodinamik özellikleri,çeşitli konfigürasyonlarda ve ortamlarda atomların enerjik ve dinamik verilerini elde etmek için yoğunluk fonksiyon yöntemi (DFT) kullanılarak temel prensiplerden hesaplatıldı. Bu hesaplamalara göre bant yapılarının yarı iletken özelliklere sahip olduğu, kırılgan bir malzeme olduğu ve iletken bir davranış gösterdiğini belirlediler. Sm 4f durumlarının, Sm dopinginden sonra LaB<sub>6</sub>'nın Fermi yüzeyindeki DOS'u değiştirdiğini ve iletken elektronların sayısının azaldığını gösterdiler.

# **Referans:**

- **1.** Aslan, M. Bimetalik Cu Pt Nanoparçacıkların Stabilitesi, Yapısal ve Elektronik Özellikleri. DÜMF Mühendislik Dergisi, **2019**,10(2): 511-522.
- 2. Ji, X. H., Zhang, Q. Y., Xu, J. Q., & Zhao, Y. M. Rare-earth hexaborides nanostructures: recent advances in materials, characterization and investigations of physical properties. Progress in Solid State Chemistry, 2011, 39(2): 51-69.
- **3.** Li, C., Wang, B., Li, Y., & Wang, R. First-principles study of electronic structure, mechanical and optical properties of V<sub>4</sub>AlC<sub>3</sub>. Journal of Physics D: Applied Physics, **2009**, 42(6): 065407.



E

- 5. Qi, X., Bao, L., Chao, L., & Tegus, O. Experimental and theoretical investigation on tunable optical property of nanocrystalline Ca-doped CeB<sub>6</sub>. Physica B: Condensed Matter, **2018**,530: 312-316.
- 6. Chao, L., Bao, L., Shi, J., Wei, W., Tegus, O., & Zhang, Z. The effect of Sm-doping on optical properties of LaB<sub>6</sub> nanoparticles. Journal of Alloys and Compounds, **2015**, 622:618-621.
- 7. X. Guo-Liang, C. Jing-Dong, X. Yao-Zheng, L. Xue-Feng, L. Yu-Fang, and Z. Xian-Zhou, Chin. Phys. Lett.,2009,26:056201
- 8. Gürel, T.,& Eryiğit, R..Ab initio lattice Dynamics and thermodynamics of rare-earth hexaborides LaB<sub>6</sub> and CeB<sub>6</sub>. Physical Review B.,**2010**, 82(10): 104302.
- 9. Schmidt, K. M., Graeve, O. A., & Vasquez, V. R. Ab initio and molecular dynamics-based pair potentials for lanthanum hexaboride. The Journal of Physical Chemistry C.,2015, 119(25): 14288-14296.
- 10. Kohn, W., & Sham, L. J. "Self-Consistent Equations Including Exchange and Correlation Effects,".Phys. Rev., **1965**, vol. (140)
- 11. M.D. Segall, P.J.D. Lindan, M.J. Probert, C.J. Pickard, S.J. Clark, M.C. Payne, J.Phys. Condens. Matter, 2002, 14:2717.
- 12. Huang, B., Duan, Y. H., Sun, Y., Peng, M. J., & Chen, S. Electronic structures, mechanical and thermodynamic properties of cubic alkaline-earth hexaborides from first principles calculations. Journal of Alloys and Compounds, 2015, 635:213-224.
- 13. Zhao, J.J.; Winey, J.M.; Gupta, Y.M. First-principles calculations of second- and third-order elastic constants for single crystals of arbitrary symmetry. Phys. Rev. B., 2007, 75: 94105-94111
- 14. Wang, H.;Li, M. Ab initio calculations of second-,third-,and fourth-order elastic constants for single crystals. Phys. Rev. B., 2009, 79:224102-224111
- 15. Zeng, X., Ye, Y., Zou, S., Gou, Q., Wen, Y., &Ou, P. First-Principles Study of the Nonlinear Elasticity of Rare-Earth Hexaborides REB<sub>6</sub> (RE= La, Ce). Crystals, **2017**, 7(11): 320.
- 16. [16] J.M. Wills, M. Alouani, P. Andersson, A. Delin, O. Eriksson, A. Grechnev, Full Potential Electronic Structure Method.Berlin, Springer, 2010.
- 17. Gulans, A., Kontur, S., Meisenbichler, C., Nabok, D., Pavone, P., Rigamonti, S., & Draxl, C..Exciting: a full-potential all-electron package implementing density-functional theory and many-body perturbation theory. Journal of Physics:CondensedMatter, 2014, 26(36): 363202.
- 18. Bai,L.,&Ma, N. .GGA+ U method investigating structural and chemical bond properties of CeB<sub>6</sub> and EuB<sub>6</sub>. Physica B: Condensed Matter, **2010**, 405(22):4634-4637.
- 19. Chao, L., Bao, L., Wei, W., & Tegus, O. Optical properties of Yb-doped LaB<sub>6</sub> from firstprinciples calculation. Modern Physics Letters B., 2016, 30(07):1650091.
- 20. Liu, S., Cui, C., Wang, X., Han, C., Chen, H., &Shi, J..Interfacial microstructure and nucleating mechanism of melt-spun CeB<sub>6</sub>/Al composite inoculant. Applied Surface Science, **2018**,431:202-206.
- 21. Ning, S. Y., Iitaka, T., Xu, D. D., Li, Z., Wang, Y., Yang, X. Y., & Zhang, J. X.. Preparation and Properties of High-Quality Ce<sub>x</sub>La<sub>y</sub>Pr<sub>y</sub>Nd<sub>0.05</sub>Gd<sub>0.05</sub>B<sub>6</sub> Single Crystal by Optical Float-ZoneTechnique. Physica status solidi (a).,2019,216(4): 1800706.
- 22. Liu, H., Zhang, X., Xiao, Y., & Zhang, J. The electronic structures and work functions of (100) surface of typical binary and doped REB<sub>6</sub> single crystals. Applied Surface Science, 2018, 434:613-619.
- 23. Liu, H., Zhang, X., Ning, S., Xiao, Y., & Zhang, J. The electronic structure and work functions of single crystal LaB<sub>6</sub> typical crystal surfaces. Vacuum, **2017**, 143: 245-250.

190

# **PRINCIPLES AND FINITE ELEMENT SIMULATION OF MULTI-POINT** FORMING TECHNOLOGY FOR SHEET METAL

5

The International Conference of Materials and Engineering Technology

# Mahmut TANDOGAN<sup>\*1</sup>, Omer EYERCIOGLU<sup>2</sup>, Mustafa DULGER<sup>3</sup>

<sup>1</sup>Adiyaman University, Technology Faculty, Mechatronics Engineering Department, Adiyaman, TURKEY. <sup>2</sup>Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep,

TURKEY.

<sup>3</sup> Istanbul Cerrahpasa University, Engineering Faculty, Mechanical Engineering Department, Istanbul, TURKEY.

### Abstract

Multi-point sheet metal forming is a relatively new and developing flexible sheet metal forming technology. In this type forming, die is separated into small discrete pins which can be arranged in height. So, this reconfigurable die meets the customer requirements mainly used in prototyping of sheet metal parts. Dies in conventional forming process are replaced with these pair of matrices of pins. In this study, the fundamental principles of multi-point forming (MPF) are examined and main parameters of this process are determined with numerical analysis. In FE modeling each pin is actively controlled to form the sheet part. Aluminum 1100 H14 with 1 mm sheet is used as workpiece material for sheet metal part and finite element simulations are employed then, differences between solid die forming and multi-point forming are indicated. Tearing and dimple formation are investigated and compared with conventional method. The results of the numerical analysis show the multi-point forming is very suitable for forming the sheet metal with complex shape.

Keyword: Forming, Multi, Point, Finite Element.

UWE

### 1. Introduction

Three dimensionally formed sheet metal parts are widely used in aerospace, aircraft, automobile and architectural industry. These parts are produced by using metal forming dies. During the design and development of them, prototypes are prepared. Conventional prototyping methods are time consuming and so as results in high production costs. Many dies must be manufactured and small changes of the part geometry, requires new die components. These problems can be overcome by using multi-point forming (MPF) technique, also called as reconfigurable die forming. Solid dies in conventional forming methods are divided to many discrete pins, utilizing punch elements to form the sheet metal instead of solid dies. This means that MPF dies are used to shape different geometries using one die set by adjusting the pins in dies. First idea was introduced by Nakajima; discrete pins are mounted on headstock of NC milling machine then all pins pressed to sheet metal [1]. Discrete pin dies were arranged to desired shape, fixed into a rigid tool which behaves as solid die set by using NC system [2, 3]. Deformation characteristic, fundamental principles and some defects were investigated with describing four main types of multi-point forming. Non-spingback forming was obtained due to step-by-step forming [4]. Numerical analysis for multi-point forming was described by the basis of updated Lagrangian formulation and elastic-plastic material model. Contact points between sheet and pins are discontinuous and was modelled based on the penalty method and on the Coulomb non-classical friction law of elastic-plastic formalism. Finite element package code was newly developed for analyzing the multi-point sheet metal forming. Applicability of the algorithm was showed as a result and numerical examples stated good general coincidence with experiments



reported in the literature [5]. Multi-step and sectional forming of MPF were applied for dish head to obtain principles and characteristics of these methods. Numerical simulations were investigated for both methods. Multi-step forming deformed the sheet more equably. It can restrain wrinkle effectively and improve the deformation capacity by optimizing the deformation path [6]. The principles of multi-point die forming (MPDF) and multi-point press forming (MPPF) are described. Two group of forming pins are changed with continuous male and female dies. For any shape changes in sheet metal part, all elements' position was set by computer control. CAD software, two element arrangements, computer control system, press and CMM are the components of MPDF. The results show that three dimensional sheet metal parts were formed without tearing [7]. Reconfigurable bottom die and a rubber top die were utilized in multi-point sandwich forming (MPSF). The effect of workpiece and tool dimensions and workpiece properties were investigated. Two types of elastic material and three types of sheet metal were used in experiments. The results show that increasing of thickness reduces the dimple formation and increases the forming load. Also rubber pad stiffness has an effect on the geometry of final product [8].

The International Conference of Materials and Engineering Technology

In this study, two types of MPF of sheet metal arrangement are used. Forming operations are simulated numerically by using DEFORM 2D finite element package. In finite element modeling each pins are actively controlled. Effect of pins geometry on final geometry is investigated. Tearing or damage factor, dimple formation are compared with each other and conventional forming method.

## 2. Finite Element Simulations

DEFORM<sup>TM</sup> 2D finite element package is used in this study. This software is mainly adapted to forming operations. Pre-Processor (modelling or input data section), Simulator and Post Processor (results section) are the three levels of finite element package. Plane strain condition is used and cold forming is applied for simulations. The die and pins were modelled as a rigid body, because the stress and strain of the die were not analyzed. Due to this any material is not attached and mesh was not created in DEFORM for rigid bodies. This is usable application to reduce the running time in simulations. The Newson-Rapson Method and the Langrangian incremental type were used for iteration method and the solver, respectively.

# 2.1 Multi-Point Forming Arrangements

MPF dies are includes the bottom or female die sheet part and forming pins. Sheet metal part and bottom die geometries are shown in Figure 1 and Figure 2, respectively. Two types of multi-forming dies were used in simulations. Arrangement 1 includes 13 pins with 10 mm diameter. Other arrangement has 25 pins with 5 mm diameter tip. The distances between the pins are 1 mm for both arrangements. Pin geometries are shown in Figure 3.



Figure 1. Sheet Final Shape


30 40 20

all dimensions are in mm.





Figure 3. Pin Geometries

Both arrangement simulations are shown Figure 4 and Figure 5, respectively.



Figure 4. Arrangement 1



Figure 5. Arrangement 2

## 2.1 Workpiece Material

1 mm thick Aluminium 1100 H14 sheet is used as workpiece material. The workpiece was modelled as elasto-plastic material and 500 2D quadratic mesh element were generated by tool called automatic mesh generator in DEFORM. The bottom die and pins are not investigated in simulations so they were modelled as rigid body. This feature reduces running time in finite element simulations. The flow stress-strain curve existing in the finite element package database and the experimentally determined one from tensile test are shown in Figure 6. These curves are very similar to each other. Fracture or tearing criteria is also important factor for simulations. Normalized Cockroft & Latham (1968) was chosen as the fracture criterion. Normalized Cockroft & Latham criteria predicts the fracture strain more precisely than the other criterions in metal forming processes [9]. So, damage factor is defined as 0.34 taken from tensile test.



Figure 6. Flow stress – Strain Curve of Al 1100



## 3. Results and Discussion

In arrangement 1, left end and right end pins firstly pressed to sheet metal. This movement behaves like a blank holder. After that center pin is descended and formed the sheet metal. Then, pins correspond the inclined area were activated and pressed to sheet metal. All forming operations for both arrangements is 0.5 mm/sec. Final shape of forming operations and pin numbers according to pressing is shown in Figure 7.

aterials and Engineering Technology



Figure 7. Final Shape in Arrangement 1

Damage factor was not reach the defined value. So, there is no tearing on sheet metal. Final damage factor obtained as 0.0523 is shown in Figure 8.



Figure 8. Highest Damage Factor Value

Same procedure was applied to arrangement 2 and also more pins were pressed to sheet to get final shape. Figure 9 shows the final shape in arrangement 2 and pin numbers according to pressing.



Figure 9. Final Shape in Arrangement 2



In this forming operation, damage factor is obtained as 0.0808 (see Figure 10). Same as other arrangement tearing was not observed. But the damage value is higher than arrangement 1. Shape formation is also better than arrangement 1 due to more forming contact points.



Figure 10. Highest Damage Factor Value

Dimples is the one of the main problem in MPF of sheet metal [10]. Pin compresses the sheet and crushed area was occurred due to concentrated loads. Elastic cushion can be used to overcome this problem.

Conventional forming process (solid die) was also performed in simulation to compare with MPF. Male die and female die were used to form sheet. Same forming rate and friction value was defined as in MPF simulations. For this type forming, damage factor was taken as 0.118. So again tearing not observed but higher than MPF. When complexity of sheet metal part is increased, tearing possibility of sheet part is also increased. Final shape of conventional forming is shown in Figure 11.



Figure 11. Final Shape of Conventional Forming

## 4. Conclusions

Finite element simulation of basic MPF is presented in this paper. As a result of numerical simulations, the followings can be concluded:

- 1. This method is mainly useful for complex sheet metal parts and prototyping of the sheet metal products.
- 2. Manufacturing cost of sheet metal parts are reduced due to readjusting of discrete pins. One die set can be useful for different sheet metal geometries.
- 3. Pin geometry is also important to form sheet metal. Smaller forms better but increases damage factor. Also, initial cost of press construction is higher because of controlling of each pin.
- 4. Dimple formation is the one of the problem which must be overcome.

#### **References:**

1. Nakajima N., A newly developed technique to fabricate complicated dies and electrodes with wires. J Japan Soc Mech Eng, **1969**, 72(603):498–506.

The International Conference

aterials and Engineering Technology

- 2. Hardt D.E. and Gossard D.C., A variable geometry die for sheet metal forming: machine design and control. Proc Jt Autom Control Conf., **1980**, USA No.2, FP7–C:1–5.
- 3. Webb R.D. and Hardt D.E., A Transfer Function Description of Sheet Metal Forming for Process Control. Trans ASME, J Eng Ind, **1991**, 113:44–52.
- 4. Li M. and Liu Y., Multi-point forming: a flexible manufacturing method for a 3-d surface sheet. J Mater Process Technol, **1999**, 87:277–280.
- 5. Cai Z.Y. and Li M.Z., Finite element simulation of multi-point sheet forming process based on implicit scheme. J Mater Process Technol, **2005**, 161(3):449–455.
- 6. Qian Z.R., Li M.Z. and Tan F.X., The analyse on the process of multi-point forming for dish head, Journal of Materials Processing Technology, **2007**,187: 471-475.
- 7. Liu C., Li M. and Fu W., Principles and apparatus of multi-point forming for sheet metal, Int J Adv Manuf. Tech. **2008**, 35:707-714.
- 8. Zhang Q., Wang Z. R. and Dean T. A., The Mechanics of Multi-Point Sandwich Forming. Int. J. Mach. Tools Manuf., **2008**, 48:1495–1503.
- Engin K. E. and Eyercioglu O. Investigation of the Process Parameters of Sheet Metal Blanking Process by Using Finite Element Method, International Conference on Advanced Technology & Sciences (ICAT'16), 2016, v4:1141-1145.
- 10. Cai, Z. Y., Wang, S. H., Xu, X. D. and Li, M. Z., Numerical Simulation for the Multi-Point Stretch Forming Process of Sheet Metal. J. Mater. Process. Technol., **2009**, 209: 396–407.

# ABRASIVE FLOW MACHINING OF FIBER EXTRUSION SPINNERET HOLES

he International Conference

## ADEM AKSOY<sup>\*1</sup>, OMER EYERCIOGLU<sup>1</sup>, KURSAD GOV<sup>2</sup>

<sup>1</sup> Gaziantep University, Faculty of Engineering, Mechanical Engineering Department, Gaziantep, TURKEY.

<sup>2</sup> Gaziantep University, Department of Aeronautics and Astronautics Engineering, Gaziantep, TURKEY.

#### Abstract

Abrasive flow machining (AFM) is a non-traditional surface finishing method which is extrudes abrasive media through the surface. Rising surface quality demands and developing manufacturing technologies provide high costs and time. In this study, abrasive flow machining is applied to micro holes. Spinneret is a set of nozzles which is used for all types of fiber extrusion of yarn, staple, spunbond, meltblown and spunlace applications. Spinneret has various types of small holes. These holes are generally produced by electrical discharge method. The nozzles have bigger and circular entrance while smaller and shaped holes at the exit. In this study, finishing of two types of spinnerets (round-hole and trilobal-hole) by one-way AFM process subsequent to EDM manufacturing was presented. The quality of the holes before and after AFM process was compared. The results show that AFM process is an effective method for finishing of spinneret holes. The residual asperities are smoothed or even completely removed while keeping the geometry of the exit.

Keyword: Abrasive flow machining, surface finishing, micro holes, spinneret, nozzle.

#### Introduction

Abrasive flow machining (AFM) is a non-traditional surface finishing method which is extrude abrasive media through the surface. Rising surface quality demands and developing manufacturing technologies provide high costs and time. AFM method is literally adequate option for these demands. Complex shapes and micro holes can be finished easier than conventional methods by AFM method.

Conventional surface finishing methods are limited to simple shaped geometries as circular, rectangular or flat surfaces (lapping, honing, polishing, grinding, etc.). Due to processing of complex shaped surfaces, surface roughness, sharpness of the corners and fillets cannot be obtained with the desired precision and homogeneity. Abrasive fluid surface treatment method is an effective method to minimize these undesired problems [1-2].

Abrasive media is a mixture of abrasive particle and abrasive carrier. It contains silicon carbide (SiC) or aluminum oxide ( $Al_2O_3$ ) particles, polymer, hydraulic oil and water. This mixture makes the operation easy. Non-newtonian abrasive media is extruded through the surface by

high pressure. This pressure makes the particles touch the surface and remove asperities. Abrasive media can flow easily in small holes and reach all the surfaces on complex shapes [3].

Technology

Small holes are generally produced by electrical discharge machining (EDM) method, although other micro-hole drilling methods (machining, electron-beam, laser beam etc.) may be used. During the EDM process, all of melted material cannot be removed; some of them solidifies again and creates a recast layer also known as white layer. There are large number of micro- cracks, asperities and pores on the surface of this layer, which cause residual stress and affect the surface quality. The removal of white layer by AFM method was shown by Gov, et al. [4-5].

he International Conference of Materials and Engineering Technology

Different workpiece materials and different material properties affect surface quality of AFM process. Previous studies which are performed on Ti-6Al-4V and AISI D2 hardened tool steel show that AFM process has better results on harder materials [6-7]. Abrasive material and abrasive properties affect the surface quality. Viscosity and concentration are the main abrasive parameters which improves the quality [8].

A spinneret is a set of nozzles which is used for all types of fiber extrusion of yarn, staple, spunbond, meltblown and spunlace applications. Melt spinning is a typical spinning of synthetic fiber such as polyester, nylon, polypropylene. Melted polymer is discharged from the spinneret. The discharged fiber is wound after quenching process and drawing process. Melt spinning is used for various polymers and applications [9]. The most sensitive step in the filament production system is the extrusion of the polymer. The polymer that comes out of the process is a liquid mass that has not yet become a yarn and is flowing at a very high temperature. This liquid is sprayed into a solidified medium. The quality of the fiber is mostly affected by the geometry and the surface quality of the spinneret hole.

Spinnerets which are made of corrosion resistant tool steels have various types small holes. The number of holes on the spinneret is usually between 10 and 150 pieces (see figure 1) [10]. These holes are usually produced by electrical discharge method. The nozzles have bigger and circular entrance, smaller and shaped holes at the exit (see figure 2). The geometry of the hole at the exit is various (round, trilobal, octalobal etc.) as shown in Figure 3.



Figure 1. A photograph of a melt spin spinneret



Figure 2. Section view of a spinneret hole



Figure3. Various types of spinneret nozzle shapes



In this study, two types of spinnerets (round-hole and trilobal-hole) were finished by one-way AFM process subsequent to EDM manufacturing of the holes. The quality of the holes before and after AFM process was compared. It is expected that the finished hole quality may eliminates extrusion problems such as slow holes, drips, stoppages and dog legs.

#### **Materials and Methods**

#### Workpiece

In the experimental study, heat-treated corrosion resistant tool steel spinnerets have been used. Two types of spinnerets (round-hole and trilobal-hole) were finished to show the effect of AFM process (see figure 4-5). The holes were manufactured by using EDM method and the spinnerets were cleaned by ultrasonic cleaner and pressurized air before and after AFM process to remove any dirt and debris.



Figure 4. Round-hole spinneret



Figure 5. Trilobal-hole spinneret





#### **Abrasive Flow Machining**

A machine having specifications given in table 1 is used for this process. The machine is placed on material laboratory of Gaziantep University (figure 6). This machine has 4 main units which are hydraulic, control, cooling and extrusion units. Hydraulic unit provides the pressure on the machine for extrusion. Cooling unit is to balance abrasive media temperature in the piston. Control unit is using for defining the parameters of process. Extrusion section is the most important section. Process is being done on this part. The workpiece is placed and fixed between cylinders. A piston extrudes abrasive media into the nozzles. Abrasive media is prepared a concentration of 50% with 400 mesh silicon carbide.

#### Table 1. AFM machine specifications

#### **AFM Specifications**

Hydraulic pressure	10-400 bar
Volume	6 liters
Piston range	400 mm
Bore diameter	50-200 mm

#### **Images of holes**

The spinneret holes were investigated by using a light microscope (Olympus Tokyo 150x zoom) and a profile projector (Leitz PP 500). The geometry of the nozzle exits and the shadows of the debris can be compared by these images before and after AFM finishing.



Figure 6. AFM machine



## **Results and Discussion**

## **Profile projector images**

Figure 7 shows the images of the holes taken from the profile projector after EDM. Although the holes were cleaned by pressurized air and ultrasonic methods, the asperities can be seen in the figure both for round-hole and trilobal-hole.





Using the one-way AFM process, 5 liters of abrasive media was passed through each hole and then the finished the holes were cleaned by pressurized air and ultrasonic methods. The profile projector images of the same holes after AFM process are shown in figure 8. The results show that the residual asperities are smoothed or even completely removed while keeping the geometry of the exit.



Figure 8. After AFM images (a) round hole nozzle (b) trilobal shaped nozzle

Technology





#### **Microscope images**

From the images obtained by the microscopic examinations, shows similar results with the profile projector ones. The sharpness of the circumferences of the exit profiles and the removal of the debris can be seen from figure 9 and figure 10.



**Figure 9.** Round-hole microscope views (a) before AFM process (b) after AFM process



(c)

(d)

Figure 10. Trilobal-hole microscope views (a,b)before AFM process (c,d) after AFM process



#### Conclusions

In this study, the feasibility of one-way AFM process for the finishing of spinneret holes has been investigated. The results show that the process can be successfully applied to complex geometries of spinneret holes. Both microscopic and profile projector images are in well agreement that the residual asperities are smoothed or even completely removed while keeping the geometry of the exit. It can be expected that the quality of the extruded fibers is going to be increased by AFM finishing process.

The International Conference of Materials and Engineering Technology

#### Acknowledgments

The authors would like to acknowledge the contributions of the Scientific Project Bureau (BAPYB) of The Gaziantep University and Mennan Makina Co.

#### References

- 1. Eyercioglu, O, Yilmaz, N. F. and Dereli, T., "Aşındırıcı Akışkanla Yüzey İşleme TİMAK Tasarım İmalat Analiz Kongresi," TİMAK-Tasarım İmalat Analiz Kongresi, Balıkesir, 2006.
- 2. Eyercioglu, O., Gov, K., Aksoy, A., "Abrasive Flow Machining of Asymmetric Spur Gear Forging Die" 6th International GAP Engineering Conference – GAP2018
- 3. Gov K., Eyercioglu O. (2017). "Abrasive Flow Machining of Ti-6AL-4V" International Science and Technology Conference: 2146-7382
- 4. Gov, K., O. Eyercioglu and M. V. Cakir (2013). "Hardness Effects on Abrasive Flow Machining." Journal of Mechanical Engineering 59(10): 626-631
- O. Eyercioglu, Gov, K. and M. V. Cakir "Influence of machining parameters on the surface integrity in small-hole electrical discharge machining" originally published online 23 August 2013 Proceedings of the Institution of Mechanical Engineers, Part B: Journal of Engineering Manufacture 2014 228: 51
- Gov, K., O. Eyercioglu, "Effects of abrasive types on the surface integrity of abrasive flow machined surfaces," Proceedings of the Institution of Mechanical Engineers, Part B: Journal of Engineering Manufacture, vol. 232, no. 6, pp. 1044–1053, Aug. 2016.
- 7. Gov, K., O. Eyercioglu and M. V. Cakir "Aşındırıcı Akışkan ile İşleme (AFM) Parametrelerinin Tel Erozyonla Kesilmiş Kalıp Yüzeyine Etkisinin İncelenmesi" TMMOB Makina Mühendisleri Odası Konya Şubesi VI. Makina Tasarım ve İmalat Teknolojileri Kongresi 22-23 Ekim 2011
- 8. Gov K, Abrasive Flow Machining of EDMed Surfaces, Ph. D. Thesis, Gaziantep University, Institute of Science >Mechanical Engineering, 20142014.
- 9. https://www.kasen.co.jp/english/product/spinneret/melt.php
- 10. http://www.mennanmakina.com.tr/spinnerette-machine-detail-25.html

# NADIR TORAK METAL HEKZABORURLERIN KATALIZOR OLARAK KULLANILMASI

The International Conference of Materials and Engineering Technology

## CENGİZ BOZADA\*1, MİKAİL ASLAN2

<sup>1</sup> Gaziantep Üniversitesi, Mühendislik Fakültesi, Fizik Mühendisliği, Gaziantep, Türkiye <sup>2</sup> Gaziantep Üniversitesi, Mühendislik Fakültesi, Metalürji ve Malzeme Mühendisliği, Gaziantep, Türkiye

## ÖZET

Nadir Toprak Metal hekzaborürler (REB<sub>6</sub>) H<sub>2</sub>O, CO, H<sub>2</sub>, radyoaktif Cs ve O<sub>2</sub> gibi gazların adsorpsiyonu için katalizör olarak kullanılmaktadır. Bu amaçla çeşitli teknikler kullanılmıştır. Bunlardan bazıları yansıma emilim kızılötesi spektroskopisi (RAIRS),X-ışını fotoelektron spektroskopisi(XPS), yüksek çözünürlüklü elektron enerji kaybı spektroskopisi (HREELS), yarı kararlı çarpma elektron spektroskopisi (MIES) ve ultraviyole fotoelektron spektroskopisi (UPS) gibi tekniklerdir. Bu malzemelerin katalizör olarak kullanılmasının anlaşılması için hesaplamalı malzeme mühendisliği alanına giren temel prensip hesaplamaları (ab initio) yöntemlerinden olan yoğunluk fonksiyon teorisi (DFT) hesaplamaları ve Car-Parrinello moleküler dinamikleri (CPMD) analizleri kullanılmaktadır. Bu çalışmadaki amaçlardan biride REB<sub>6</sub>ların katalizör olarak kullanılmasıyla ile ilgili bilgilerin organize edilerek yeni ufukların açılmasıdır.

#### Anahtar Kelimeler: Nadir Toprak Hekzaborürler, RAIRS, XPS, MIES, DFT, CPMD ve UPS

#### 1.Giriş

MB<sub>6</sub> yüzeylerinde adsorpsiyonu araştıran deneysel ve teorik çalışmalar, temel olarak CO, O<sub>2</sub> ve H<sub>2</sub>O gibi ortak çevresel gazların neden olduğu yayıcı düzlemler üzerindeki zehirlenme etkilerini belirlemeye odaklanmıştır[1-2].Her ne kadar yüksek vakum koşullarında hidrojen bir arka plan gazı olarak kabul edilse de sadece az sayıda çalışmayla hidrojenin MB<sub>6</sub> yüzeyleri ile etkileşimini göz önünde tutulmaktadır. MB<sub>6</sub> temiz yüzeylerin yapısını ve özelliklerini ve ayrıca H<sub>2</sub>O, CO ve O<sub>2</sub> gibi gazlarla etkileşimlerini daha iyi anlamak için çeşitli ultra yüksek vakumlu yüzey yöntemleriyle çalışılmıştır. İlk çalışmalar temiz yüzeylerin yapılarına ve özelliklerine ve bunların oksijenle etkileşimlerine odaklanmış olsa da, son zamanlardaki birkaç raporda CO ve H<sub>2</sub>O gibi türlerin bazı koşullar altında moleküler olarak adsorbe edilebildiğini ve diğer koşullar altında ayrıştığını, hekzaborürlerin katalizörler olarak yararlı olabileceği gösterildi[3].Biz bu çalışmada, REB6 malzemelerin katalizör olarak kullanımı ile ilgili literatürde bulunan deneysel ve hesaplamalı çalışmaların organize edilmesi üzerine odaklandık. Genel olarak, REB<sub>6</sub>lar CO, O<sub>2</sub>, H<sub>2</sub> ve H<sub>2</sub>O emilimiyle ilgili kullanıldığı tespit edilmiştir.

## 2. Tartışma ve Sonuç

#### 2.1 CO Emilimi

CO, herhangi bir molekülün en yüksek ayrışma enerjisine sahip olan basit bir moleküldür ve ayrıca heterojen kataliz alanında teknolojik olarak önemli bir moleküldür[4]. CO, yüzey bilimlerinde en çok çalışılan prob molekülüdür[5]. Gaz-yüzey etkileşimleri, adsorpsiyon bölgeleri ve çeşitli metal, yarı iletken ve yalıtkan yüzeylerde reaktif dinamikleri hakkında temel bilgiler sağlar[6].

Ayrı bir çalışmada ise Yorisaki ve diğerleri;  $LaB_6$  (100) ve  $LaB_6$  (111) yüzeylerinde karbon monoksitin adsorpsiyonu, RAIRS ve XPS teknikleriyle deneysel olarak incelemişlerdir. CO'nun

iki yüzeyle etkileşimi de DFT (Spin-polarizasyonu içeren periyodik DFT hesaplamaları VASP kodu[7] kullanılarak yapılmıştır) ile incelenmiştir. Her iki yüzey de CO'yu moleküler olarak düşük sıcaklıklarda ve farklı biçimlerde absorbe eder. Yorisaki'nin hem doğrudan deneysel kanıtları hem de deneysel sonuçların DFT hesaplama sonuçları, CO'nun (111) yüzeyinin bor bölgelerinde adsorbe ettiğini göstermiştir[8].

he International Confer

aterials and Engineering Technology

Şekil-1, yüzey birimi hücresi başına bir molekülü kaplayan borla sonlandırılmış LaB<sub>6</sub> (111) yüzeyinde olası bir CO yapısını göstermektedir. Bu şekilde gösterilen optimize edilmiş geometri, enerjide yerel bir asgari seviyeye karşılık gelir. CO'nun karbon atomu boyunca bir B<sub>3</sub> üçgeninin tek bir bor atomuna bağlandığını gösterir [8].



Şekil 1. (a) Yan ve (b) üst, 1.0 ML'lik bir kaplamada, LaB<sub>6</sub> (111) yüzeyinde bir CO sitesi için bir döşeme modeli ve DFT hesaplamasından elde edilen optimize edilmiş CO yapısının görünüşleri Adopted from[8]Copyright © 2009 Elsevier B.V.

La 3d tepe noktası için sonuçlar, Şekil-4(a)'da gösterilmiştir. Temiz ve arka plan sonuçları, 440 K'ye ısıtmadan önce ve sonra CO maruziyetinden sonraki sonuçlarla aynıdır. Kirlenmiş yüzey, CO maruz kalan yüzeyle aynı konumda bir La 3d tepe noktası gösterir, ancak aynı zamanda 838 ve 839 eV arasında daha belirgin bir düşük bağlayıcı enerji gösterir. Şekil-4(b), 90 K'de LaB<sub>6</sub> (100) yüzeyine 7 L CO maruziyetini ve yüzeyin 440 K kadar ısıttıktan sonra CO maruziyetin C1 bölgesindeki XPS sonuçlarını göstermektedir.



Şekil-3. (a) La 3d ve (b) C 1s bölgelerinde XPS spektrumu Adopted from[8] Copyright© 2009 Elsevier B.V. All rights reserved

209



## 2.2 H<sub>2</sub> Emilimi

Hidrojen adsorpsiyonu temel olarak yüzeylerdeki fiziksel ve kimyasal temel işlemler için önemlidir [9]. Molekül-yüzey etkileşimleri, özellikle katalitik işlemlere olan ilgilerinden dolayı H<sub>2</sub> ile geniş çapta çalışılmıştır[10]. Kevin M. Schmidt ve diğerleri, kendi kendine tutarlı DFT hesaplamalarını ve CPMD[11]analizlerinin bir kombinasyonunu kullanarak, hidrojenin MB<sub>6</sub> yüzeylerle temel enerjilerini ve etkileşimli davranışlarını hesapladılar. Tercihli bağlama yerleri için arama alanını azaltmak amacıyla, DFT içindeki CPMD kullanarak moleküler dinamik simülasyonları gerçekleştirdiler. Hesaplanan enerjik sonuçlara göre, yüzey birimi hücresi (ML) başına tek bir hidrojen molekülünün lokal adsorpsiyonunun (emmenin) mümkün olduğunu ve sarkan bağ konumunda kimyasal olarak emilen hidrojen atomlarının çok uygun olduğu sonucuna vardılar. CPMD simülasyonlarının analizi ayrıca, ayırıcı adsorpsiyon yoluyla MB<sub>6</sub> yüzeylerinde hidrojen rekombinasyon tepkimelerine eşlik eden mekanizmalara ve yüzeydeki ayrı ünite hücrelerini birbirine bağlayan köprüleme bölgelerine girmesini sağlar. Şekil-5, bor uçlu hekzaborürlerin yüzeyi üzerindeki tekli ve moleküler hidrojen için olası bağlanma yerlerini göstermektedir. Sol panel, iki hidrojen atomlarının bağlanma yerlerini açıklanmaktadır[12].

The International Conference

aterials and Engineering Technology



Şekil 4.Moleküler hidrojen (sol panel) ve Tek bir hidrojen atomu (sağ panel) metal hekzaborür yüzeylerinde adsorpsiyon için potansiyel bağlanma yerleri Adopted from [12]Copyright © 2019 American Chemical Society



## 2.3 H<sub>2</sub>O Emilimi

Mikrokatılarda H<sub>2</sub>O adsorpsiyonu,ısı transformasyon sistemlerindeki büyük uygulamaların-dan dolayı büyük ilgi görmüştür[13].H<sub>2</sub>O adsorpsiyon / desorpsiyona dayalı adsorpsiyonlu ısı pompaları (AHP'ler) düşük sıcaklıkta ısı toplamak için daha verimli ve çevre dostu bir yaklaşım sağlar[14].Yorisaki ve diğerleri,LaB<sub>6</sub> (100) yüzeyindeki suyun (H<sub>2</sub>O ve D<sub>2</sub>O) kimyasal olarak emilimini; RAIRS ve HREELS ile incelediler.LaB<sub>6</sub> (100) yüzeyini oda sıcaklıkları ile belli sıcaklıklar arasında H<sub>2</sub>O ve D<sub>2</sub>O 'ye maruz bıraktılar ve spektrumları yüksek sıcaklıklara ısıttıktan sonra elde ettiler. Oda sıcaklığı adsorpsiyonu ayrıca HREEL spektrumlarında düşük frekans kayıp özelliklerini de ortaya koymaktadır, bu durum O<sub>2</sub>'nin ayrışma adsorpsiyonunu takiben elde ettikleri sonuçlara oldukça benzerdir. Yorisaki, frekans kayıp özelliklerini; iki La atomu arasında bağlanmış O atomlarının La-O uzantısına bağlamıştır. Buna karşın H<sub>2</sub>O'nun ayrışma adsorpsiyonundaki düşük frekans kayıp özelliklerini ise, absorbe edilmiş O veya absorbe edilmiş OH'nin La-O titreşimlerinden kaynaklandığını belirlemiştir [1].

The International Conference of Materials and Engineering Technology

## 2.4 O<sub>2</sub> Emilimi

Adsorbe maddeler arasında, oksijen (O<sub>2</sub>) en önemlilerinden biridir, çünkü yalnızca doping yoluyla özellikleri önemli ölçüde değiştirmez, aynı zamanda atmosferdeki en çok bulunan ikinci gazdır ve bu nedenle de atmosferin performansını etkileme olasılığı yüksektir[15].Kawanowa ve diğerleri, O2 ile adsorbe edilmiş LaB<sub>6</sub> (001) yüzeyini, yarı kararlı çarpma elektron spektroskopisi (MIES) ve ultraviyole fotoelektron spektroskopisi (UPS) ile incelediler. Bu sonuçlara göre; yüzeyin La ile sonlandırıldığını belirlediler. Oksijenle indüklenen iki yapı hem MIES hem de UPS spektrumlarında 5.8 ve 7.5 eV bağlanma enerjisinde ortaya çıktı. Oksijenin LaB<sub>6</sub> yüzeyinde ayrıldığı sonucuna vardılar [16]. Ayrı bir çalışmada ise Lavrenko ve diğerleri, LaB<sub>6</sub>'in1200 <sup>0</sup>C'lik bir sıcaklığa kadar oksijen içerisinde nispeten reaktif olmadığını tespit ettiler. 800 ila 1000 °C arasındaki sıcaklıklarda LaB<sub>6</sub> ölçeğinin temelde LaB<sub>4</sub> ve B<sub>2</sub>O<sub>3</sub>'ten oluştuğunu ve 1000 <sup>0</sup>Cüzerindeki sıcaklıklarda LaB<sub>4</sub>, B<sub>2</sub>O<sub>3</sub> ve La<sub>2</sub>O<sub>3</sub>'ten oluştuğunu belirlediler.Oksidasyon oranının, oksijen basıncına ve numunenin gözeneklerine bağlı olduğu sonucuna vardılar.Oksidasyon kinetiğinin, 700 ila 1200°C sıcaklık aralığında parabolik hızı takip ettiğini gözlediler[17]. Yamamoto ve diğerleri LaB<sub>6</sub> (100) ve (111) yüzeylerinde oksijenin adsorpsiyonunu, HREELS ile çalıştılar.LaB<sub>6</sub>(100) ve (111) yüzeyleri arasındaki oksijen adsorpsiyonunda bir fark gözlediler.(100) yüzeyindeki dipol alanının serbest elektronlar tarafından koruma etkisinin (111) yüzeyinden çok daha güçlü olduklarını belirlediler.(100) yüzeyinde, oksijenin oda sıcaklığındaki La atomlarının köprü bölgesinde adsorbe ettiğini ve yapıyı 700 °C'nin üzerinde yeniden düzenlediğini gözlediler[18].

Yamamoto ve diğerlerinin yaptığı farklı bir çalışmada ise LaB<sub>6</sub>(100) ve PrB<sub>6</sub> (100) yüzeylerinde oksijenin adsorpsiyonunu, düşük enerjili elektron kırınımı (LEED) ve HREELS ile incelediler. Oda sıcaklığındaki oksijen adsorpsiyonun, çeşitli titreşim kaybı özellikleri verdiklerini gözlediler.600 °C'ye kadar tavladıktan sonra hem LEED modeli hem de HREELS kaybı özellikleri daha keskin hale gelir[19]. RAIRS ve HREELS, LaB<sub>6</sub>(100) ve (111) yüzeylerinde oksijenin adsorpsiyonunu incelemek için Yorisaki ve diğerleri tarafından kullanıldı.95 K ve üzerindeki sıcaklıklarda yüzeyleri O<sub>2</sub>'e maruz bıraktılar, yüzeylerde atomik oksijen ürettiğini ve HREELS çalışmalarında titreşimsel tepeler verdiğini gözlediler.LaB<sub>6</sub>(100) yüzeyindeki RAIRS zirvelerinin, bor kafesin titreşimlerine karşılık geldiğini tespit ettiler. Bor uçlu LaB<sub>6</sub> (111) yüzeyinde, hem bor kafesin hem de bor oksidin titreşimlerine geniş ve zayıf özellikler kazandıklarını gözlediler.(100) yüzeyindeki oksijenin CO için adsorpsiyon bölgelerini bloke ettiği ve adsorbe edilmiş CO'in, O<sub>2</sub>'nin ayrışma adsorpsiyonunu önlediği sonucuna vardılar[20]. Farklı bir çalışmada ise tek bir LaB<sub>6</sub> kristali (100) yüzeyindeki oksijen adsorpsiyonunu ilk aşamalarını araştırmak için XPS tekniği Perkins ve diğerleri tarafından kullanılmıştır. Oksijen adsorpsiyonunu La 3d çizgi



şekillerini belirgin şekilde değiştirdiğini, oysaki B1s'in tepe noktasının etkilenmediğini tespit ettiler. XPS verilerinin La bölgelerinde oksijen adsorpsiyonunu açıkça ortaya koyduğunu ve düşük  $O_2$ 'e maruz kaldıklarında ise bor bölgelerinde adsorpsiyon belirtisi göstermediği sonucuna vardılar. Yüksek sıcaklıklarda ise daha kapsamlı oksidasyonun, hem bor hem de lantan oksitlerin oluşum gösterdiğini gözlediler[21]. Şekil 5, B1 bölgesini kapsayan beş XP spektrumundan oluşmaktadır. Yukarıdan aşağıya spektrumlar temiz kristaldir. 10 Langmuir (L) oksijene maruz kalan kristal, ac (2 x 2) LEED modeli sergileyen kristal, bir ap (2 x 1) LEED modeli sergileyen kristal yüksek sıcaklıkta 1000 L  $O_2$  dozu ile ağır biçimde oksitlenir



Şekil 5.Temiz ve oksijen dozlu LaB<sub>6</sub> (100) için B1s bölgesinin XP spektrumları Adopted from [21] Copyright © 1999 Elsevier Science B.V. All rights reserved.

Berrada ve diğerleri sinterlenmiş ve erimiş polikristalin LaB<sub>6</sub> ve havaya maruz kalmış CeB<sub>6</sub> örneklerini incelemek için AES ve XPS kullandılar. Bu çalışmanın sonucunda hem karbon hem de oksijenle kirlenme gözlendi[22].Farklı bir çalışmada ise Klauser ve diğerleri, 1500-1600 K sabit sıcaklıklardaO<sub>2</sub> basıncına karşı tutulan LaB<sub>6</sub> kristallerin (100), (111) ve (110) yüzeylerindeki La, B ve O konsantrasyonlarını izlemek için AES kullandılar [23].

#### **Referanslar:**

- 1. Yorisaki, T.;Tillekaratne, A.; Ren, Y.; Moriya, Y.; Oshima, C.; Otani, S.; Trenary, M. Adsorption and dissociation of water on LaB<sub>6</sub>(100) investigated by surface vibrational spectroscopy. Surf. Sci. ,2012, 606:247–252.
- **2.** Davis, P. R.,&Chambers, S. A. A study of oxygen interaction with a LaB<sub>6</sub>(100) single crystal surface. Applications of Surface Science,**1981**, 8(1-2)197-205.
- **3.** Trenary, M. Surface Science Studies of Metal Hexaborides. Sci.Technol. Adv. Mater.,**2012**, 13:023002.
- **4.** R.R. Ford, Carbon Monoxide Adsorption on Transition Metals, in: Adv. Catal. Relat. Subj.Academic Press, New York, **1970**, Vol,(21 51).



The International Conference of Materials and Engineering Technology

- 6. [Somorjai, G.A.,&Li,Y. Introduction to surface chemistry and catalysis. John Wiley&Sons.,2010.
- 7. Kresse, G., & Furthmüller, J. Efficient iterative schemes for ab initio total-energy calculations using a plane-wave basis set. Physical review B, **1996**,54(16):11169.
- 8. Yorisaki, T.,Tillekaratne, A., Ge, Q., Oshima, C., Otani, S., &Trenary, M..Probing the properties of the (111) and (100) surfaces of LaB<sub>6</sub> through infrared spectroscopy of adsorbed CO. Surface Science, **2009**,603(19):3011-3020.
- **9.** Barth, J. V. Transport of adsorbates at metal surfaces: from thermal migration to hot precursors. Surface Science Reports, **2000**,40(3-5):75-149.
- **10.** A. Groβ, Surf. Sci., **1998**, 32:291

UWE ditte

- **11.** Car, R.;Parrinello, M. Unified approach for molecular Dynamics and density functional theory. Phys. Rev. Lett.,**1985**, 55:2471–2474.
- Schmidt, K. M., Misture, S. T., Graeve, O. A., &Vasquez, V. R..Interaction of Hydrogen with MB<sub>6</sub> (M= Ba, Ca, La, andSr) Surfaces from First Principles. ACS Omega, 2019,4(1):65-72.
- **13.** Tan, K.;Zuluaga, S.; Gong, Q.; Canepa, P.; Wang, H.; Li, J.;Chabal, Y. J.; Thonhauser, T. Water Reaction Mechanism in Metal Organic Frame Works with Coordinatively Unsaturated Metal Ions: MOF-74. Chem. Mater.,**2014**, 26:6886–6895.
- 14. Henninger, S. K.; Jeremias, F.; Kummer, H.; Janiak, C. MOFs for Use in Adsorption Heat Pump Processes. Eur. J. Inorg. Chem., 2012, 2012: 2625–2634.
- **15.** Lamoen, D.,&Persson, B. N. J. Adsorption of potassium and oxygen on graphite: A theoretical study. The Journal of chemical physics,**1998**,108(8):3332-3341.
- **16.** Kawanowa, H.,Souda, R., Otani, S., Ikeuchi, T., Gotoh, Y.,Stracke, P., ...&Kempter, V..Interaction of O<sub>2</sub>with LaB<sub>6</sub>(001) surfaces as studied with MIES and UPS. Surface science,**2001**, 482: 250-253.
- Lavrenko, V. A., Glebov, L. A., Lugovskaya, Y. S., &Frantsevich, I. N..Investigation of high-temperature oxidation of lanthanum hexaboride in oxygen and the effect of internal oxidation on the protective properties of the scale. Oxidation of Metals, 1973,7(2): 131-139.
- **18.** Yamamoto, N.,Rokuta, E., Hasegawa, Y., Nagao, T., Trenary, M., Oshima, C., &Otani, S..Oxygen adsorption on LaB<sub>6</sub> (100) and (111) surfaces. Surface science,**1996**,357:708-711.
- **19.** Yamamoto, N.,Rokuta, E., Hasegawa, Y., Nagao, T., Trenary, M., Oshima, C., &Otani, S..Oxygen adsorption sites on the PrB<sub>6</sub> (100) and LaB<sub>6</sub> (100) surfaces. Surface science, **1996**,348(1-2):133-142.
- **20.** Yorisaki, T., Tillekaratne, A., Moriya, Y., Oshima, C., Otani, S., &Trenary, M..Vibrational spectroscopy of oxygen on the (100) and (111) surfaces of lanthanum hexaboride. SurfaceScience, **2010**,604(13-14): 1202-1207.
- **21.** Perkins, C.L., Trenary, M., Tanaka, T., & Otani, S..X-ray photoelectron spectroscopy investigation of the initial oxygen adsorption sites on the LaB<sub>6</sub>(100) surface. Surface science, **1999**, 423(1):L222-L228.
- **22.** Berrada A, Mercurio J P, Etourneau J, Alexandre F, Theeten J B and Tran Minh D.Surf. Sci.,**1978**, 72 :177
- 23. Klauser S J and Bas E B .Appl. Surf. Sci., 1979, 3: 356

# THE EFFECTS OF PUNCH SPEED ON THE FORGING LOAD OF HOT PRECISION BEVEL GEAR FORGING

The International Conference of Materials and Engineering Technology

Mehmet ALADAG<sup>1</sup>, Omer EYERCIOGLU<sup>1</sup>, Gulaga TAS<sup>1</sup>

<sup>1</sup>Mechanical Engineering Department, Gaziantep University, Gaziantep, TURKEY

#### Abstract

The mass production bevel gear is mainly realized by precision forging process. The process can be carried out cold, warm or hot conditions by using various forging presses (hydraulic or mechanical presses and HERF machines). The process cycle time is changing with the type of forging press (i.e. the speed of punch). During hot forging, hot billet is in contact with the relatively colder die and heat is transferred to the environment. Therefore, the billet temperature is changing during deformation and the process time is an effective parameter of the temperature distribution of the billet. The flow stress of the material is dependent on both the temperature and strain rate (punch speed). In this study, the effects of punch speed on the forging load were investigated by finite element method. To simulate different forging presses, various punch speeds were chosen as 0.001 m/s, 0.01 m/s, 0.1 m/s, 1 m/s. and 10 m/s. The temperature distributions of the billets and the forging loads were also determined for these punch speeds. The results show that the temperature drop of the billet and the forging load are decreasing with the increasing punch speed. So that forging with HERF machines is advantageous than the hydraulic and mechanical presses in the precision hot forging process of bevel gears.

Keywords: Bevel gear, precision forging, finite element method, forging temperature, punch speed.

#### 1. Introduction

Spur bevel gears are the most extensively used in the power transmission mechanism. To reduce cost and manufacturing time of spur bevel gears, the plastic deformation (forging method) which is one of the manufacturing process, is desirable to the other fabrication process (machining, hobbing, etc.) [1]. Hence, precision hot forging technology to spur bevel gear forging process has technological advantage with accompanying features. Also, precision forging, which gives the nearest shape to the final geometry after operation completed, therefore, is little or no further finishing processes are required [2,3]. Due to the directional alignment of grains during the forging, the mechanical properties are significantly improved by this process [4]. In the previous studies, many features in hot forging process have been examined. Due to longer time and higher cost requirements of experimental studies on precision gear forging, computer aided numerical methods have been gained significant roles. In most of the numerical analyses, finite element or finite volume (FE - FV) methods have been widely used due to the success in prediction of the loads and stresses of forging process. In particular, FEM has been effectively used for large deformation problems.

Many researches successfully applied FE simulations in gear forging operations, such as Doege and Nale [5], Szentmihali et al. [6], and Mamalis [7]. Song and Im [8–10] have determined process design parameters by FE analyses. Zhuang et al [11] worked on tooth variation in hot forging of

spur bevel gears. The effect of forming speed in precision forging with servo-press machine was evaluated by Kim et al.[12].

e Internationa

In present study, hot forging process of bevel gear in different punch speeds were examined by using Simufact Forming FE package. Defined press types and their velocities are hydraulic press, crank press and HERF (High Energy Rate Forming) machine, and 0.001 m/sec, 0.01 m/sec, 0.1 m/sec, 1 m/sec and 10 m/sec, respectively. Behavior of the material under subjected load and different velocities were presented. Moreover, the changing in the contact pressure between the workpiece and the die, and distribution of workpiece temperature were investigated.

## 2. Preparation of model

## 2.1. Bevel Gear Model and Workpiece Preparation

The selected bevel gear model is used in the differential gear box of a passenger car. The solid model of the bevel gear was created by using Solidworks 3D Package. The tooth number, module and cone angle of the bevel gear are 11, 8mm and 113,4 degrees, respectively. The dimensions of the gear are given in Figure 1. The initial billet size is calculated by using the final gear dimensions keeping in mind that the volume is constant during forging.



Figure 1. Representation of the bevel gear.

Because of the limited formability of the material, the forging operation is carried out in two steps. The first step is preforming the cylindrical billet and the second is precision forging of the bevel gear. The final shape of the preform is shown in Figure 2. However, preforming stage is not included to this study and precision forging stage which is critical is investigated.

Technology



Figure 2. Representation of the workpiece after preforming process.

## 2.2. Precision Bevel Gear Forging Die Set

The forging die is also created in Simufact Forming FE software environment. The geometry and dimensions of the die set are provided by the forging company. Thus, it was aimed to compare the results with the experimental study. The die set used in the simulations consists of 2 pieces for simplicity; punch and die as shown in Figure 3. Punch is the moving part which is attached to the ram of the press while the die is fixed on the press bed.



Figure 3. Bevel Gear forging die used in FE simulations.



## 2.3. Finite Element Modeling of Precision Forging

In order to provide more accurate and consistent information from the result of the forging process, the selected material and the consistency of the defined parameters are very important. As the material flow changes according to the mechanical properties of the material under load and the direction of the fibers, the analysis results also change. Thus, the material and the process parameters have to be selected carefully in the simulation and must be same or closer to the real forging operation. The punch speeds used in the simulations are given in Table 1. Also, in all models, the analyses were carried out for 99.5% filling of the die so that the results can be compared with each other accurately. The finite element parameters used in all analyses are given in Table 2.

of Materials and Engineering Technology

**Table 1.** Forging simulation models and corresponding punch speeds.

Study	Punch Speed (m/sec)
Model 1 (Hydraulic)	0.001
Model 2 (Hydraulic)	0.01
Model 3 (Crank Press)	0.1
Model 4 (Crank Press)	1
Model 5 (HREF)	10

<u>1</u>	0	
Workpiece Material	AISI 4340	
Die Material	H13	
Workpiece Temperature T <sub>w</sub> (°C)	1200	
Die Temperature T <sub>d</sub> (°C)	100	
Workpiece Material Model	Elasto-Plastic	
Die Material Model	Elastic	
Punch Material Model	Rigid	
Friction Coefficient	0.3	
Workpiece Mesh Type	Hexahedral	
Workpiece Mesh Size	219318 elements	
Bevel Gear Die Mesh Type	Tetrahedral 134	
Bevel Gear Die Mesh Size	578816 elements	
Solver Machine	2 x Xeon 4114 CPU with 30	
	Solver Core – 32GB Ram	

Table 2.	FE	parameter	settings.
----------	----	-----------	-----------

#### 3. Results and discussion

The temperature distributions on the forged gear at the final die filling stage (99.5%) are shown in Figure 4 for all punch speeds. The preform and the die were at 1200 °C and 100 °C respectively at the beginning of the forging operation. The heat is transferred from preform to the die and environment. Although some amount of heat is generated during deformation, the temperature drops by the time pass. As it is expected that the maximum temperature drop occurs for the lowest punch speed (longer forging time). The maximum temperature of the forging is found as 335 °C for 0.001 m/sec punch speed. Similarly, the maximum temperatures are 722 °C, 928 °C, 1056 °C and 1165 °C for 0.01 m/sec, 0.1 m/sec, 1.0 m/sec and 10 m/sec, respectively.

The effective (von Mises) stress distributions with respect to punch speeds are given in Figure 5. For high speed forming, there is a contradiction between temperature and strain rate effects in terms of flow stress. Lower forming time (higher punch speed) means less temperature drop, therefore lower flow stress. On the other hand, the flow stress increases with increasing strain rate and this is more affective at high temperatures (see flow stress-strain diagram of AISI 4340 in Figure 6). The results of FE analyses show that the maximum effective stresses are 334 MPa, 188 MPa, 96 MPa, 69 MPa and 62 MPa for 0.001 m/sec, 0.01 m/sec, 0.1 m/sec, 1.0 m/sec and 10 m/sec, respectively. The differences of effective stresses among higher punch speeds are smaller and this is due to strain rate effect.

The International

aterials and Engineering Technology

UWE Bristol





(b)

Figure 4. (a) Temperature distributions and (b) maximum temperatures at the maximum stroke of the punch for all models.



Figure 5. (a) Effective stress distribution and (b) maximum effective stress at the maximum stoke of punch for all model.



Figure 6. Flow stress-strain diagram of workpiece material (AISI 4340)

The interference pressure between the workpiece and the die surface (i.e. contact pressure) is also investigated by FE analysis. As shown in Figure 7, the maximum contact pressure is obtained for the lowest punch speed (0.001 m/sec). The value of contact pressure is very near to the yield strength of the die material (AISI H13), therefore there is a danger of plastic deformation on the die surface for 0.001 m/sec punch speed. For other punch speeds the maximum contact pressures are not more than 650 MPa, so that the die is safe.

The load-stroke diagrams of all cases are plotted in Figure 8. The maximum forging loads are 445 tonf, 250 tonf, 235 tonf, 215 tonf and 216 tonf for 0.001 m/sec, 0.01 m/sec, 0.1 m/sec, 1.0 m/sec and 10 m/sec punch speeds, respectively. The change in maximum load with respect to punch speed is shown in Figure 9. Due to lower flow stresses at high temperatures, the corresponding forging loads are also lower. Although punch speed is increase from 1.0 m/sec to 10 m/sec (so as lower temperature drop), the forging load is not reduced. This is due to increasing flow stress due to higher strain rate.

An experimental study was carried out in the laboratory by using a crank press and the photograph of the forged bevel gear is given in Figure 10. The forging parameters taken as similar to the FE model 3; the preform and die temperatures were 1200 °C and 100 °C respectively, punch speed was 0.1 m/sec, the die set was lubricated by Molykote Longterm2. The maximum forging load was measured as 225 tonf which is slightly higher than the corresponding FE result (215 tonf). The slight difference may be encountered due to transfer time of the perform form the furnace to forging press. The result of experimental work validates the FE simulation models and the analyses.



Figure 7. (a) Contact Pressure distribution and (b) maximum contact pressure at the maximum stroke of the punch for all model.

221



UWE Bristol

Figure 8. Load – Stroke Diagram for all models.



Figure 9. The change in maximum load with respect to punch speed.



Figure 10. The forged bevel gear by using crank press (0.1 m/sec press speed).

Technology



The results can be concluded as follows.

1. The result of experimental study validates the FE model used in this study for gear forging simulations.

The International Conference of Materials and Engineering Technology

- 2. The heat is transferred from workpiece to the die and environment during forging operation. Although some amount of heat is generated during deformation, the temperature drops by the time pass. As it is expected that the maximum temperature drop occurs for the lowest punch speed (longer forging time). For higher speeds (above 1 m/sec), the temperature drops are very small because of very short forging time.
- 3. For high speed forming, there is a contradiction between temperature and strain rate effects in terms of flow stress. Lower forming time (higher punch speed) means less temperature drop, therefore lower flow stress. On the other hand, the flow stress increases with increasing strain rate and this is more affective at high temperatures. The differences of effective stresses among higher punch speeds are smaller and this is due to strain rate effect.
- 4. When the forging process is carried out at low speed, the required press capacity is higher due to more cooling of the workpiece. At the lowest punch speed (0.001 m/sec), the maximum forging load, effective stresses and contact pressure are all higher than the other cases. More cooling (so as higher forging load) may cause deformation of the die surface.

#### Acknowledgement

The authors would like to acknowledge the contributions of the Scientific Project Bureau (BAPYB) of The Gaziantep University and Kanca Forging Co. (Kanca Dövme Çelik ve Makina A.Ş.).

#### References

- 1. Behrens, B.A., Doege, E., Reinsch, S., Telkamp, K., Daehndel, H., and Specker, A. (2007) Precision forging processes for high-duty automotive components. *J. Mater. Process. Technol.*
- 2. Eyercioglu, O., Dean, T., and Wlaton, D. (1996) Precision Forging of Gears. *Int. Mach. Des. Prod. Conf.*
- 3. Eyercioglu, O., Dean, T., and Walton, D. (1994) Dimensional accuracy of hot precision forged spur gears. *Int. Gearing Conf.*
- 4. Yilmaz, N.F., and Eyercioglu, O. (2018) Near Net Shape Spur Gear Forging Using Concave Preform. *Mechanics*, **24** (2), 268–277.
- 5. Doege, E., and Nägele, H. (1994) FE Simulation of the Precision Forging Process of Bevel Gears. *CIRP Ann.*, **43** (1), 241–244.
- 6. Szentmihali, V., Lange, K., Tronel, Y., Chenot, J.-L., and Ducloux, R. (1994) 3-D finite-element simulation of the cold forging of helical gears. *J. Mater. Process. Technol.*, **43** (2–4), 279–291.
- 7. Mamalis, A.G., Manolakos, D.E., and Baldoukas, A.K. (1996) Simulation of the precision forging of bevel gears using implicit and explicit FE techniques. *J. Mater. Process. Technol.*, **57** (1–2), 164–171.
- 8. Song, J.H., and Im, Y.T. (2007) Process design for closed-die forging of bevel gear by finite element analyses. *J. Mater. Process. Technol.*



- 9. Kim, S.-Y., and Im, Y.-T. (2002) Three-dimensional finite element analysis of nonisothermal shape rolling. *J. Mater. Process. Technol.*, **127** (1), 57–63.
- Kim, S.-Y., Tsuruoka, K., and Yamamoto, T. (2014) Effect of Forming Speed in Precision Forging Process Evaluated Using CAE Technology and High Performance Servo-press Machine. *Procedia Eng.*, 81, 2415–2420.
- 11. Zhuang, W., Han, X., Hua, L., Xu, M., and Chen, M. (2019) FE prediction method for tooth variation in hot forging of spur bevel gears. *J. Manuf. Process.*, **38**, 244–255.
- 12. Kim, S.Y., Tsuruoka, K., and Yamamoto, T. (2014) Effect of forming speed in precision forging process evaluated using CAE technology and high performance servo-press machine. *Procedia Eng.*, **81** (October), 2415–2420.

# SPİRAL KAYNAKLI ÇELİK BORULARIN KOROZYONA KARŞI FBE KAPLAMALARIN TEST VE KARAKTERİZASYONU

The International Conference of Materials and Engineering Technology

# MAHMUT GEL<sup>1</sup>, GÖKHAN ÇİL<sup>1</sup>, EKREM ALTUNCU<sup>\*2</sup>

<sup>1</sup>Erciyas Çelik Boru, Tasarım Merkezi, Düzce, TÜRKİYE. <sup>2</sup>Sakarya Uygulamalı Bilimler Üniversitesi, Metalurji ve Malzeme Müh. Sakarya, TÜRKİYE.

## Özet

Doğalgaz boru hatlarında koruyucu kaplamalara olan gereksinim ve daha uzun ömür talebi sürekli artmaktadır. Kaplama- altlık arayüzey arası kirlikler, uygun olmayan yüzey hazırlama koşulları ve bunun yanında optimize edilmemiş kaplama proses parametreleri zorlu çalışma şartlarında kaplamanın kullanım ömrünü sınırlamaktadır. Bu makale çalışmasında farklı ortam koşullarında, toprak üstünde çalışan sprial kaynaklı çelik (X70M PSL2) doğalgaz borularının katodik korozyona karşı korunması amacıyla elektrostatik sprey tabancası ile uygulanan ergiterek bağlanan epoksi esaslı (FBE, fusion bonded epoxy) kaplamaların test sonuçları ve kaplamaların karakterizasyonu sunulmaktadır. Tek katmanlı FBE kaplamaların performansına etki eden birçok faktör arasında kaplama öncesi çelik ön yüzey hazırlıkları; kaplamanın performansını önemli ölçüde etkilemektedir. Yapılan deneysel çalışmalar, ilgili uluslararası test standartları ve şartnameler (CSA-Z245.20 seri 14) kapsamında kontrol edilmiş, kimyasal karakterizasyon yöntemleri (FTIR, SEM) ile analiz edilmiştir. Değerlendirme sonucunda FBE kaplamaların başarılı bir şekilde üretilebildiği ve farklı sıcaklık koşullarında katodik soyulma, yapışma testlerinden başarı ile geçtiği gözlemlenmiştir.

#### Anahtar Kelimeler: Çelik boru, Korozyon, FBE kaplama, Katodik soyulma

#### 1.Giriş

FBE kaplamalar; boru hatlarında, çelik borularda ve birçok bağlantı parçası üzerinde korozyona karşı koruma amacıyla tek (300-450µm) veya çok katmanlı (1000µm) şekilde uygulanan termoset (epoksi) esaslı polimerik kaplamalardır. Geleneksel boyalardan farklı olarak çapraz bağlanma sonucunda nihai özelliklerini kazanmaktadır. Hem iç yüzeylerde hem de parçanın dış yüzeylerinde rahatlıkla kullanılabilmektedir. Kaplama kompoziyonu; reçine, sertleştirici (kürleme kolaylaştırıcı), pigment ve dolgu malzemelerinden oluşmaktadır. 180-250 °C arasında toz karışım hazırlanarak sıvı halde kaplanacak yüzeye uygulanmaktadır. Kaplama öncesinde yüzey iyice temizlenmekte ve ön ısıtma uygulandıktan sonra elektrostatik sprey tabancası ile uygulanmaktadır. Kontrollü bir atmosferde hızla kürleşme sonrası katı forma dönüşen kaplama nihai dayanımını ve kalınlığı kazanmaktadır. Birçok polimerik esaslı kaplama türü arasında üstün korozyon direncine sahiptir. Boru hatlarında kullanılan bu tür kaplamalardan: çalışma ortamlarında fiziksel ve kimyasal kararlılık, dış mekanik etkilere karşı direnç (gerilme, yapışma ve darbe), katodik korozyon şartlarına direnç göstermesi beklenmektedir [1-7]. Bu çalışma kapsamında çelik boru üzerine uygulanan FBE kaplamaların test ve karakterizasyonu detaylı bir şekilde sunulmaktadır.

#### 2. Malzemeler ve Test/Analiz Yöntemleri

Bu çalışmada, Tablo 1'de kimyasal bileşimleri verilen API 5L standart gerekliliklerine uygun, petrol ve gaz borusu imalatında kullanılan, düşük alaşımlı, ince taneli, yüksek mukavemetli 14 mm et kalınlığında X70M PSL2 malzemeden imal edilmiş 12m uzunluğunda, Ø813 mm çapında

spiral kaynaklı çelik boru elektrostatik sprey tabancası ile uygulanan ergiterek bağlanan epoksi esaslı kaplama (FBE, fusion bonded epoxy) ile tek katman olarak sistem CSA-Z245.20 seri 14 standardı 1A gerekliliklerine göre kaplanmıştır. Kaplama malzemesi olarak toz epoksi (Nap Gard 7-2514 EN) kullanılmıştır. Toz epoksiye ait teknik özellikler Tablo 2'de verilmiştir. Çelik yüzeyin hazırlanması ve kaplama uygulaması ve performans testleri Erciyas Çelik Boru A.Ş. üretim tesisinde gerçekleştirilmiş olup, yüzey karakterizasyonu Sakarya Uygulamalı Bilimler Üniversitesi laboratuvarlarında gerçekleştirilmiştir.

The International Conference of Materials and Engineering Technology

Malzeme			Kimyasal Ko	mpozisyon, %		
muzeme	С	Si	Mn	Р	S	Cr
	0,0396	0,0429	1,551	0,0038	0,00091	0,253
X70M PSL2	Мо	Ni	Cu	В	Nb+V+Ti	CEpcm
	0,0807	0,0016	0,0143	0,00021	0,0966	0,140

#### Tablo 1. Çelik borunun kimyasal özellikleri

#### Tablo 2. Toz epoksi teknik özellikleri

Renk	Kırmızı		
Yoğunluk, g/L	1440±50		
Partikül Boyutu	Max. %3>150µm, Max. %0,2>250µm		
Jel süresi, saniye	204°C'de	232°C'de	
	16±3	10±3	
Nem İçeriği, %	0,34		

Kaplanacak boru yüzeyinin FBE kaplamaya uygun hale getirebilmek için ortam şartları, kumlamada kullanılacak kum kalitesi kontrol edilmiş ardından kumlama işlemine tabi tutulmuştur. Kumlama işleminden hemen sonra asit karışımı uygulanmış ardından saf su ile durulanıp basınçlı hava ile yüzey kurutulmuştur. Yüzey kalitesinin uygunluğunu belirlemek için yüzey profili (Rz), yüzey temizliği, toz kirlilik seviyesi, tuz kirlilik seviyesi ölçülmüştür. Kaplama öncesinde boru yüzeyi 235°C ye kadar indüksiyon bobini ile ısıtılmıştır. Isıtılan boru yüzeyine toz epoksi elektrostatik sprey tabancalar ile tek katman şekilde uygulanmıştır. Toz epoxy kaplama işleminden hemen sonra jel süresi dikkate alınarak boru yüzeyi su ile soğutulmuştur. Uygulama sonrasında kaplama performansının belirlenmesi için ara yüzey ve arka yüzey gözeneklilik tayini, ara yüzey kirlilik tayini, katodik soyulma, yapışma ve kaplama gözeneklilik testleri gerçekleştirilmiştir. Kaplamanın kimyasal karakterizasyonunu tespit edebilmek için kaplama yüzeyi DSC, FTIR ve SEM yöntemleri ile analiz edilmiştir.

#### 3. Sonuçlar ve Tartışma

## 3.1. Yüzey Hazırlama Test ve Kontrol Sonuçları

Kumlama öncesi ortam şartları, kum, asit ve saf suyun kontrolleri yapılarak ölçümler kayıt altına alınmıştır. Kayıt altına alınan ölçümler Tablo 3' de verilmiştir.



Bağıl Nem, %	72,8	ΔT Sıcaklığı (T1- T2)°C	38,9
Ortam Sıcaklığı °C	10,2	Saf Su İletkenliği µS/cm	0,79
Yüzey Sıcaklığı (T1)°C	44,5	Kum İletkenliği µS/cm	8,8
Çiğlenme Noktası (T2)	5,6	Asit Karışım Oranı %	5,8

## Tablo 3. Kumlama öncesi kontroller

Yüzey hazırlığı öncesi ve sonrası yapılan kontrollerin CSA-Z245.20 seri 14 [1] standart gerekliliklerini sağladığı görülmüştür. Test sonuçları incelendiğinde; yüzey pürüzlülük seviyesinin  $80\mu$ m - $90\mu$ m aralığında olduğu görülmüştür. Çelik üzerine toz epoxy uygulaması yapılan bir çalışmada [2],  $60\mu$ m- $90\mu$ m arasındaki yüzey pürüzlülük değerinin katodik soyulma testi için en uygun aralık olduğu belirtilmiştir. Bunun yanında, ISO 8501-1[3] standardına göre uygun karşılaştırma kataloğu ile görsel olarak incelenmiş ve kumlanmış yüzey kalitesinin Sa 3 seviyesinde olduğu görülmüştür. Bu temizlik derecesi ilgili standartta ifade edilen beyaz metal yüzeyinin elde edildiği en üstün temizlik derecesidir. Kumlanmış yüzeyin toz seviyesinin ölçümü ISO 8502-3 [4] standart gerekliliklerine göre gerçekleştirilmiş, alınan ölçümlerde en düşük ve istenilen seviye-1 derecesinde olduğu görülmüştür. Kumlanmış yüzey üzerinden alınan tuz ölçümlerinde ise, tuz seviyesinin  $0,2 \mu g/cm^2$  olduğu görülmüş ve çok düşük değere sahip olduğu için kaplama performansına etki etmeyeceği düşünülmüştür.



Şekil 1. Yüzey hazırlama sonrası ölçümler (a)Yüzey pürüzlüğü, b)Yüzey kalitesi, c)Toz testi, d)Tuz testi)

# 3.2. Kaplama Fiziksel ve Mekanik Test Sonuçları

Ara yüzey kirliliği ve gözeneklilik tayini gerçekleştirmek için toz epoksi kaplanmış borudan çıkartılan deney numunesi ortasından kesilip, -30°C de alkol içerisinde 1 saat bekletilmiştir. Soğutucudan çıkartılan numune bükme cihazına alınarak Şekil 2'de görüldüğü gibi deney numuneleri çıkartılmıştır. Çıkartılan numune 40X büyütme altında mikroskopta incelenerek CSA-Z245.20 seri 14 standardında verilen tablo ile karşılaştırılmıştır. Karşılaştırma sonuçlarına göre

ara yüzey gözeneklilik seviye-1 olduğu görülmüştür. Şekil 3 de kesit gözeneklilik oranı ise seviye-2 olduğu tespit edilmiştir.

he International Conference

aterials and Engineering Technology



Şekil 2. Gözeneklilik tayini için arayüzey numune görüntüsü



Şekil 3. Kesit gözeneklilik

Ara yüzey kirliliği tayini sonucunda CSA-Z245.20 seri 14 standardında verilen tablo ile karşılaştırıldığında Şekil 4'de görüldüğü gibi kirlilik %15 seviyesinde olduğu görülmüştür.



Şekil 4. Ara yüzey kirliliği

## 3.3. Kaplamaların Katodik Soyulma ve Yapışma Test Sonuçları

Toz epoksi kaplamalarının katodik soyulma test sonuçlarının belirlenmesi için, boru üzerine toz epoksi kaplama sonrasında 100x100 mm ebatlarında test plakaları çıkartılmıştır. Katodik soyulma testlerine ait parametreler ve test sonuçları Tablo 4'de verilmiştir. Katodik test sonuçları incelendiğinde (Şekil 5) süre ve soyulma sonuçlarının kendi aralarında, sıcaklık ve soyulma sonuçlarının da kendi aralarında doğru orantılı olduğu görülmüştür. En yüksek soyulma değeri 80°C altında 28 gün süren numunelerde olduğu görülmüştür. Elde edilen katodik soyulma test


sonuçlarının ilgili standart [1] ve şartnamede [5] yer alan tolerans değerlerin içerisinde olduğu görülmüştür (Tablo 4).



Şekil 5. Katodik test numuneleri

	Ort. Kuru		Test	Test			Ortalam	Ortalama Soyulma	
Panel	Film Kalınlık	Süre	Öncesi	Sonrası	Sıcaklık	Voltaj	Panel	Panellerin Ort.	
	μm		рн	рн			(mm)	(mm)	
1	458	24					1,55		
2	484	saat	8,2	8,4	65°C	-3,5 V	1,95	1,82	
3	429						1,96		
1	452	48					2,01		
2	446	Soot	8,1	8,4	65°C	-1,5 V	1,72	1,94	
3	485	Saat					2,09		
1	432	14 gün					7,66		
2	433		gün	8,7	8,5	65°C	-1,5 V	9,34	7,71
3	476						6,13		
1	457	28					5,21		
2	485	20 aiin	20 8,8	8,2 20°C	-1,5 V	2,83	3,86		
3	562	guii					3,54		
1	458	28					5,91		
2	519	28	8,7	8,4	50°C	-1,5 V	6,53	6,71	
3	428	guii					7,68		
1	461	28					20,13		
2	420	28 gün	<sup>28</sup> 8,7 8,2 65°C -1	-1,5 V	18,53	11,30			
3	421						12,18		
1	494	28				-1,5 V	15,98		
2	501		8,9	8,4	80°C		15,02	15,39	
3	541	guii					15,22		

Tablo 4. Katodik soyulma test parametreleri ve sonuçları

Toz epoksinin metal yüzeyindeki yapışma mukavemetinin tayini için boru üzerinden 100x100 mm ebatlarında çıkartılan numuneler Tablo 5'de verildiği gibi farklı sıcaklıklar altında, farklı sürelerde bekletildikten bir saat sonra kaplama yüzeyinde bir bıçak yardımı ile 30x15 mm ebatlarında açılan dikdörtgen köşeleri incelenmiştir. Yapışma test sonuçları ilgili standard kapsamında uygun aralıktadır (Şekil 6).



Panel	Ort. Kuru Film Kalınlığı	Sıcaklık	Süre	Yapışma Seviyesi
				Panel
1	416 µm		24	1 - 1
2	421 μm	75°C	24	1 - 1
3	409 µm		saat	1 - 1
1	470 μm		4.9	1 - 1
2	512 μm	75°C	48 soot	1 - 1
3	458 μm		saat	1 - 1
1	476 µm		24	1 - 1
2	463 µm	95°C	24 soot	1 - 1
3	436 µm		saat	1 - 1
1	487 µm		20	1 - 1
2	477 μm	75°C	28	1 - 1
3	492 μm		guii	1 - 1
1	586 µm		29	1 - 1
2	613 μm	95°C	2ð	1 - 1
3	621 μm		guii	1 - 1

# **Tablo 5.** Yapışma test parametreleri ve sonuçları



Şekil 6. Yapışma test numuneleri

# 3.4. Kaplamaların Karakterizasyonu

Kaplamadan elde edilen numuneler üzerinde DSC analizi (Şekil 7), Üst yüzey elektron mikroskobu incelemeleri, EDX element analizi ve FTIR analizi ile kimyasal bağ yapısı yanında temas açısı ölçümü ile yüzey ıslatma kabiliyeti ile FBE kaplamanın yüzey özellikleri incelenmiştir. Termal analiz yöntemi ile yapılan ölçümlerde cam geçiş sıcaklıkları Tg3 ve Tg4 sırasıyla ortalama 106 ve 108 °C tespit edilmiştir. Kaplamalı numunelerin uygun sıcaklıkta kaplandığı ve FBE kaplamanın tam kür seviyesine ulaştığı saptanmıştır.



Kaplama tabakasının üst yüzeyinde elektron mikroskobu (SEM-EDX) ile yapılan incelemelerde farklı büyütmelerde görüntüler aşağıda Şekil 8 de sergilenmektedir. Karışım kompozisyonunda mevcut dolgu maddeleri açık renkte ana matrisi oluşturan reçine ise koyu renktedir. İnce uzun iğnesel formlarda gelişigüzel homojen bir şekilde dağılmış olan dolgu malzemeleri açıkça görülebilmektedir. Karışım sonrası yüzeyde dolgu malzemelerinin boyutları 10-14µm arasındadır. EDX analizinde açık renkli görünen dolgu malzemeleri üzerinde Ca, Si, Al, O ve C elementleri gözlenmektedir. Dolgu bileşenlerinin kalsit, alümina ve silika esaslı olduğu ön görülmektedir.



Şekil 8.Kaplamaların üst yüzey SEM incelemeleri ve EDX analizi



Şekil 9.da kaplamanın yüzey ıslatma açısı ölçülmüş olup, damlacık formaları aşağıda sergilenmektedir. Çelik yüzeyin hazırlığına ve kürleme sonrası elde edilen yüzey morfolojisine bağlı olarak ıslatma açısı yüzeyin farklı bölgelerinden alınan ölçüm sonuçlarında 45-60° arasında değiştiği tespit edilmiştir.



Şekil 9. Islatma açısı ölçüm sonuçları

Şekil 10.da kaplamanın üst yüzeyinden farklı bölgelerden alınan FTIR analiz grafiği sunulmaktadır. Her iki grafiğin benzer karakteristiği kaplamanın homojen bir şekilde kürlendiğini göstermektedir. 2800-3100cm<sup>-1</sup> arasında C-H bağları, 3200-3600 cm<sup>-1</sup> arasında O-H bağları, 1400-1600cm<sup>-1</sup> arasında C-C veya C=C bağları, 1080-1000 cm<sup>-1</sup> arasında Si-O, C-O-C bağları gözlenmektedir.



Şekil 10. FTIR analizi sonuçları

# 4. Sonuçlar

FBE kaplamaların çelik boru yüzeylerine başarılı bir şekilde uygulanabildiği gözlenmektedir. Kaplamanın arzulanan homojen kalınlıkta ve kararlı bir yapıya sahip şekilde kürlendiği belirlenmiştir. Uluslararası standard ve testler kapsamında yapılan incelemelerde kaplamaların arzulanan fiziksel ve mekanik özellikleri %100 sağladığı, bunun yanında karakterizasyon çalışmaları kapsamında ise kaplama öncesi işlemler ile kaplama sonrası işlemlerin kaplama kalitesi üzerinde önemli bir etkiye sahip olduğu tespit edilmiştir. Katodik soyulma test sonuçları tek katmanlı kaplamanın performans kriterlerini yerine getirdiğini işaret etmektedir.

#### **References:**

1. CSA Z245.20 Series-14, Plant-Applied External Fusion Bond Epoxy Coating for Steel Pipe, 2014 Edition.

The International Conference of Materials and Engineering Technology

- 2. Aşkar, İ., Petrol Ve Doğalgaz Borularının Tek Kat FBE Kaplanmasında Yüzey Profili Derinliklerinin Katodik Soyulma Performansına Etkileri, Yüksek Lisans Tezi, İTÜ, 2009.
- ISO 8501-1, Preparation Of Steel Substrates Before Application Of Paints And Related Products — Visual Assessment Of Surface Cleanliness — Part 1: Rust Grades And Preparation Grades Of Uncoated Steel Substrates And Of Steel Substrates After Overall Removal Of Previous Coatings, 2007 Edition.
- 4. ISO 8502-3, Preparation of steel substrates before application of paints and related products -- Tests for the assessment of surface cleanliness -- Part 3: Assessment of dust on steel surfaces prepared for painting (pressure-sensitive tape method), 2017 Edition.
- 5. TES-CO-FBE-GL External Fusion Bond Epoxy for Steel Pipe Specification, TransCanada Engineering, 2017 Edition.
- Shan Qian, Y. Frank Cheng; Degradation of fusion bonded epoxy pipeline coatings in the presence of direct current interference, Progress in Organic Coatings, 2018, Volume 120, pp. 79-87.
- F. Mahdavi, M.Y.J. Tan, M. Forsyth, Electrochemical impedance spectroscopy as a tool to measure cathodic disbondment on coated steel surfaces: capabilities and limitations, Prog. Org. Coat., 2015, 88, pp. 23-31.



The International Conference of Materials and Engineering

# ONUR ACAR<sup>1</sup>, İBRAHİM GÖV<sup>1</sup>, M. HANİFİ DOĞRU<sup>2</sup>

<sup>1</sup> Gaziantep University, Faculty of Aeronautics and Aerospace, Aircraft and Aerospace Engineering Department, Gaziantep, TURKEY.

<sup>2</sup> Gaziantep University, Faculty of Aeronautics and Aerospace, Pilotage Department, Gaziantep, TURKEY.

#### Abstract

Flight controllers have been continuously updated and improved in line with the increasing needs of UAV's (unmanned aerial vehicles) from the past to the present. The inadequacy of manual systems contributed to the development of such technologies. In this study, evaluations about advantages and disadvantages of required flight controllers for a drone to be able to remain stable in the air and fulfill its mission are been presented, and for a drone, which flight controller make process commands faster, which is more efficiency etc are decided.

In addition, in this study, the advantages and disadvantages of hardware and software auxiliary ports such as S-BUS, TELEMETRY, SPI (Serial Peripheral Interface) are evaulated. As we know, there are a lot of flight controllers in the market. Flight controllers are divided into closed and open with source code. In general, open source flight controllers are used in project and R & D (research, development) studies. In markets, the current flight controllers have a lot of comparable features of their own. In this study, there are comparisons between open source flight controller and suitable flight controllers for quadcopters just as we compare which one has the more advanced processor and which one has larger the memory size when buying a phone or computer.

Key Words: Flight controller, Drone, Quadcopter, UAV

#### **1. Introduction**

The flight controller is one of the basic required components and it provides autopilot support. Thanks to the autopilot system, control of aircraft on the specified route is provided continuously control without controlled manually. In this way, the pilot gets rid of paying attention to simple flight parameters. As a result, they are like "Fly by Wire" systems and if the computer doesn't work, you can't fly. Flight controllers reduce pilot workload and reduce energy consumption, thus it provides longer battery life. The efficiency of the flight controllers in functional and functional terms has been increased by using peripherals in the new model flight controllers.

The quadcopter is the simplest type of multicopter, with each motor/propeller spinning in the opposite direction from the two motors on either side of it. A quadcopter can control its roll and pitch rotation by speeding up two motors on one side and slowing down the other two. So for example if the quadcopter wanted to roll left it would speed up motors on the right side of the frame and slow down the two on the left. Similarly if it wants to rotate forward it speeds up the back two motors and slows down the front two. The copter can turn ("yaw") left or right by speeding up two motors that are diagonally across from each other, and slowing down the other two. Horizontal motion is accomplished by temporarily speeding up/slowing down some motors so that the vehicle is leaning in the direction of desired travel and increasing the overall thrust of all motors so the vehicle shoots forward. Generally the more the vehicle leans, the faster it travels. Altitude is controlled by speeding up or slowing down all motors at the same time.

Technology



Firstly, the topic that we will evaluate is about to how a FC (flight controller) runs. Of course, there is also artificial intelligence inside of this device as with other device. As well as, you can add something to improve it via open source-code. How can you decide that this control board suits your needs? A flight controller is definitely a powerful and essential part of the quadcopter. In order to choose the best flight controller for your Quadcopter, see below flight controller types.

#### 2. Materials and Methods

## **2.1 PIC (Programmable Intelligent Computer)**

PIC stands for Peripheral Interface Controller and is found in many devices today. First PICs, made with microprocessors were low information processing speed and they had few bit-number. As used in many fields with developing technology, both processor speeds and bit-numbers have been increased. All flight controllers also includes PIC in its structure. In determining a flight controller, it's processor speed and number of bits are one of the important options. Therefore, when specifying a flight controller, we will be careful the following questions.

Processor speed

Number of bits

Processor frequencies

#### **2.2 Flight Controller Types**

A flight controller is a device that normally made up of a microcontroller, sensors, communication busses and input/output pins which gives aircraft stability and control. Before determining a flight control card, we should look at the question of what kind of application it will be used. If used for hobby purposes for viewing and photographing, the number of ports and sensors of the flight controller must support our application. The more technical tasks we assign to the control card, the more software and sensors must be changed.

A control card with open source software allows you to access many features effortlessly. The flight control card must have software support and be up to date. You cannot add extra sensors and assign ports on control cards with closed-circuit software. There are fixed features and you cannot change them. However, if the manufacturer of the flight controller has introduced a new update, a few of changes may be made, but the hardware features of the control card are the same. On open source control cards with port support, you can add a new sensor (GPS, telemetry modem, IMU), and assign new tasks through the control card interface.

# 2.3 Open Hardware

Like open source software, the "source code" for open hardware—schematics, blueprints, logic designs, Computer Aided Design (CAD) drawings or files, etc.—is available for modification or enhancement by anyone under permissive licenses. Users with access to the tools that can read and manipulate these source files can update and improve the code that underlies the physical device. They can add features or fix bugs in the software. They can even modify the physical design of the object itself and, if they wish, proceed to share such modifications.



Example FC's (Flight Controller)

# 2.3.1 Open Hardware FC's (Flight Controller) [1]

- **1.** Pixhawk
- 2. The Cube
- 3. Pixracer
- **4.** CUAV v5
- 5. Beagle Bone Blue
- 6. Erle-Brain
- **7.** F4BY
- 8. OpenPilot Revolution
- **9.** PXFmini RPi Zero Shield
- 10. TauLabs Sparky2

# 2.3.2 Closed Hardware FC's (Flight Controller) [1]

- **1.** Emlid Edge
- **2.** NAVIO2
- 3. Furious FPV F-35 Lightning and Wing FC-10
- 4. Holybro Kakute F4
- 5. Holybro Kakute F7 AIO
- **6.** Holybro Pixhawk 4
- 7. Holybro Pixhawk 4 Mini
- 8. Mateksys F405-STD and variants
- 9. Mateksys F405-Wing
- 10. Omnibus F4 Pro
- 11. Omnibus F7
- 12. Parrot C.H.U.C.K (for the Parrot Disco)
- 13. Parrot Bebop Autopilot
- **14.** RadioLink MiniPix
- 15. SpeedyBee F4

Among of the given examples, FCs that is open source hardware will be able to satisfy our needs and offer improvable software. Therefore, we will compare and introduce features a few among open-hardware control cards. Already we don't need to think about closed-hardware because they have not improvable hardware and software- interface.



# **Pixhawk Overview**



Figure 1. Pixhawk

- 1. Spectrum DSM receiver
- 2. Telemetry (on-screen display)
- **3.** Telemetry (Radio telemetry)
- **4.** USB.
- 5. SPI (Serial Periheral Interface) bus
- 6. Power Module
- 7. Safety Switch Button
- 8. Buzzer
- 9. Serial
- 10. GPS module
- 11. CAN (ControllerArea Network)
- **12.** I2C splitter or compass module
- 13. And 15. Anolog to Digital Converter 6.6 V and 3.3 V
- 14. LED Indicator

_		The In	nternational Conference of Mate	1100-
	Table 1. Pi	xhawk Specificati	on Informations	
Processor		32-bit ARM Cort 168 Mhz/256 K 32-bit failsa	ex M4 core with F B RAM/2 MB Fla ife co-processor	PU sh
Sensors	MPU6000 as main accel and gyro ST Micro 16-bit gyroscope ST Micro 14-bit accelerometer/compass (magnetometer) MEAS barometer			
Power	Servo rail high-power (7 V) and high-current ready			
Interfaces	Futaba S.BUS input (output not yet implemented) I2C, SPI, 2x CAN, USB 3.3V and 6.6V ADC inputs			
Dimensions	Weight 38 g	Width 50 mm	Height 15.5mm	Length 81.5mm

TICME

gineering Technology

The autopilot supports multiple flight stacks, including PX4 and APM, and comes preinstalled with the latest PX4 v1.8 release [1-5]. It powers various multi-copters, fixed-wing aerial platforms, VTOL cargo drones, and even ground based autonomous delivery robots [1]. The applications range from remote sensing in agriculture, security and surveillance, 3D mapping, to robot based deliveries.

# Peripherals

Digital Airspeed Sensor

Telemetry Radio Modules

Rangefinders/Distance sensors

UWE Bristol

Peripherals is important for your quadcopter while operating in the air and making easy to fly smoothly. Peripherals offer information about distance, quantity of battery, airspeed and much more.





# **OpenPilot Revolution and RevoMini**



Figure 2. Open Pilot Revolution

Table 2. CC3D Mini Revolution specification informations			
	STM32F405RGT6 ARM Cortex-M4		
	microcontroller 168		
Processor	Mhz/1 MB Flash		
	InvenSense MPU6000 IMU (accel, gyro)		
	Honeywell HMC5883L compass		
Sensors	MS5611 barometer		
	8V ~ 10V input power provided through ESC		
	connection for fullsize Revolution 5V max on		
Power	RevoMini		
	GPS (SERIAL3) on Flexi Port Telemetry		
	(SERIAL1) on Mainport USB (SERIAL0)		
	port		
	SWD Port for flashing and debugging, including		
Default Interfaces	3.3V output for optional		
	periphereals		

Despite its size the mini CC3D has a fantastic range of functions to manage all of your flight parameters [6]. But number of port and output power are not enough to satisfy our needs.

Intended for use with multirotors, helicopters and fixed wing aircraft in fact in any RC application where stability and navigational systems are required [6].



The International Conference of Materials and Engineering Technology

F4BY FMU



Figure 3. F4 FMU

Table 3. F4BY FMU specification informations				
	Single 32-bit ARM Cortex M4 core with			
Processor	FPU STM32 F407			
	InvenSense MPU6000 IMU			
	(accelerometer gyro)			
Sensors	MS5611 barometers			
	HMC5983 compass			
	Servo rail backup power diode			
	reverse voltage and overvoltage power			
Power	protection			
	board voltage and servo rail voltage			
	sensors			
	5x UART serial ports, 1 with inverter for			
	frsky telemertry			
	Up to 12x PWM outputs			
Interfaces	Futaba S.BUS input support (with			
	external inverter)			
	I2C, SPI, CAN, USB			
	3.3V and 6.6V ADC inputs			
Dimensions	50mm x 50mm			
	micro SD card (for logs)			
Other	Fram memory for parameters			

Table 3 FARV FMU .:.... 



The International Conference of Materials and Engineering Technology

Naze-M Lite



Figure 4. DJI Naza-M Lite

Table 4. DJI Naza M-Lite specification information

	Drive system		
	Automatic balance out of control		
	protection		
	Low voltage protection Support 2 shaf		
Built-in function	Low voltage protectionSupport 2 shart		
	Support S-BUS receiver		
	Support 2 axis holder		
Support multiple rotor aircraft type	e X four rotor style		
	V six rotor style		
	-		
Transmitter	PCM or 2.4GHz ,at least for 4 channel		
Size	Controller Size:45.5mm x 31.5mm x		
	18.5mm		
Weight	master controller:25g		
2	5		

We will evaluate Naze M-lite series flight controller apart from mentioned on ArduPilot Dev Team website because information's about the Naze is not giving on this website [3]. We obtained information's about Naze from other websites and authors.





# Advantages of the Naze-M Lite;

Naze offers you easy placement[3].

Naze has also a magnetometer, barometer and accelerometer sensor[3].

# Disadvantage of the Naze-M Lite;

Even though Naze is minimum size and usage of the easy interface, it only supports 2 multiple rotor types configuration.

# A Briefing About the Our Project;

Let's imagine that designed with single engine and four servos, a quadcopter will be obtaining to propeller power from a single engine with four servos in order to change angle of attack of propellers.

The quadcopter will be improved apart from conventional quadcopters. So that it needs to be changed as programming and hardware. Therefore, it is required a flight controller which has an efficient and good power distribution for electronic equipment's.

What we have to pay attention while selecting flight controllers?

# 2.4 Flying Style

The flying style is very important before purchasing a drone flight controller. Since each flight controller is designed for the specific flying purpose, you must choose a one which suits your needs.

Cinema flying Autonomous flying Sports flying

# 2.5 Open Source Flight Controller

This is one of the important features you have to consider before choosing your flight controller. If the controller has an open source board, then you can do changes onto your board, add or remove features as you like. You can add new devices such as GPS (global positions system), gimbal stabilizer for camera either by using interface program or by updating your programmable flight board.

#### 2.6 Price

Really a key thing to consider before buying any product. But here the price may vary depends on your need. If you like to fly as a hobby, you can choose a cheap flight controller. At the same time, your purpose is to get clear videos or images or even for an event or aerial photography, you must go for somewhat costlier drone flight controller.

# **2.7 Other Important Features**

Without knowing the features of the controller board, how can you choose it? Here are a few features you must keep in mind before buying the best flight controller.

Altitude hold– This is an advanced feature that allows the quadcopter to hover and remain stable at a certain distance from the ground level. There is no need for the pilot does to adjust the quadcopter.

of Materials and Engineering Technology

**GPS**– It not only allows the device to maintain its position but also keeps a record of the flying data for convenience.

**Gyro stabilization**– This keeps the quadcopter stable during the flight time. **Orientation mode**–The pilot can control the orientation of the drone with this feature.

**Position hold**– Similar to the altitude hold, this feature allows the quadcopter to stay at a fixed position during flight times.

**Return home**– Again an advanced feature to get back the lost drone. With a single click, you drone comes to your home. This prevents the loss of your drone.

**Self-leveling**– This allows the quadcopter to stay level in the air by letting go of the pitch.

#### **Powering the Board**

There are two type board. First board is powered from battery source outside, second board is powered from built-up 5V inside of the board regulator which is supplied from battery. The board which is built-up 5V regulator inside of itself connect directly to battery through the socket.

Some boards are powered from 5V;

Five volt are applied to the board from an external voltage regulartor thay you provide

The external regulator is often part of a Power Distribution Board (PDB) that is used to connect the battery lead to the ESC power wires

> The board does not contain an internal voltage regulator

Some boards are powered from VBAT;

Battery voltage is applied directly to the board

> The board contains a built-in voltage regulator to step-down vBat to the 5v that the board uses internally

➤ When vBat boards are connected to a battery, their 5v pads will have 5v power from their internal regulator

#### Advantages of powering the board from VBAT without extra BEC;

Preventing power lost

Avoiding much cable consumption

Gaining volumetric space

Weight reduction





What is protocols? This word has a lot of means; It means generally that to ensure that operations proceed according to a particular procedure.

The International Conference of Materials and Engineering Technology

# **Paralel PWM:**

One set of pins and one wire for each channel Originally designed for use with fixed-wing servos

## Serial (SBUS, iBUS, Spectrum Satallite, etc...):

One signal wire carries all channels

Digital protocol

#### CPPM

One signal wire carries all channels Analog protocol

In some receiver selling website give information about feature of receivers. There are a lot of information about the receiver and that is read both below the receiver pictures and on the receiver. These informations offer us to choose suitable a receiver with protocols for our FC (Flight Controller).

Example; Radiolink R10D

- > The product specs clearly say this receiver outputs SBUS
- SBUS is a serial protocol
- Channel 10 is marked "digital servo only"

#### Protocols advantages;

Protocols aim is to provide a better data flow

Avoiding much cable consumption

Preventing data lost



**3.** Resulting and Discussing

Table 5. Other flight controllers according to Pixhawk					
Features	NAZA M-LITE	CC3D MINI	F4BY FMU		
Interface	DJI Nazam Assistant 2.20	Open Pilot Software	Copter-3.6.0		
Power consumption	5v standart	4.8v and 10v input	3.3v and 6.3v input		
Port number	4 pwm line for motors and 2 position switch	Port line with 6 channels	Port line with 8 channels extra 3 GPIO, 2 Internal and 1 CAN		
Process	STM32 FI 32-bit processor	STM32 32-bit	single 32-bit ARM Cortex M4 core with FPU STM32 F40		
Variety of aircraft model number	2 type	Applies to V wings and quadcopters	Suitable for all models		

The International Conference of Materials and Engineering Technology

Table 6. Disadvantages and advantages of Pixhawk

Price	Disadvantage
Port-number	Advantage
Process speed	Advantage
Volume (used place)	Disadvantage
Open source ease of use	Advantage
Number of aircraft models	Advantage
Power consumption	Advantage

What we desire to improve in our project is to able to managed to different duties with just one board. As you know, the board is represent to flight controller. We selected Pixhawk among of all flight controllers we compared. Pixhawk has not only a lot of advantages but also some disadvantages.



#### Conclusions

With the ever-evolving technology, the features and capabilities of flight controllers are increasing. As a result of this, it becomes very difficult to choose between flight controllers. Among the three flight controllers we choose, Pixhawk is at the forefront in terms of both interface simplicity and extensibility like that based on the references, as well as it has plenty number of ports and provides high current power-out for servos [1-5]. As a result of providing our needs, the pixhawk is the best in this field according to compares we did.

The International Conference of Materials and Engineering Technology

#### Limitations!

The capability of the platforms is changing all the time so this data may already be out of date when you view it!!!

This may be useful for pilots who just want a high level view of what the difference are... Because used materials are professional materials and only recommended for professional drone pilots.

#### **References:**

- Copyright 2019.ArduPilot Dev Team. Introducing Pixhawk® 4 the flight controller for developers. (http://ardupilot.org/copter/docs/common-pixhawk-overview.html) (http://ardupilot.org/copter/docs/common-f4by.html) (http://ardupilot.org/copter/docs/common-autopilots.html#closed-hardware)
- 2. Copyright ©2019 Red Hat, Inc. (https://opensource.com/resources/what-open-source)
- 3. NAZA-M LITE web page (<u>https://www.dji.com/</u>) for latest information from Copyright © 2018 DJI All Rights Reserved.
- 4. PIC Programlama, Devrim Çamoğlu (https://cdn.shopify.com/s/files/1/0158/0204/files/pic\_programlama\_icindekiler.pdf)
- 5. Agris Kipurs, CEO Airdog, Armin Ambühl, CTO Wingtra, A decade of rich history and a fast growing community. (<u>http://pixhawk.org/</u>)
- 6. Mini CC3D Revolution Setup Guide www.Hyperion-world.com Mini CC3D Revolution Setup Guide http://www.dronetrest.com/t/cc3d-flight-controller-guide/830

# INVESTIGATION OF THE EFFECT OF CARBONATION CAKE ON RHEOLOGICAL PROPERTIES OF BITUMINOUS BINDERS

The International Conference of Ma

aterials and Engineering Technology

# AHMET MÜNİR ÖZDEMİR<sup>1</sup>, ERKUT YALÇIN<sup>2</sup>, HASAN ARSLANOĞLU<sup>3</sup>, MEHMET YILMAZ <sup>\*4</sup>, BAHA VURAL KÖK <sup>5</sup>

<sup>1</sup> Firat University, Engineering Faculty, Department of Civil Engineering, Elazig, TURKEY.
<sup>2</sup> Firat University, Engineering Faculty, Department of Civil Engineering, Elazig, TURKEY.
<sup>3</sup> Firat University, Engineering Faculty, Department of Chemical Engineering, Elazig, TURKEY.
<sup>4</sup> Firat University, Engineering Faculty, Department of Civil Engineering, Elazig, TURKEY.
<sup>5</sup> Firat University, Engineering Faculty, Department of Civil Engineering, Elazig, TURKEY.

#### Abstract

Aggregates and bitumen used in road flexible pavements are non-renewable natural materials and in terms of sustainability, it is of great importance that the service life of road pavements is long. Waste materials are also rising with increasing products due to industrial development. The evaluation of waste materials both prevents the destruction of nature by reducing the use of very limited natural materials and reduces the environmental problems in case of storage of waste materials. In this study, carbonation cake, which is a waste of sugar factory, was added to bitumen. Penetration, softening point, rotational viscosity and dynamic shear rheometer tests were applied on pure and modified bitumen. According to the results, it was determined that carbonation cake had no positive or negative effect on medium and high temperature rheological properties of bitumen. From the experiments, promising results have been obtained for the disposal of waste carbonation cake in a different area.

Keyword: Carbonation Cake, Bitumen, Addition, Rheology.

#### **1. Introduction**

Almost all highways in Turkey was applied in the form of flexible pavements and there are 63415 km of asphalt pavements on motorways, state and provincial roads as of 2019 [1]. The basic materials used in highway pavements are aggregate and bituminous binders. Aggregates are obtained by crushing and sieving suitable rocks, while bituminous binders are obtained by refining oil. Aggregates and bitumen used in hot mix asphalts are non-renewable materials and re-use rates are low at the end of the service life of the pavements. Therefore, it is necessary to produce solutions that increase the service life of the pavement during design, construction and application. Due to the development of the industry, the amount of waste material is increasing day by day. Ashes formed in thermal power plants, blast furnace and steel mill slag in iron and steel industry, kiln dusts which are by-products of cement industry, marble dust wastes formed in marble industry, scrap automobile tires, glass shards obtained from glass industry are classified as industrial solid waste. In the world, most of these industrial wastes have the possibility to be used in every layer from the base course to the wearing course on highways [2]. Some of the waste materials (such as slag, fly ash and marble dust) are used as aggregates, while others (such as rubber and shingles) can be used as bitumen additives [3-9]. In our country, approximately 11 million tons of sugar beets are processed every year during the sugar production campaign. During this production, approximately 1 million tons of carbonation cake with 50% dry matter is disposed as waste (Web). At the end of the sherbet treatment processes applied in sugar production from sugar beet, carbonation cakes are formed which contain a large amount of calcium carbonate and also contain colloidal substances of organic origin coming from beet. These



cakes are then thrown by mixing with water. These residues, which have a high pollution load of organic matter, are removed by purification in some sugar factories. In many sugar factories, they are directly fed to surface waters. Various methods have been proposed for the recovery of lime, its use in cement production and the use of acidic soils for the evaluation of these wastes [11-13]. In this study, the usability of the carbonating cake, a waste material during sugar production, as a bitumen additive was investigated. The carbonation cake was added to the bitumen in 3 different ratios by weight of bitumen. The penetration, softening point, rotational viscosity and dynamic shear rheometer experiments were performed on the modified bitumen and the effect of carbonation cake on the rheological properties of bituminous binders was evaluated.

#### 2. Materials and Methods

A B50/70 class bituminous binder procured from Batman TÜPRAŞ refinery was used as the main binder. Carbonation cake, which was used in the experiments, was obtained from Elazığ Sugar Factory in 2016 campaign period. The carbonation cake was supplied from the outlet of the rotary filter. The dried carbonation cake plant grinder was also milled and sieved. The -200 mesh fraction was dried for 2 hours in an oven at 80°C and kept in glass jars with lids during the experiments. The chemical composition of the carbonation cake used in the experiments is shown in Table 1 in terms of oxides. The XRD diffractogram, SEM-EDX image and particle size distribution of the carbonation cake were also characterized.

Component	Compound (%)	
CaO	49.8	
MgO	3.56	
K <sub>2</sub> O	0.11	
Na <sub>2</sub> O	0.15	
FeO	0.12	
Analysis undetectable	2.46	
Weight Loss (50-850 °C)	43.8	

Table 1. Chemical composition of carbonation cake used in experiments

In Figure 1, it is seen from the SEM image that the carbonation cake has a small particle structure. It was also determined from the EDX analysis that the calcium compound in the structure was intensely present. It is seen that the carbonation cake has a particle size of less than 30  $\mu$ m. The carbonation cake can be considered to have formed as a very small particle by the reaction of dissolved calcium hydroxide with carbon dioxide in the carbonation process (Figure 2).



Fig. 2. Particle size distribution of carbonation cake

Modified bitumen were obtained by the addition of carbonation cake in the rates of 5%, 10%, and 15% per bitumen weight to the bitumen. Mixing temperature was selected as 180°C during the preparation of modified bitumen. Neat bitumen and the bitumen modified with the selected ratios of additive were stirred at 1000 rpm rate of rotation for 1 hour (Figure 3).



Fig 3. Modified bitumen mixer and mixing cap

Penetration (EN 1426), softening point (EN 1427), rotational viscometer (ASTM D 4402), and dynamic shear rheometer (AASHTO TP5) tests were conducted on neat and modified bitumens.

# **3. Experimental Study**

Results of the penetration tests conducted on neat and modified binders are displayed in Figure 4. As shown in Figure 4, when the 5%, 10% and 15% carbonation cake was added to the pure bitumen, there was no significant change in penetration values. Although the penetration values of 5% and 15% carbonated cake added binders appear slightly higher than other binders, this change does not make a significant difference when evaluated for consistency.



Fig 4. Average penetration values of pure and modified binders.

Softening point test results of neat and modified binders are shown in Fig. 5 As shown in Figure 5, softening point values were slightly increased by adding carbonation cake to pure bitumen. Similar softening point values were obtained by adding 5%, 10% and 15% carbonation cake. With the addition of 5% carbonation cake to the pure bitumen, the softening point value increased by 4.2% compared to pure bitumen, 4.7% when 10% was added and 4.9% when 15% was added.

Technology

Materials and Engineering



Fig 5. Softening point values of binders.

The penetration indexes showing the heat sensitivity of pure and modified bitumen were determined using the following formulas:

$A = \frac{\log 800 - \log P_{25}}{T_{YN} - 25}$	(1)
$PI = \frac{20 - 500A}{1 + 50A}$	(2)

The penetration index values of the binders are shown in Figure 6. As can be seen in the figure, the penetration index values were generally increased with the use of additives compared to the pure binder. This increase in penetration indexes indicates that the use of carbonating cake reduces the heat sensitivity of the pure binder.



Fig 6. Penetration index values of binders



The results obtained from the rotational viscometer tests applied to the binders at 135°C and 165°C are shown in Figure 7. When the viscosity values at 135°C were examined, it was found that the lowest value was 10% carbonation cake added binder and the highest value was 15% carbonation cake added binder. At 165°C, it was determined that there was a very small difference (5.7%) between the viscosity values of all binders. Although the biggest difference between viscosity values is between binders containing 10% and 15% carbonation cake (14.3%) at 135°C, when the most commonly used bitumen additives styrene-butadiene-styrene (SBS) (14) and waste rubber (CR) (15) additives are taken into consideration, this change can be said to be negligible. From the obtained results, it was determined that not all binders exceed the maximum 3000 cP which is the limit value in terms of pumpability.



Fig 7. Viscosity values of binders at 135 and 165°C

In order to determine the effect of the use of carbonation cake as bitumen additive on the mixing and compaction temperatures of hot mix asphalt production, temperature values at recommended viscosities ( $280 \pm 30$  cP for compaction and  $170 \pm 20$  cP for mixing) were determined (16). The calculated values are given in Table 2. It was determined from the obtained results that modified bitumens containing three different ratios of carbonation cake need similar temperature and similar energy during mixing with aggregate.

		Equation	Mixing temperature range (°C)	Compaction temperature range (°C)
B 50/70		$y = 166954e^{-0.04x}$	169,5-175,4	157,2-162,6
Componetion	5%	$y = 200860e^{-0.041x}$	169,8-175,6	157,9-163,2
Carbonation	10%	$y = 171106e^{-0.041x}$	165,9-171,7	154,0-159,2
Cake	15%	$y = 372229e^{-0.045x}$	168,4-173,7	157,6-162,4

Table 2 Mixing and co	mpaction temp	perature ranges o	f pure and	modified	bitumens
-----------------------	---------------	-------------------	------------	----------	----------

Dynamic shear rheometer (DSR) test is used to evaluate the viscoelastic behavior of bituminous binders at medium and high temperatures. The DSR test characterizes the viscous and elastic behavior of asphalt cement by determining the complex shear modulus (G\*) and phase angle ( $\delta$ ). G\* is an indicator of the total resistance of asphalt cement against deformations caused by repeated shear stresses.



Both G\* and  $\delta$  values vary significantly with the heat and loading speed of asphalt cement (16-17). In this study, neat and modified bitumen samples were filled into 25 mm diameter silicone containers and cooled to ambient temperature. The samples were then placed in the DSR test instrument until the temperature of the test was reached and the different temperature was tested. The effect of the additives on the rheological behavior of the pure binder was tried to be evaluated in a wider range by applying the DSR test. Rutting parameters (G\*/sin $\delta$ ) obtained from the DSR experiment are shown in Figure 8 and phase angles are shown in Figure 9.



Fig 8. Variation of rutting parameters of binders with temperature.

When Figure 8 is examined, the rutting parameters of all binders decreased with increasing temperature. It was determined that the binder containing 5% and 10% carbonation cake with pure binder had similar rutting parameter values. According to the Superpave specification, pure binders containing 5% and 10% carbonation cake meeting the 1000 Pa requirement provided 70°C and binder containing 15% carbonation cake at 76°C. According to the results, it was determined that the additive binders other than 15% carbonation cake did not significantly change the rutting parameter compared to pure bitumen.



Fig 9. Variation of the binder phase angle values with temperature.

Figure 9 shows that the phase angle values increase with increasing temperature, hence they exhibit more viscous behavior. It was found that the binder containing 5% and 10% carbonation cake and pure binder, had similar phase angle values and only the binder containing 15% carbonation cake had lower phase angle values than the other binders. It can be said from the phase angle values that the elasticity of the binders increases with the use of 15% carbonation cake by bitumen weight.

The International Conference of Materials and Engineering Technology

## 4. Conclusions

In this study, the usability of carbonating cake which is a waste material in sugar factories as bitumen additive was investigated. For this purpose, 3 different ratios (5%, 10% and 15%) of the bitumen weight carbonation cake were added to the pure bitumen. Penetration, softening point, rotational viscosity at 2 different temperatures (135 and 165°C) and dynamic shear rheometer at 5 different temperatures (52, 58, 64, 70 and 76°C) were applied on pure and modified bitumens. It was found from the obtained results that the penetration values did not change significantly with the use of carbonation cake, the softening point values increased by about 5%, and the sensitivity of the binders to heat decreased with the use of carbonation cake. It was observed from the rotational viscosity tests that the use of a 15% carbonation cake at 135°C increased the viscosity values, that all binders at 165°C had similar values, and that there was no significant difference in mixing with aggregate and compaction temperatures. Dynamic shear rheometer experiments show that all binders exhibit more viscous behavior with increasing temperature, binders containing 5% and 10% carbonation cake have similar rutting parameters and phase angle values, whereas binder containing 15% carbonation cake has higher rutting strength and lower phase angle values.

When all the test results were evaluated, it was determined that the medium and high temperature rheological properties of the bituminous binder were not adversely affected when carbonation cake was added to the bitumen. This is hopeful that the waste carbonation cake can be used in a different area to prevent environmental damage. However, in order to determine the usability of a material as bitumen additive, the effect of hot mix asphalts on different parameters such as resistance to low temperature cracks, moisture damage and fatigue cracks should be examined. Therefore, it would be useful to prepare hot mix asphalt samples with binders containing carbonation cake and keep them in various performance tests.

# **References:**

- 1. http://www.kgm.gov.tr/SiteCollectionDocuments/KGMdocuments/Istatistikler/DevletIIYolE nvanter/SatihYolAgiUzunlugu.pdf
- **2.** Çağlar, G.A., Endüstriyel atık malzemelerin karayollarında kullanımı. Yüksek Lisans Tezi. İstanbul Teknik Üniversitesi. 2007.
- **3.** Şengöz, B., Topal, A. Use of asphalt roofing shingle waste in HMA, Construction and Building Materials, 2005, 19(5): 337-346.
- **4.** Karaşahin, M., Terzi, S. Evaluation of marble waste dust in the mixture of asphaltic concrete. Construction and Building Materials, 2007, 21(2): 616-620.
- **5.** Okubay, M., Kök, B.V., Yardım, M.S., Yılmaz, M. Agrega olarak mermer atığı kullanımının bitümlü sıcak karışımların nem hasarı üzerindeki etkisi. Journal of Balıkesir University Institute of Science and Technology, 2018, 20(2):495-507.
- 6. Kök B.V., Yılmaz, M., Akpolat, M. Effect of CR and FT-paraffin versus SBS modification in terms of conventional and rheological properties, International Journal of Pavement Engineering, 2017, 18 (12):1052-1059.

**7.** Xiang L., Cheng, J., Que, G. Microstructure and performance of crumb rubber modified asphalt. Concstruction and Building Materials, 2009, 23: 3586-3590.

The International Conference of Materials and Engineering Technology

- **8.** Yılmaz, M. ve Kök, B. Effects of ferrochromium slag with neat and polymer modified binders in hot bituminous mix. Indian Journal of Engineering & Materials Sciences, 2009, 16(5): 310-318.
- **9.** Alataş, T., Kizirgil, M.E. The Effects of using Styrene-Butadiene-Styrene and Fly Ash Together on the Resistance to Moisture-Induced Damage, Permanent Deformation and Fatigue of Hot Mixture Asphalt. KSCE Journal of Civil Engineering, 2013,17(5): 1030-1039.
- 10. Web: https://www.turkseker.gov.tr/PersonelSekersatisEkimUretim.aspx (12.04.2019)
- **11.** Magdy, Y. H., & Daifullah, A. A. M. Adsorption of a basic dye from aqueous solutions onto sugar-industry-mud in two modes of operations. Waste Management, 1998, 18(4): 219-226.
- **12.** Güler, O., Gür, F., Özer, A. ve Tümen, F. A study on the removal of heavy metals by carbonatation cake discarded in sugar industry. International Sugar Journal, 2002, 104(1246): 458-462.
- **13.** Vaccari, G., Tamburini, E., Sgualdino, G., Urbaniec, K., & Klemeš, J. Overview of the environmental problems in beet sugar processing: possible solutions. Journal of Cleaner Production, 2005, 13(5): 499-507.
- **14.** Kaya, D., Topal A., McNally, T. Relationship between processing parameters and aging with the rheological behavior of SBS modified bitumen. Construction and Building Materials, 2019, 221: 345-350.
- **15.** Liang, M., Xin, X., Fan, W., Luo, H., Wang, X., Xing, B. Investigation of the rheological properties and storage stability of CR/SBS modified asphalt. Construction and Building Materials, 2015, 74: 235-240.
- **16.** Zaniewski, J.P., Pumphrey, M.E. Evaluation of Performance Graded Asphalt Binder Equipment and Testing Protocol, West Virginia University, Morgantown, 2004, 109p.
- McGennis, R.B., Anderson, R.M., Kennedy, T.W., Solaimanian, M. Background of Superpave Asphalt Mixture Design and Analysis, U.S. Department of Transportation Federal Highway Administratio, 1995, Publication No. FHWA-SA-95-003, 172p.



# GREY WOLF OPTIMIZATION BASED PTS SCHEME FOR PAPR REDUCTION IN OFDM SYSTEMS

# YÜKSEL TOKUR BOZKURT

Gaziantep University, Vocational School of Technical Sciences, Department of Electronics and Automation, Gaziantep, TURKEY

# Abstract

The problem of PAPR reduction in OFDM has been the subject of much academic research in recent years. These researches have been chiefly prompted by the fact that high PAPR has existed as a potential source of inefficiency in OFDM systems. Partial Transmit Sequence (PTS) scheme is one of the prominent and powerful techniques to reduce PAPR in OFDM systems. However, due to the substantial computational complexity, searching optimum phase factors, especially for large number of subblocks, requires huge computations, thereby eliminating its application to practical systems. The object of this work is to develop Grey Wolf Optimization based PTS scheme for PAPR reduction in OFDM systems and examine its efficiency through a variety of simulations. Cumulative distribution function performance (CCDF) for PSO-PTS, HS-PTS, RS-PTS, CS-PTS, and the conventional PTS systems are plotted to comparatively analyze. It is revealed from the CCDF plot and computational complexity analyses that the PAPR reduction and computational efficiency of the proposed scheme is better than the conventional PTS scheme.

Keyword: PAPR, PTS, GWO, OFDM.

# **1. Introduction**

Orthogonal frequency division multiplexing (OFDM) systems have attracted significant interest in the past few decades due to their high bandwidth performance, robustness to frequency selective fading, implementation efficiency, and narrowband interference [1-4]. OFDM systems also provide high capacity data transmission with higher bit rate [5]. With these outstanding properties, OFDM systems have become promising technique for the application of digital communication. However, OFDM systems suffer from high peak-to-average power ratio (PAPR) [6] which can cause significant reductions in bit error rate (BER) performance and efficiency of radio frequency power amplifiers and, in turn, leads to a complexity in analog-to-digital and digital-to-analog converters.

Over the years, several researches have been proposed to suppress PAPR in OFDM systems, such as tone reservation [6], clipping [7], interleaving [8], active constellation extension [9], tone injection [10], peak windowing [11], coding [12], selected mapping [13], clipping and filtering [14], and partial transmit sequence (PTS) [15]. With remarkable PAPR reduction performance, PTS scheme is one of the talented and distortionless methods to reduce PAPR in OFDM systems [15]. However, PTS scheme requires an exhaustive search operation for the selection of optimum phase factors which resulting with the decrements in its potential for practical applications due to reduction in computational efficiency. In recent years, an increasing interest has emerged on the selection of optimum phase factors using a suboptimal search algorithm in which computational complexity of search procedure is suppressed. Various studies devoted to the combination of PTS scheme with a suboptimal search algorithm such as random search [15], harmony search (HS) [16], artificial bee colony (ABC) [17], particle swarm optimization (PSO) [18], differential evolution (DE) [19], and parallel tabu search (parallel-TS) [20] has been presented, in the literature, for the PAPR reduction with less computation load.

Despite the large number of publications on the combination of PTS scheme with a suboptimal search algorithm, there is very little literature about the phase factor selection performance of grey wolf optimization algorithm. Therefore, the aim of this work is to analyze the PAPR reduction performance of grey wolf optimization-based PTS scheme in OFDM systems. A set of simulations were performed in MATLAB environment to evaluate PAPR reduction and computational complexity as a measure of the performance of the proposed scheme.

e International Conference

# 2. Materials and Methods

# 2.1. System Model

A block diagram of the system model for PTS scheme based OFDM system, used in simulations, is shown in Figure 1. First, the burst errors induced in communication channel are eliminated through interleaving input bit streams that coming from the users. Following the modulation of interleaved signals with QAM, PTS scheme is employed for PAPR reduction. A side information is transmitted to acquire original OFDM signal in the receiver. In order to suppress intersymbol interference (ISI) induced in the communication channel, a cyclic prefix (CP) is introduced into signal after the amplification, by high power amplifier (HPA). After the removement of CP from the transmitted signal at the receiver, fast Fourier transform (FFT) is implemented and phase of the original OFDM signal is obtained with the help of phase rotation and side information. Finally, the symbols are demodulated and transferred into their original place in the bit sequence with the help of deinterleaver [20].



Figure 1. Block diagram of the system model.

# 2.2. Conventional PTS

In a conventional PTS scheme, the input signal is sectioned into a number of disjoint sub-blocks, each with a set of sub-carriers in equal size, and is mathematically expressed by

$$x(t) = \frac{1}{\sqrt{M}} \sum_{m=0}^{M-1} X_k e^{j2\pi f_k t} \qquad 0 \le t < MT$$
(1)

where X (in the form of  $X = [X_0, X_1, ..., X_{M-1}]$ ) and *M* are input data vector and the number of subcarriers. The symbols in the input data vector are mapped by modulating with 16-QAM and assigning to subcarriers at a frequency of  $f_k$ . The frequency of a subcarrier is stated as  $f_k = k/MT$ , where *k* and *T* are the index number of subcarrier and the symbol period of an OFDM signal.

Technology

Due to discrete-time signal requirement of PTS scheme for PAPR reduction, the input signal is subjected to inverse fast Fourier transform to transfer signal into time domain and is stated by

$$x(t) = \frac{1}{\sqrt{M}} \sum_{m=0}^{M-1} X_k e^{j2\pi km/LM} \qquad 0 \le m < LM$$
(2)

and subblocks are transformed into

 $X_m = [x_0^m, x_1^m, ..., x_{LM-1}^m]$   $0 \le m \le M-1$  (3) where *L* is the oversampling factor and 4 as the value of oversampling factor is sufficient to accurately express the PAPR in the discrete-time domain. Each subblock is weighted with a phase rotation vector,  $b_m = e^{j\emptyset}$ , where  $\emptyset \in [0, 2\pi]$ . After the weighting, subblocks are summed and OFDM signal becomes

$$x'(t) = \sum_{m=0}^{M-1} b_m x^m$$
 (4)

Technology

By taking the phase factor for the first sub-block as  $b_0 = 1$ , there are  $W^{M-1}$  different combinations for b, where W is the number of allowed phase factors. In a phase rotation vector, the values of  $b_m$  are as follows:

$$b_m = \begin{cases} \pm 1 & \text{for } W = 2\\ \pm 1, \pm j & \text{for } W = 4 \end{cases}$$
(5)

The operable block diagram of a distinctive PTS combined grey wolf algorithm optimization is shown in Figure 2.





#### 2.3. The Grey Wolf Optimizer (GWO) algorithm

The Grey Wolf Optimizer (GWO) algorithm a recently developed meta-heuristic optimization algorithm, proposed by Mirjalili et al. [21]. It mimics hunting operation of grey wolves' swarm. Wolves are generally social animals and exhibit cooperative behaviors in a swarm by obeying rigid social hierarchy. The wolves in a swarm are grouped into four groups with respect to leadership hierarchy and from the highest to lowest level, are termed as alpha, beta, delta and omega. The hunting operation of wolves based on determination of the best area in search of prey with the guidance of alpha, beta and delta wolves to other wolves.

After the identification of prey, wolves encircle it to approach by constructing a circle. Hunting operation that comprised of searching, encircling and attacking prey, is mathematically expressed using the vector functions as follows;

$$\vec{G} = \left| \vec{H} \cdot \vec{X}_p(t) - \vec{X}(t) \right| \tag{6}$$

Technology

$$\vec{X}(t+1) = \vec{X}_p(t)\vec{G} \tag{7}$$

where  $\vec{X}_p(t)$ ,  $\vec{X}(t)$ ,  $\vec{G}$ ,  $\vec{H}$ , and t are position vector of prey, grey wolf position, new position of grey wolf, coefficient vector and the number of iterations, respectively. The values of H and G vectors are re-evaluated for each new position vectors.

$$\vec{G} = 2\vec{h}\cdot\vec{r_1} - \vec{h} \tag{8}$$

$$\vec{H} = 2 \cdot \vec{r_2} \tag{9}$$

where  $\vec{h}$  is the coefficient vector that linearly decreases from 2 to 0 for each iteration,  $\vec{r_1}$  and  $\vec{r_2}$  are the random vectors located in the scope [0, 1]. Usually, alpha and beta performs the hunting operation with the supports of delta to the alpha. In GWO algorithm, due to leadership hierarchy, best solution is attributed to alpha, the second and third best solutions are beta and delta.

## 3. Results and Discussion

The OFDM signals having N = 256 sub-carriers with 16-QAM modulation were randomly generated for computer simulations. Different number of the generation/the iteration *G*, different number of the population/the particle *P*, number of the phase factor W = 2, and the number of sub-blocks M = 16 were selected. The fundamental parameters of the simulations were listed in Table 1.

Table 1.	Parameters	for	simulations
Labit I.	i urumeters	101	simulations

Quantity	Symbol	Value	
modulation method	QAM		
number of phase factor	W	2 (±1)	
number of the population	Р	20	
number of the iteration	G	50	
number of sub-blocks	Μ	16	
number of sub-carriers	N	256	

Table 2 shows comparison of computational complexity among different methods for M = 16, N = 256 and W = 2.

Table 2.	Computational	Complexity	of the	PTS	Methods
----------	---------------	------------	--------	-----	---------

Method	Search	PAPR
Original	0	10.95
HS-PTS	1000	7.44
PSO-PTS	1000	7.26
RS-PTS	1000	7.2
CS-PTS	1000	7.04
GWO-PTS	1000	6.9
O-PTS	32768	6.37



In Figure 3, the performance of the proposed scheme is compared with the optimum, the random search and different suboptimal based-PTS PAPR reduction schemes used. When CCDF=10-3, the PAPR of the original is 10.95 dB, the random search (RS) is 7.2 dB, the PAPR values for and the suboptimal methods are 7.04 dB for the cuckoo search (CS), 6.37 dB for the Optimum-PTS (O-PTS) and 6.9 dB for the proposed system. For same search complexity, the PAPR of the GWO-PTS is smaller 0.3 dB, 0.14 dB than that of RS-PTS and CS-PTS respectively. The comparison shows that the GWO-PTS gives better PAPR reduction compared with RS-PTS and CS-PTS in OFDM system.



**Figure 3.** The PAPR<sub>0</sub> (dB) versus CCDF of the original, RS-PTS, CS-PTS, GWO-PTS and optimum PTS.

In Figure 4, the performance of the proposed scheme is compared with the optimum, and the particle swarm optimization (PSO)- PTS and the harmony search (HS)-PTS PAPR reduction schemes used. When  $CCDF = 10^{-3}$ , the PAPR of the original is 10.95 *dB*, the PAPR values for and the suboptimal methods are 7.44 *dB* for the PSO-PTS, 7.26 *dB* for the HS-PTS, and 6.9 *dB* for the proposed system. For same search complexity, the PAPR of the GWO-PTS is smaller 0.54 *dB*, 0.36 *dB* than that of PSO-PTS and HS-PTS, respectively. The comparison shows that the GWO-PTS gives better PAPR reduction compared with HS-PTS and PSO-PTS in OFDM system.



**Figure 4.** Comparison of the PAPR<sub>0</sub> (dB) versus CCDF the original, HS-PTS, PSO-PTS, GWO-PTS and optimum PTS.

#### 4. Conclusions

In this paper, we present PTS based on grey wolf algorithm in OFDM system to handle the PAPR problem to reduce the complexity. The CCDF simulations are performed to interpret the PAPR reduction performance of the proposed GWO-PTS scheme. Also, performance of proposed method is compared with the PTS performances of optimum, original, random search, cuckoo search, particle swarm optimization and harmony search. Simulation results show that the PAPR reduction of the GWO-PTS with less computational load performance than Optimum-PTS is better than that of PTS performances of original, random search, cuckoo search, particle swarm optimization and harmony search in the OFDM system.

#### References

- 1. Müller, S.H., and Huber, J.B. OFDM with reduced peak-to-average power ratio by optimum combination of partial transmit sequences. Electron Lett ,1997, 33: 368-369.
- 2. Chini, A., Wu, Y., El-Tanny, M., and Mahmoud, S. Hardware nonlinearity's in digital TV broadcasting using OFDM modulation. IEEE T Broadcast, 1998, 44: 12-21.
- 3. Costa, E., Midrio, M., and Pupolin, S. Impact of amplifier nonlinearities on OFDM transmission system performance. IEEE Commun Lett, 1999, 3: 37-39.
- 4. Jiang, T., and Wu, Y. An overview: peak-to-average power ratio reduction techniques for OFDM signals. IEEE T Broadcast, 2008, 54: 257-268.

5. Cimini, L.J. Jr. Analysis and simulation of a digital mobile channel using orthogonal frequency division multiplexing, IEEE Trans. Commun, 1985, 33: 665–675.

The International Conference of Materials and Engineering Technology

- 6. Chen, J.C., and Li, C.P. Tone reservation using near-optimal peak reduction tone set selection algorithm for PAPR reduction in OFDM systems. IEEE Signal Proc Let, 2010, 17: 933-936.
- Krongold, B.S., and Jones, D.L. PAR reduction in OFDM via active constellation extension. IEEE T Broadcast, 2003, 49: 258-268.
- 8. Wang, J., Guo, Y., and Zhou, X. PTS clipping method to reduce the PAPR in ROF-OFDM system. IEEE T Consum Electr, 2009, 55: 356-359.
- 9. Jayalath, A.D.S., and Tellambura, C. Reducing the peak-to- average power ratio of an OFDM signal through bit or symbol interleaving. Electron Lett, 2000, 36: 1161-1163.
- 10. Chen, J.C., and Wen, C.K. PAPR reduction of OFDM signals using cross-entropy-based tone injection schemes. IEEE Signal Proc Let, 2010, 17: 727-730.
- 11. Chen, G., Ansari, R., and Yingwei, Y. Improved peak windowing for PAPR reduction in OFDM. In: IEEE Vehicular Technology Conference; 26–29 April 2009; Barcelona, Spain. New York, NY, USA: IEEE. pp. 1-5.
- 12. Jones, A.E., Wilkinson, T.A., and Barton, S.K. Block coding scheme for reduction of peak to mean envelope power ratio of multicarrier transmission scheme. Electron Lett, 1994, 30: 2098-2099.
- 13. Jie, Y., Lei, C., Quan, L., and De, C. A modified selected mapping technique to reduce the peak-to-average power ratio of OFDM signal. IEEE T Consum Electr, 2007, 53: 846-851.
- 14. Li, X., and Cimini, L.J. Effect of clipping and filtering on the performance of OFDM. IEEE Commun Lett, 1998, 2: 131-133.
- 15. Cimini, L.J.Jr., and Sollenberger, N.R. Peak-to-average power ratio reduction of an OFDM signal using partial transmits sequences. IEEE Commun Lett, 2000, 4: 86-88.
- 16. Gao, J., Wang, J., and Wang, B. Improved particle swarm optimization for PAPR reduction of OFDM systems. In: International Conference on Networking, Sensing and Control; 10–12 April 2010; Chicago, IL, USA. New York, NY, USA: IEEE. pp. 621-624.
- 17. Hung, H.L., and Huang, Y.F. Peak-to-average power ratio reduction in orthogonal frequency division multiplexing system using differential evolution-based partial transmit sequences scheme. Commun IET, 2012, 6: 1483-1488.
- 18. Taşpınar, N., Karaboğa, D., Yıldırım, M., and Akay, B. PAPR reduction using artificial bee colony algorithm in OFDM systems. Turk J Electr Eng Co, 2011, 19: 47-58.
- 19. Taşpınar, N., Kalınlı, A., and Yıldırım, M. Partial transmit sequences for PAPR reduction using parallel tabu search algorithm in OFDM systems. IEEE Commun Lett, 2011, 15: 974-976.
- 20. Kermani, E.M, Salehinejad, H., and Talebi, S. PAPR reduction of OFDM signals using harmony search algorithm. In: 18th International Conference on Telecommunications; 8–11 May 2011; Ayia Napa, Cyprus. New York, NY, USA: IEEE. pp 90-94.
- 21. Mirjalili, S., Mirjalili, S.M., and Lewis, A. (2014) Grey wolf optimizer. Adv Eng Softw , 2014, 69(12): 46-61.

# THE EFFECTS OF FLUX TYPE ON MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF S235 STRUCTURAL STEEL BY SUBMERGED ARC WELDING

# AZİZ BARIŞ BAŞYİĞİT<sup>\*1</sup>, BÜŞRA SOLAK<sup>2</sup>.

 <sup>1</sup> Kırıkkale University, Faculty of Engineering, Department of Metallurgical and Materials Engineering, Kırıkkale, TURKEY. \*abbasyigit@kku.edu.tr
<sup>2</sup> Kırıkkale University, Faculty of Engineering, Department of Mechanical Engineering, Kırıkkale, TURKEY.

# Abstract

S235 fine grained steel alloys are demanded especially in structural components of buildings, factories and mechanical parts of machines where the tensile strength values approximately close to minimum 370 MPa. are in concern. As they are classified in low alloyed groups, in consequence of the cheapness of these alloys their consumption is increasing as well.

In this work; 15mm of S235 structural steel is submerged arc welded by 2 types of welding fluxes. The effects of flux composition on microstructural and mechanical properties of S235 steel alloy is examined. Transverse tensile tests, microvickers hardness surveys and microstructural invesigations are applied for comparison of these two types of welding fluxes.

Keywords: Fine grained steel alloys, S235 steel alloy, Submerged arc welding, Welding fluxes.

# 1. Introduction

Fine grained steel alloys are mainly used in structural parts and also at buildings in various industries. The consumption of these alloys is increasing day by day in consequence of the low alloying elements and hence low costs in products. They have good weldability properties. They are classified according to their minimum yield strength values. They have been also grouped by minimum tensile strength values prior to their yield strength classifications. S235 wrought fine grained steel is alloyed to ensure minimum 235 mega pascals of yield strength values [1,2].

These alloys are joined with numerous kinds of fusion welding methods as mostly pronounced electrode arc and MIG/MAG welding processes also with screwing and fastening by rivets. Submerged arc welding process is preferred for joining thick parts of these alloys and where high speed of welding is very important [1]. Many studies are prepared about joining of these fine grained alloys but this study focuses the effects of flux types on micro-structural and mechanical developments of weldments in submerged arc welding. Hence, the transverse strength, micro-hardness surveys and micro-structural inspections of welded samples are thoroughly investigated.

# 2. Materials and Methods

S235 steel alloy couples of 15 mm in thicknesses and 70x200 mm in dimensions are prepared for joining with submerged arc welding. The elemental spectral analysis results of S235 steel by AMETEK Spectromax Argon Optical Emission Spectrometer are given in Table 1. The samples chemical composition is consistent with the specification [2].



Submerged arc welding is applied with totally 7 individual passes. After the first (root) pass has completed, the temperature control for sub-pass is maintained as all of the last 6 passes are made also by waiting until the temperature of the previous pass decreases but not below to  $100^{\circ}$ C. The temperature control is made by temperature chalk pencil that is capable of measuring approximately  $100^{\circ}$ C in color contrast.

The welded samples are shown in Figure 1.



Figure 1. Welded samples

Samples surfaces are all grinded before the last root pass (indicated by 7 in Figure 1) from their back sides.

After welding operation samples are cut for micro-vickers hardness testing [3], micro-structural inspections [4] and transverse tensile tests [5].

Submerged arc welding parameters are listed in Table 2.

rechnology


**-	Passes							
*Parameters are applied at the same conditions for both of the welding fluxes	1 <sup>st</sup> (root) pass	2 <sup>nd</sup> pass	3 <sup>rd</sup> pass	4 <sup>rd</sup> pass	5 <sup>th</sup> pass	6 <sup>th</sup> (final) pass	7 <sup>th</sup> (Back side- root pass)	
Current AC (Amperes)	350	400	470	470	600	520	450	
Welding speed (mm/s)	50	50	45	45	40	45	50	
Welding wire	TS EN ISO 14171-A; S2 (Ø4mm) [6]							
Flux types	1. TS Basicity Basicity	EN ISO :1.4) <b>2</b> :0.7) [7]	14174; . SA AF	SA AB 8 1 77	1 68 AC (R	AC H5 Sutile-Low	(Basic- v basic,	

 Table 2. Welding parameters.

The chemical compositions of welding wire from the manufacturer analysis are given in Table 3.

Table 3.					
Welding wire (S2)	С	Si	Mn		
	0.12	0.10	1.0		

Microstructural investigations of welded samples were carried out by Leica Brand optical metallurgical microscope after etching samples with 3% nitric acid solution. Base metal, weld metals and heat affected zones are all investigated.

The micro-vickers hardness test is applied to base metals and also with three distinct places on weld metals and heat affected zones separately according to the EN ISO 9015-2 standard [3] by 0.3 kg loading for 15 seconds at 22°C constant laboratory temperature. Transverse tensile tests of 3 samples are made per each welding condition according to AWS B4.0 [5]. standard as given in Figure 2.



Figure 2. Transverse tensile test samples according to AWS B4.0 standard.

Technology

aterials and Engineering





# 3. Results and Discussion

# 3.1. Micro-structural investigations

Micro-structural examinations are applied on weld metals and both sides of heat affected zones.

# Micrographs of Base Metal

Microstructure of S235 fine grained steel alloy is given in Figure 3. The structure basically consists of ferrite and perlite. Darker phases are perlite and lighter are ferrite in Figure 3.



Figure 3. S235 base metal microstructure, nital 3%, (50X)

The alloying elements are intentionally kept in low amounts in S235 fine grained steel alloy in order not to be hardened by thermal processes mainly like welding as a result of hardening effects of fast cooling problems of welded parts. Hence, there should be no other harder phases like martensite or bainite be found in normal fusion welding conditions of these alloys below 15 mm in thicknesses.

But for such materials more than 15 mm in thicknesses in extremely fast cooling thermal welding conditions such as laser or electron beam welding processes it is possible to meet martensite or bainite in minor amounts within HAZ regions of these alloys weldments [8,9].

Neverthless, in unwelded base metal microstructure of this study there are no other phases noted.

# Micrographs of welded samples

The micrographs of samples joined by basic and rutile fluxes are investigated.

The weld metals microstructures of the sample welded with basic and rutile fluxes are given in Figure 4.



**Figure 4.** Weld metal microstructures of samples joined by (**a**) basic (**b**) rutile welding fluxes. (100X)

Basic flux maintained the ferritic-perlitic microstructure more globular while rutile flux transformed it into needle like view. Perlitic-ferritic structure is observed in both weld metals of samples.

Darker phases are perlite and the lighter phases are ferrite in both microstructures.

# Microstructures of heat effected zones on welded samples

Microstructures of heat affected zones are given in Figure 5.



Figure 5. HAZ microstructures of samples joined by (a) basic (b) rutile welding fluxes. (100X)

Samples welded with rutile and basic flux exhibited view of the ferritic-perlitic microstructure but rutile flux displayed needle like view while basic flux ensured it more globular in heat affected zones. Perlitic-ferritic structure is observed in both heat affected zones of welded samples.

Darker phases are perlite and the lighter phases are ferrite in both samples microstructures.



# 3.2. Micro-hardness surveys

Micro-hardness test results on base metal and weld metal centerline and heat affected zones of left right sides in  $HV_{0.3}$  values are given in Table 4.

<b>Table 4.</b> Micro-hardness test results $(HV_{0.3})$													
Samples	Base Metal	Н	$\mathrm{AZ}_{1}$ (left	side)	Average Values	We	eld Me	tal	Average Values	HA	$\mathbb{Z}_2$ (right	t side)	Average values
Samples welded by basic flux	119	139	139	141	140	140	141	173	151	141	141	142	141
Samples welded by rutile flux	11)	127	127	133	129	142	143	167	151	136	145	145	142

According to micro-vickers hardness test results all of the regions in weldments have qualified. Fine grained low carbon alloyed S235 steels have to exhibit micro-hardness values below 350HV according to IIW specifications [10, 11].

The hardness results of weld metals of both samples are a few greater than heat affected zones. That's most probably because of welding wire carbon content supported the total carbon amounts in weld metal regions. Hence, the hardenability of weld metal region is preserved as compared to heat affected zones under cooling conditions of welding operations.

### **3.3. Transverse Tensile tests**

All of the tensile test samples are split apart from their close HAZ regions.

Transverse tensile test results are given in Table 5.

Table 5	Table 5. Transverse tensile test results							
Samples	Ten	sile Stre (MPa)	Mean Values					
Samples welded by basic flux	425	430	445	433				
Samples welded by rutile flux	430	430	430	430				

According to transverse tensile tests all of the samples are qualified. These steels base metals typically exhibit approximately 360MPa to 510MPa of tensile strength values within 3 up to 100 mm thicknesses [2].

Besides, there are no major tensile strength differences between the samples that welded by basic and rutile fluxes.



# 4. Conclusions

Basic and rutile flux submerged arc welded samples are both qualified in micro vickers hardness surveys and transverse tensile tests.

The International Conference

aterials and Engineering Technology

Rutile flux transformed the ferritic-perlitic microstructure into needle like while basic flux provided it globular.

All of the samples have exhibited ferritic-perlitic microstructure besides no other phases observed because of the low amounts of carbon and alloying elements in S235 steel and samples were also controlled during welding cooling conditions.

Basic or rutile welding fluxes both can be selected in submerged arc welding of S235 fine grained structural steel alloys.

Neverthless, whether the globular microstructure is desired in case of toughness considerations in applications, basic flux characterized submerged arc welding should be a well choice in joining of S235 alloys.

Acknowledgments: Authors express their respects to Firat Company and MKEK Armament Corporation and Laboratories staff for their precious experimental supports.

#### **References:**

- 1. ASM Metals Handbook, Welding. Brazing and Soldering **1993**, Vol 6, p1059.
- 2. TS EN 10025-2, Hot-rolled Products of structural steels, **2006**, Part 2.
- 3. EN ISO 9015-2, Destructive tests on welds in metallic materials, Hardness testing, Part 2, **2016**, Micro-hardness testing of welded joints.
- 4. ASM Metals Handbook Vol 9, Metallography and Microstructures, 2004, ASM International.
- 5. AWS B4.0: 2016, Standard test methods for mechanical testing of welds.
- 6. TS EN ISO 14171, **2016**, Welding consumables, Solid wire electrodes, tubular cored electrodes, and electrode/flux combinations for submerged arc welding of non alloy and fine grain steels.
- 7. TS EN ISO 14174, **2012**, Welding consumables, fluxes for submerged arc welding and electroslag welding.
- 8. EWF, IIW, Welding Engineering Course Notes 2005, GSI SLV München, Germany.
- 9. Kou S. Welding Metallurgy, 2<sup>nd</sup> Edition, **2003**, New York, John Wiley and Sons. p232-238.
- 10. Reeve, L., **1939**, "Metallurgy of ferrous welding", Note on Cambridge Welding Conference, Transactions of the Institute of Welding, Vol. II p7-18.
- 11. Dearden, J. and O'Neill, H., **1940**, "A guide to the selection and welding of low alloy strucutural steels", Transactions of the Institute of Welding, Vol III, p203-214.



# ÖZKAN ÖZBEK<sup>\*1</sup>, ÖMER YAVUZ BOZKURT<sup>2</sup>, AHMET ERKLİĞ<sup>3</sup>

<sup>1,2,3</sup> Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY

#### Abstract

One of the major drawbacks of fiber reinforced composite laminates is the poor impact damage resistance. Several effective techniques to improve impact damage resistance have been proposed in the literature, among which, fiber hybridization technique has been taken considerable investigation. However, little attention has, as yet, been received for the improvement of impact behavior of basalt fiber reinforced composite laminates by fiber hybridization. The objective of this study is to assess the effect of glass fiber hybridization on Charpy impact behavior of basalt fiber reinforced composite laminates. For this purpose, a series of Charpy impact tests have been performed on composite laminates made of basalt and glass fiber reinforced epoxy resin matrix in five different stacking sequences. Hybrid composite laminates have been fabricated using vacuum assisted resin transfer molding method. The test results show that the absorbed impact energy and impact strength are significantly influenced by the hybridization.

Keyword: Basalt fiber, Glass fiber, Hybridization, Charpy, Impact energy

#### **1. Introduction**

In recent years, the desire to use of polymeric composite materials in scientific studies has been gaining a great deal of attention due to their superior mechanical characteristics such as higher specific strength and stiffness, longer service life and better damping than classical engineering materials. Today, they are used in a wide range of engineering applications ranging from daily life tools to space industry. Even though they are so popular, some deficiencies such as high material costs, very expensive repairs and maintenance have been encountered in the usage of polymer based composites. Furthermore, one of the major drawbacks of fiber reinforced composite laminates is the poor impact damage resistance. Scientists have been developed several effective techniques like nanoparticle inclusion in resin, fiber hybridization, and different design parameters [1, 2] to improve impact characteristics of composite laminates. Amongst these ways, fiber hybridization method taking into account of change in behaviors of reinforcement component of laminates has been considered as an attractive way.

Several studies devoted to low velocity impact properties of composite laminates are present in the literature [3-6]. Najafi [7] examined the Charpy impact behaviors of hybrid composite laminates for various weight ratio of basalt and carbon fibers as 1:0, 0.83:0.17, 0.68:0.32, 0.61:0.39, 0.34:0.66 and 0:1, respectively. Maximum energy absorption for hybrid configuration as 219 kJ/m2 was found from 0.83:0.17 weight ratio of fabrics. Bozkurt et al. [8] investigated the effects of hybridization on the basalt/aramid fiber reinforced composite laminates.

When the basalt fiber/matrix layer was at the impacted surface, the hybrid laminates exhibited a lower impact energy due to the restriction in deformation of aramid layers. Ramesh et al. [9] conducted on a hybridization process of sisal fiber, jute fiber and glass fiber. The results indicated that the incorporation of sisal-jute fiber with GFRP can improve the properties and used as an alternate material for glass fiber reinforced polymer composites.

he International Conference

The objective of this study is to assess the effect of glass fiber hybridization on impact behavior of basalt fiber reinforced composite laminates. To this aim, low velocity impact experiments of composite laminates have been carried out on a Charpy impact tester. The energy absorption capability and impact strength characteristics have been determined by considering composite laminates made of basalt and glass fiber reinforced epoxy resin matrix in five different stacking sequences.

#### 2. Materials and Methods

#### 2.1 Sample Fabrication

Basalt fabric with 300 g/m<sup>2</sup> areal density, is an eco-friendly obtained from molten basalt rocks in volcanic regions, was procured from Tila Kompozit company, Turkey. S-glass fiber reinforcement with an areal density of 200 g/m<sup>2</sup>, epoxy resin (MOMENTIVE MGS L285) and hardener (MOMENTIVE MGS H285) was supplied from Dost Kimya company, Turkey. The matrix component of laminates consist of resin and hardener mixture with a stoichiometric weight ratio of 100:40, respectively. The thickness and naming informations of the non-hybrid laminates (G<sub>6</sub> and B<sub>6</sub>) and hybrid laminates (G<sub>5</sub>B<sub>1</sub>, G<sub>4</sub>B<sub>2</sub>, G<sub>3</sub>B<sub>3</sub>, G<sub>2</sub>B<sub>4</sub> and G<sub>1</sub>B<sub>5</sub>) were given in Table 1.

Sample	Naming	Thickness, mm
(G <sub>6</sub> ) <sub>S</sub>	GFRP	2.45
$(G_5B_1)_S$	H1	2.23
$(G_4B_2)_S$	H2	2.21
$(G_3B_3)_S$	H3	2.12
$(G_2B_4)_S$	H4	2.12
$(G_1B_5)_S$	H5	2.09
$(\mathbf{B}_6)_{\mathbf{S}}$	BFRP	2.05

**Table 1.** The naming and thickness information of the composite laminates.

All laminates consist of 12 number of fabric layers with  $(0^{\circ}/90^{\circ})$  fiber orientations in each one. Two non-hybrid fabric materials (basalt/epoxy and glass/epoxy) were hybridized at five different stacking sequences, as shown in Fig. 1, to examine effectively of hybridization impression. The stacking sequences of laminates were started with basalt fiber/epoxy laminate and were ended with glass fiber/epoxy laminate, by the replacement of inner 2, 4, 6, 8, 10, and 12 basalt fabric layers.



Figure 1. Stacking sequences of glass and basalt fabric layers.

Technology

aterials and Engineering



The laminates having 250 mm x 300 mm dimensions were fabricated by vacuum assisted resin transfer molding (VARTM) technique as seen in Fig. 2. The fabrics were firstly put on the process table. Then, necessary preparations such as application of peel ply, resin distribution film were performed to provide closed environment. Prepared resin was impregnated from one end of the system. After whole laminate was wet, the closed system was exposed to 700 mmHg vacuum process for 8 hours. Lastly, curing procedure was conducted at room temperature. After fabrications, CNC router was used to cut Charpy samples with required dimensions.



Figure 2. Schematic illustration of vacuum assisted resin transfer molding [8].

# **2.2 Charpy Impact Test**

Charpy impact experiments in accordance with ISO 179/92 standard [10] were performed to investigate the effects of fiber hybridization on low velocity impact characteristics, in terms of energy absorption and impact toughness, of the composite laminates. A Köger 3/70 Charpy test machine with an energy measuring capacity of 15.0 J, shown in Fig. 3, were employed for all experiments. The samples were prepared as notched and unnotched types for the edgewise and flatwise impact loadings, respectively.



Figure 3. Köger 3/70 Charpy impact tester.

Impact energy, E was directly measured from the differences of potential energies of pendulum before,  $E_a$  and after,  $E_b$  impact event as seen in Fig. 4.

$$E = \tilde{E}_a - E_b \tag{1}$$

Impact toughness, a<sub>cu</sub> which is the absorbed energy per unit area, was calculated from Eq. (2):

$$c_u = E/(bh)$$
 (2)

where b and h are the thickness and width of the samples, respectively.



Figure 4. Schematic illustration of Charpy impact test.

At least, five number of samples for each configuration were tested to ensure experimental reliability. The dimensions of Charpy samples for flatwise and edgewise loadings, settlements, were presented in Fig. 5.



Figure 5. The dimensions of Charpy samples

# 3. Results and Discussion

The absorbed energy and impact toughness graphs of the unnotched samples subjected to flatwise impact loading were presented in Fig. 6. Non-hybrid basalt fiber reinforced composite laminates with 2.41 J and 4.26 kJ/m<sup>2</sup> exhibited the best energy absorption capability and impact toughness, respectively. In hybrid samples, maximum value of energy absorption and impact strength as 2.36 J and 4.09 kJ/m<sup>2</sup> was obtained from H1 ((G<sub>5</sub>B<sub>1</sub>)<sub>s</sub>) configuration. There is a decreasing trend of the results of hybrid samples when increase of number of basalt layers inside the laminates. This is attributed to weak interfacial adhesion between basalt and glass layers in composite laminate samples.





Figure 6. Impact responses of unnotched samples subjected to flatwise loading; a) impact energy, b) impact strength

Figure 7 presents the impact energy and impact toughness characteristics of unnotched composite laminates subjected to edgewise impact loading. Negative effects as deteriorations were detected in hybrid configurations, but the trend was almost same with the notched samples. The worse response of hybrid samples compared to non-hybrid ones can be explained weak interfacial adhesion between glass and basalt fabrics. This situation were increasingly seen when basalt fabrics were spreading from inner layers to outer of the laminates. The maximum values in terms of energy absorption and impact strength were obtained from non-hybrid basalt fiber reinforced composite laminates with 3.13 J and 27.71 kJ/m2. H1 configuration in hybrid laminates showed the 23.77% and 18.9% better results than H5 for the energy and strength, respectively.



Figure 7. Impact responses of notched samples subjected to edgewise loading; a) impact energy, b) impact strength



The damage shapes and failure modes of each configuration were given in Figure 8. The combination of matrix cracking, delamination and fiber breakage were detected on all impacted samples. Basalt fiber reinforced laminate showed the fiber pull out due to higher energy absorption resulted with severe impact. The appearance of hybrid samples after impact showed the easy destruction takes place on samples since weaker bounds between layers.



Figure 8. Damage shapes and failure modes of impacted samples

#### 4. Conclusions

In this study, effects of glass fiber hybridization on the low velocity impact behaviors of basalt fiber reinforced composite laminates were investigated. Prepared samples with notched and unnotched structures for flatwise and edgewise loadings were impacted on Charpy test machine. Deteriorations in hybrid samples were detected since weak interfacial adhesion between layers caused from ability to fiber debonding.

Non-hybrid basalt fiber reinforced composite laminates showed the best impact energy and impact strength characteristics not only for notched configurations but also for unnotched ones. For hybrid samples, H1 or  $(G_5B_1)_s$  stacking sequences had the better characteristics than other hybrid ones. The combination of matrix cracking, delamination and fiber breakage were seen as failure modes in mostly. In conclusion, hybridization of basalt and glass fibers was not improved the composite laminates, and so not recommended for related applications.

The International Conference of Materials and Engineering Technology

#### References

- 1. Jarukumjorn, K, & Suppakarn, N, Effect of glass fiber hybridization on properties of sisal fiber–polypropylene composites, Composites Part B: Engineering, **2009**, 40(7), 623-627.
- 2. Flynn, J, Amiri, A, & Ulven, C, Hybridized carbon and flax fiber composites for tailored performance, Materials & Design, **2016**, 102, 21-29.
- 3. Caminero, MA, Rodríguez, GP, & Muñoz, V, Effect of stacking sequence on Charpy impact and flexural damage behavior of composite laminates, Composite Structures, **2016**, 136, 345-357.
- 4. Ghasemnejad, H, Furquan, ASM, & Mason, PJ, Charpy impact damage behaviour of single and multi-delaminated hybrid composite beam structures, Materials & Design, **2010**, 31(8), 3653-3660.
- 5. Tarpani, JR, Maluf, O, & Gatti, MCA, Charpy impact toughness of conventional and advanced composite laminates for aircraft construction, Materials Research, **2009**, 12(4), 395-403.
- 6. Perry, JL, & Adams, DF, Charpy impact experiments on graphite/epoxy hybrid composites, Composites, **1975**, 6(4), 166-172.
- Najafi, M, Khalili, SMR, & Eslami-Farsani, R, Hybridization effect of basalt and carbon fibers on impact and flexural properties of phenolic composites, Iranian Polymer Journal, 2014, 23(10), 767-773.
- 8. Bozkurt, ÖY, Erkliğ, A, & Bulut, M, Hybridization effects on charpy impact behavior of basalt/aramid fiber reinforced hybrid composite laminates, Polymer Composites, **2018**, 39(2), 467-475.
- 9. Ramesh, M, Palanikumar, K, & Reddy, KH, Mechanical property evaluation of sisal-juteglass fiber reinforced polyester composites, Composites Part B: Engineering, **2013**, 48, 1-9.
- ISO, I, 179–1, Plastics—Determination of Charpy Impact Properties, Part 1: Non-Instrumented Impact Test, International Organization for Standardization: Geneva, Switzerland, 2010.



# MECHANICAL PROPERTIES OF GRAPHENE NANO-PLATES REINFORCED LDPE COMPOSITES

# AHMET ERKLİĞ<sup>1</sup>, TUĞÇE GÜL POLAT<sup>1</sup>

<sup>1</sup>Gaziantep University Faculty of Mechanical Engineering Department, Gaziantep, TURKEY.

#### Abstract

In this study, the effects of graphene on the mechanical properties of low density polyethylene (LDPE) film used in packaging industry are investigated. Graphene was added to the LDPE F2-12 raw material in 0.1%, 5% and 10% by weight. The mixture prepared by twin screw extrusion machine and specimens filmed in blown line. Film thickness was obtained as 17, 19, 25 and  $27\mu$  due to the effect of graphene addition. Thickness, heat seal, tear, coefficient of friction(COF) and tensile strength tests of the produced films were carried out in accordance with ASTM standards. When the graphene additive test results were examined in LDPE film, a 58.82% increase in thickness was observed. It has been found that the mechanical properties of graphene added LDPE film generally decrease and the film is weaker than the non-graphene film.

Keyword: Nanocomposites, LDPE, Graphene, Tensile Strength, Blown Film.

#### **1. Introduction**

Control of graphene properties can be very helpful in creating graphene-based systems with magnetic and superconducting properties. Although it is still not fully understood, it is certain that graphene will have a more effective place with scientific and technological tools in accordance with the researches. Understanding the properties of this material can improve in many areas [1].

Graphene is a two dimensional nanomaterial, which has superior properties and a layer of covalently bound carbon atoms arranged in a perfect honeycomb (hexagonal) lattice. The carbon-carbon bond length of graphene is approximately 0.142 nm. The electrons on the structure of graphene acts as massless particles and therefore, it causes unique properties like quantum hall effect [2]. In low-density polyethylene, branching is at the maximum level, and branching is long and short, affecting polymer properties. These four types of polyethylene are the basic polymeric materials of industry and have very different fields of use. General characteristics of polyethylene are as follows:

- Strong
- Resistant to acids, bases and solvent
- Dielectric properties are superior
- Resistant to environmental conditions
- Easy to process.[3]

In recent years, researchers have focused their attention on polymers reinforced with nano-sized materials with an alternative to conventional filled polymers. The number of carbon-based nano reinforcement materials is increasing; they range from carbon nanotubes to carbon nanofibers and fullerenes to graphene. The superior properties of graphene compared to polymers are also reflected in polymer / graphene nanocomposites. Polymer / graphene nanocomposites have superior mechanical, thermal, gas barrier electrical and flame-retardant properties compared to neat polymer. It has also been reported that improved mechanical and electrical properties of graphene-based polymer nanocomposites are better than clay of other carbon filler-based polymer nanocomposites. For that reason, effects of graphene nanoplatelets on properties of PE material is investigated in this study.



Lee et al. [4] investigated morphology of clay dispersion on LDPE/clay nanocomposite. Nanocomposites were produced by melt blending with and without a malleated polyethylene (PE-g-MAn) as the coupling agent. As a result of their study, 0.1 wt% of clay addition gave the best performance. Mechanical and gas barrier properties of LDPE/clay nanocomposite film were examined by Arunvisut et al. [5] LDPE and clay nanoparticles were melt-mixed with twin screw extruder, after that material was re-extruded through blownfilm die. 1, 3, 5 and 7 wt.% of nano clay was used. Tensile modulus and tensile strength at yield of blown film LDPE/clay nanocomposites improved in both TD and MD tests when compared between 7 wt % PE/clay nanocomposites and neat PE. Gas permeability capacity was also increased with increase of clay content. However, elongation at yield decreased when increased in clay loading. Similar study was carried out by Zhon et al. [6]. They found that mechanical properties increased up to 5 wt.% of clay nanoparticle addition. Durmus et al. [7] investigated thermal, mechanical and gas barrier properties of the LDPE / clay nanocomposites. The added of only oxidized polyethylene compatibilizer to LDPE reduced permeability by 44%. The physical performance of the nanocomposites was not only affected by the clay dispersion. Electrical properties of LDPE/ZnO nanocomposites were examined by Hong et al. [8, 9]. Melt mixing method was used to get homogeneous dispersion of ZnO particles on PE material. They investigated the effect of particle size on dielectric properties as conventional particles (submicrons) and nanoparticles. As the decrease of particle size, dielectric properties of LDPE material were increased. Xia et al. [10] examined the structure and thermal characteristics of copper/low-density-polyethylene (Cu/LDPE) nanocomposites produced using a meltblending technique in a single-screw extruder. Structure and thermal characteristics of Cu/LDPE composites were characterized by XRD, SEM, EDS, DSC and TGA. The thermal stability of the Cu/LDPE nanocomposites decreased after 2 wt. % of copper nanoparticles. Joker et al. [11] studied on melt production of low density polyethylene (LDPE)-Silver nanocomposite film. They compared mechanical properties of LDPE/silver nanocomposites with silver free LDPE films. As a result of the study there were not significantly difference between results. Xiao et al. [12] examined the rheological and mechanical properties of LDPE composites reinforced by MWNT. Young's modulus increased by 85% and tensile strength by 46% when 10% MWNT was added to low density polyethylene.

According to known literature, there is no any study mechanical properties of LDPE/graphene nanocomposite. For that reason, the effect of graphene nano particles on the LDPE thermoplastic films is investigated in this study. The ratios of graphene nanoparticles were used as 0.1, 5 and 10 wt. %. The graphene and granular LDPE material were mixed and extruded by twin screw extruder. After that taken film nanocomposites were obtained by blow film method. Tensile strength, thickness, heat performance, tensile stress, tear, COF tests were carried out in accordance with ASTM standards.

#### 2. Materials and Methods

In this work three different nanographene ratios (0.1, 5 and 10 wt.%) have been added to the LDPE. The nanocomposites were prepared by the double screw extrusion machine and filmed in the blown line. The thickness, heat seal, tear, coefficient of friction, tensile tests of the produced films were carried out in accordance with ASTM standards. LDPE(F2-12) material was supplied from Petkim Petrokimya Holding Co, Istanbul, Turkey. Nano graphene material was supplied from Graphene Chemical Industries Co., Ankara, Turkey. LDPE F2-12 type material properties are given in Table 1 and nanographene material properties are given in Table 2.





# Table 1. Properties of LDPE

Properties	Typical Value	Unit
<b>Resin Properties</b>		
Melt Flow Rate (190°C/2.16 kg)	2.5	g/10 min
Density, 23°C	0.920	g/cm <sup>3</sup>
Melting Point (DSC)	110	°C
<b>Film Properties</b>		
Tensile Strength at Yield, MD	11	MPa
Tensile Strength at Yield, TD	10	MPa
Tensile Strength at Break, MD	23	MPa
Tensile Strength at Break, TD	17	MPa
Elongation at Break, MD	>200	%
Elongation at Break, TD	>500	%
Tear Strength, MD	330	cN
Tear Strength, TD	240	cN

Table 2. Properties of Graphene Nano Platelets

Density	$50 \text{ kg/m}^3$
<b>Fibre thickness</b>	5-8 nm
Surface area	120-150 m <sup>2</sup> /g
Diameter	5 μm

The LabThink thickness tester (figure 1) was used to determine min - max and average thicknesses by measuring the thickness of the film at exact intervals. Zwick tensile test machine (figure 2) was used to find tensile properties of the nanofilms. Tesile specimens were cut in accordance with ASTM D882 standard.



Figure 1. Labthink Thickness Tester



Figure 2. Zwick Tensile Test Machine



CLASSIC 513 Gradient heat seal tester (figure 3) is professionally designed for the determination of heat seal ability of plastic films pressure and 5 different temperatures. In order to find out the degree of thermal insulation of the LDPE product, the film is collected in two layers with a hot jaw and then allowed to cool, the film is cooled. Tests were carried out in accordance with ASTM F88 standard.



Figure 3. Heat Seal Tester

The LabThink COF tester (as shown in Figure 4) was used for measuring the lubricity of the film. It is also measuring the coefficient of friction of the film. ASTM D1894 standard was used. The film cut to a certain size ( $10 \times 10 \text{ mm}$ ) was placed on a 200gram apparatus and the table was placed on a sliding device.



Figure 4. LabThink COF Tester

The LabThink tear tester (figure 5) use the elmandorf tear method, notch is cut at certain dimensions and tearing is provided from that point, depending on the pendulum angle.



Figure 5. LabThink Tear Tester

#### **3. Results and Discussion**

The images of the films having 100% F2-12 and 0.1, 5 and 10 wt. % graphene ratios produced by the blown method are shown in Figures 5 to 8. When the graphene ratio increased, color of the films is become darker. Composition and names of nanocomposites are tabulated in Table 3 and named as F1, F2, F3 and F4.





Table 3. Comp	position and	Names of	Nanocomposites
---------------	--------------	----------	----------------

Name	<b>F</b> 1	<b>F2</b>	<b>F3</b>	<b>F4</b>
Mixing Ratios	%100 F2-12	90% F2-12 + 9.9% CaCO <sub>3</sub> + 0.1% Graphene	%95 F2-12 + %5 Graphene	%90 F2-12 + %10 Graphene



Figure 5. F1 Specimen



Figure 6. F2 Specimen



Figure 7. F3 Specimen



Figure 8. F4 Specimen

# **3.1. Measurement of Thickness**

Pure LDPE and LDPE added in different proportions of graphene thickness test results given in the Table 4. The thickness results of the 17, 19, 25 and 27 microns and an increase of 12%, 47% and 59% in thickness is observed for F2, F3 and F4 specimens, respectively. According to the thickness results, when the graphene ratio increased, the film hardened and tried to be formed under higher temperature. Therefore, the thickness increases as the graphene ratio increases due to drafting difficulty.

Name	<b>F</b> 1	F2	F3	F4
Mixing Ratios	%100 F2-12	90% F2-12 + 9.9% CaCO <sub>3</sub> + 0.1% Graphene	%95 F2-12 + %5 Graphene	%90 F2-12 + %10 Graphene
Thickness (µm)	17	19	25	27

Table 4. Thickness	Values	of Produced	Samples
--------------------	--------	-------------	---------



#### **3.2.** Tensile Test Results

Tensile test results of LDPE and LDPE with different graphene ratio are given in the Table 4 as results in machine direction (MD) and transverse direction (TD). Figure 9 gives force elongation diagram of the samples. All tensile results were compared with pure LPDE film results. Tensile strength in machine direction of LDPE/graphene films were decreased with 14.41%, 26.97% and, 31.7% ratios for F2, F3 and F4 specimens, respectively. Tensile strength in transverse direction of LDPE/graphene films were decreased with %23.02, 56.04% and, 59.8% ratios for F2, F3 and F4 specimens, respectively. The percent elongation in the machine direction of the specimens are 251, 219, 180 and 172. A decrease of 12.74%, 28.28% and 31.47% in percent elongation is observed in machine direction for F2, F3 and F4 specimens compared with pure LDPE film. The percent elongation in the transverse direction of the specimens are 569, 504, 313 and 178. A decrease of 14.42%, 44.99% and 68.71% in percent elongation is observed in transverse direction for F2, F3 and F4 specimens are 569, 504, 313 and 178. A decrease of 14.42%, 44.99% and 68.71% in percent elongation is observed in transverse direction for F2, F3 and F4 specimens are 569, 504, 313 and 178. A decrease of 14.42%, 44.99% and 68.71% in percent elongation is observed in transverse direction for F2, F3 and F4 specimens compared with pure LDPE film. The low tensile test results in LDPE/graphene nanocomposites are thought to be due to the low graphene content or due to agglomeration of the polymer in the matrix or due to poor bonding of the polymer matrix to the graphene particles.

The International Conference

of Materials and Engineering Technology

Name					
		F1	F2	F3	F4
Thickness (µm)		17	19	25	27
Tensile Strength (N)	MD	12.55	12.15	12.52	14.22
	TD	7.22	6.35	4.65	5.70
Tensile Strength (Mpa)	MD	28.92	24.75	21.12	19.75
	TD	16.72	12.87	7.35	6.72
<b>Break Elongation (%)</b>	MD	251	219	180	172
	TD	569	504	313	178

 Table 5. Tensile Values of Produced Samples

MD: Machine Direction. TD: Transverse Direction

Broken samples are shown in figures 8-12. According to breaking samples, when the graphene ratio increased, sample elongations decreased and also contraction in transverse direction of broken points decreased due to increase of hardness and thickness of films.



Figure 9. F1,F2,F3,F4 Force-Elongation Diagram



UWE Bristol

Figure 9. F1 (a) MD Break Form (b)TD Break Form



**Figure 10.** F2 (a) MD Break Form (b)TD Break Form



Figure 11. F3 (a) MD Break Form (b)TD Break Form

Figure 12. F4 (a) MD Break Form (b)TD Break Form

# **3.3. Heat Seal Test Results**

LDPE and LDPE in different proportions of graphene heat seal test results are given the Table 6. Although the micron and graphene ratio changes, the heat seal test results do not change. Because graphene did not make chemical bond with properties that could change the heat adhesion.



Table 6. Heat Seal Values of Produced Samples					
Name		F1	F2	F3	F4
Thickness (µm)		17	19	25	27
Heat Seal Temperature(°C) (120kPa - 1s)	B/B	108 - 109	108 – 109	109	109

e International Conference of Materials and Engineering Technology

# **3.4. COF Test Results**

LDPE and LDPE in different proportions of graphene COF test results are given the Table 4.4. Friction force is not changed in specimen F3 and F4, because there is no binding between maleic anhydride and graphene nano particles. COF value for F2 specimen is lower than pure specimen because there was a  $CaCO_3$  in the formulation as a binder for LDPE and graphene.

Table 7. COF Values o	of Produced Samples
-----------------------	---------------------

Name		<b>F1</b>	F2	F3	<b>F4</b>
Thickness (µm)		17	19	25	27
Coefficient of Friction (Film/Metal)	B/M	0.20	0.11	0.16	0.16
	B/M	0.19	0.12	0.19	0.15

### 3.5. Tear Test Results

LDPE and LDPE in different proportions of graphene tear test results is given the Table 8. According to the machine direction tear results, there was a 15.82% increase in F2 specimen, 44.1% and 97.16% decrease in F3 and F4 specimens. For the transverse direction there was a 15.82%, 35.25% decrease in F2 and F3 specimens and 7.91% increase in F4 specimens. As the thickness and

graphene ratio increased, the tear test values decreased. This indicating that the nanoparticles did not distribute well in the matrix.

Table 8. Tear Test Values or	of Produced Samples
------------------------------	---------------------

Name		<b>F1</b>	F2	<b>F3</b>	<b>F4</b>
Thickness (µm)		17	19	25	27
Tearing Test	MD	214 - 244	217 – 311	70 - 186	0 - 13
(800 gi)	TD	126 - 152	159 - 163	60 - 120	107 - 194

# 3.6. Microscope Image (50X)

The microscope images of the films having is shown in Figures 13. According to images, when graphene content increases, agglomeration in graphene particles increases. Due to increase of thickness and need more heat to get a film, there was scratches on the surface of the films especially on F2 specimen. Also, its seen that, when the ratio of graphene particles increased, there were not homogeneously distributed inside the matrix.



The International Conference

Materials and Engineering Technology

F2



F4 Figure 13. Microscope images of the films

### 4. Conclusions

F1

**F3** 

Thickness, heat seal, COF, tear, tensile behavior of LDPE/graphene nanocomposites were investigated under the effects of nano particle addition. LDPE/graphene films were fabricated using twin screw extruder, blown film machine by adding various weight content of graphene nano particles. Tests were carried out according to ASTM standards. Based on the experimental results following conclusions can be summarized:

- Thickness of samples were increased with increase of graphene nanoparticles. Thickness of LDPE/graphene films were increased with an amount of 12%, 47% and 59% compared with pure LDPE film for F2, F3 and F4 specimens, respectively.
- Tensile strength and elongation decreased with the addition of graphene nanoparticles.
- Tensile strength in machine direction of LDPE/graphene films were decreased with 14.41%, 26.97% and, 31.7%, for F2, F3 and F4 specimens, respectively.
- Tensile strength in transverse direction of LDPE/graphene films were decreased with %23.02, 56.04% and, 59.8%, for F2, F3 and F4 specimens, respectively.
- The percent elongation in the machine direction of the specimens are 251, 219, 180 and 172. A decrease of 12.74%, 28.28% and 31.47% in percent elongation is observed in machine direction for F2, F3 and F4 specimens compared with pure LDPE film.
- The percent elongation in the transverse direction of the specimens are 569, 504, 313 and 178. A decrease of 14.42%, 44.99% and 68.71% in percent elongation is observed in transverse direction for F2, F3 and F4 specimens compared with pure LDPE film.
- The increase in graphene contribution did not affect the heat seal results.
- The increase in graphene contribution did not affect the COF results.
- According to the machine direction tear results, there is a 15.28% increase in F2 specimen, 44.1% and 97.16% decrease in F3 and F4 specimens. For the transverse direction there is 15.82%, 35.25% decrease in F2 and F3 specimens and 7.91% increase in F4 specimens.



1. Kozal, B. (2012). Karbon tabanlı petek örgülerin elektronik özellikleri. (Doktora Tezi) Ankara Üniversitesi Fen Bilimleri Enstitüsü.

The International Conference of Materials and Engineering Technology

- 2. Bedeloğlu, A. ve Taş, M., (2016). Grafen ve Grafen Üretim Yöntemleri. *Afyon Kocatepe Üniversitesi Fen Ve Mühendislik Bilimleri Dergisi*. **16(3)**, 544-554.
- 3. Mirik, M. (2010). Karbon nanotüp takviyeli yüksek yoğunluklu polietilen (YYPE) nanokompozit malzemelerin mekanik özelliklerinin araştırılması (Doktora Tezi) Selçuk Üniversitesi Fen Bilimleri Enstitüsü.
- Lee Y. H., Wang, K., H., Park, C. B. and Sain, M. (2007). Effects of clay dispersion on the foam morphology of LDPE/clay nanocomposites. Journal of Apllied Polymer Science, 103(4-15), 2129-2134
- 5. Arunvisut, S., Phummanee, S., ve Somwangthanaroj, A. (2007). Kilin, çekilmiş film LDPE / kil nanokompozitlerinin mekanik ve gaz bariyer özelliklerine etkisi. Uygulamalı Polimer Bilimi Dergisi, 106 (4), 2210-2217.
- 6. Zhong, Y., Janes, D., Zheng, Y., Hetzer, M., and De Kee, D. (2007). Mechanical and oxygen barrier properties of organoclay-polyethylene nanocomposite films. Polymer Engineering & Science, 47(7), 1101-1107.
- 7. Durmuş, A., Woo, M., Kaşgöz, A., Macosko, C. W., and Tsapatsis, M. (2007). Intercalated linear low density polyethylene (LLDPE)/clay nanocomposites prepared with oxidized polyethylene as a new type compatibilizer: structural, mechanical and barrier properties. European Polymer Journal, 43(9), 3737-3749.
- 8. Hong, J. I., Schadler, L. S., Siegel, R. W., and Mårtensson, E. (2003). Rescaled electrical properties of ZnO/low density polyethylene nanocomposites. Applied physics letters,82(12), 1956-1958.
- 9. Hong, J. I., Winberg, P., Schadler, L. S., and Siegel, R. W. (2005). Dielectric properties of zinc oxide/low density polyethylene nanocomposites. Materials Letters, 59(4), 473-476.
- 10. Xia, X., Cai, S. and Xie, C. (2006). Preparation, structure and thermal stability of Cu/LDPE nanocomposites. Materials Chemistry and Physics,95(1), 122-129.
- 11. Jokar, M., Rahman, R. A., Ibrahim, N. A., Abdullah, L. C., and Tan, C. P. (2012). Melt production and antimicrobial efficiency of low-density polyethylene (LDPE)-silver nanocomposite film. Food and bioprocess technology,5(2), 719-728.
- 12. Xiao, K. Q., Zhang, L. C. and Zarudi, I. (2007). Mechanical and rheological properties of carbon nanotube-reinforced polyethylene composites. Composites Science and Technology, 67(2), 177-182.

# AL CU MG MGO MWCNT KOMPOZİTLERİN FARKLI YÜKLER ALTINDA AŞINMA ÖZELLİKLERİNİN İNCELENMESİ

The International Conference

aterials and Engineering Technology

# HALİL İBRAHİM KURT \*1, ENGİN ERGÜL 2, MURAT ODUNCUĞLU 3, CAN ÇİVİ 4

<sup>1</sup> Gaziantep Üniversitesi, Mühendislik Fakültesi, Metalürji ve Malzeme Mühendisliği Bölümü, Gaziantep, TÜRKİYE.
 <sup>2</sup> Dokuz Eylül Üniversitesi, İzmir Meslek Yüksekokulu, Teknik Programlar Bölümü, İzmir, TÜRKİYE.
 <sup>3</sup> Yıldız Teknik Üniversitesi, Fen Fakültesi, Fizik Bölümü, İstanbul, TÜRKİYE.
 <sup>4</sup> Celal Bayar Üniversitesi, Mühendislik Fakültesi, Makine Mühendisliği Bölümü, Manisa, TÜRKİYE

# Özet

Metallerde aşınma mühendislik temelinde karşılaşılan en önemli mekanizmalardan biridir. Özellikle sürtünmenin olduğu yerlerde sürtünme dayanımı yüksek mühendislik malzemelerinin kullanılması önem arz etmektedir. Bu çalışma, MGO/MWCNT takviyeli AL 2024 kompozitlerin abrasif aşınma davranışını etkileyen başlıca tribolojik faktörlerin incelenmesini kapsamaktadır. Kompozit malzemeler yarı-katı karıştırma metoduyla üretilmiştir. MGO/MWCNT takviye edilmiş AL 2024 alaşımlı alüminyum matris kompozitlerin aşınma davranışı incelenmiştir. Farklı yükler altında aşınma özellikleri incelenmiş olup, kayma hızı ve kayma mesafesi sabit tutulmuştur. Aşınma testleri ball on disk tipi aşınma cihazında 2N ve 5N yük altında 500 m mesafede ağırlık kaybı göz önünde bulundurularak aşınma değerleri incelenmiştir.

#### Anahtar Kelimeler: Kompozit, Aşınma, Al 2024, Ball on disk, CNT.

#### 1. Giriş

Katı bir yüzeyin bir başka katı yüzey üzerinde, hem yapay hem de doğal hareketi birçok mekanizmanın işleyişi için temel olarak önemlidir. Yüzeylerin birbiri üzerinde temassız hareket etmediği veya yuvarlanmadığı mekanik sistemler nadirdir. Bu nedenle triboloji, çok geniş bir uygulamada önem kazanmaktadır. Tribolojinin ana konusunda yer alan yüzey etkileşimleri alanındaki ilerleme, makine mühendisleri, malzeme bilimcileri, fizikçiler ve kimyagerlerin ilgi alanına girmektedir. Canlı organizmaları ilgilendiren biyo-triboloji bağlamında, disiplin daha geniş alana yayılmakta ve tıp ve biyolojik bilimlerini içermektedir. Triboloji alanındaki gelişmeler, dünyadaki mühendislik dallarının birçoğuna katkı sağlayarak desteklemiştir. Ulaşım ve enerji üretiminden tıp mühendisliğine, gıda bilimi ve kozmetik ürünlerine kadar yüzeylerin birbirine geçtiği kelimenin en geniş anlamıyla modern teknolojinin tüm yönlerine temas etmektedir [1].

Yüzeyler birbirinin üzerinde hareket ettiğinde aşınma meydana gelmektedir. Genellikle bir veya her iki yüzeyde malzeme kaybına bağlı hasar görülmektedir. Bazen aşınma belirgin şekilde olamayacak kadar yavaş olup, bazı durumlarda aşırı hızlı da gerçekleşebilir. Küçük miktarda malzemenin veya parçanın aşınma yoluyla kayba uğraması sonucu, büyük ve karmaşık makinelerin tamamen bozulmasına ve kullanılamamasına neden olabilir [2].

Aşınma DIN (DIN 50320-1979) standardındaki ifadesiyle, malzemenin başka bir malzeme ile (katı, sıvı, gaz) teması ile oluşan mekanik etkiler sebebiyle yüzeyinden küçük parçacıkların ayrılması sonucu meydana gelerek ve istenmeyen yüzey deformasyonudur" [3]. Buradaki ifadeden yola çıkılarak, makina elemanlarının şekillerinde, yüzey kalitelerinde ve boyutlarında istenmeyen etkiler oluşturmaktadır. Makina elemanlarının yaşadığı bu değişimler makinanın işleyişini olumsuz yönde etkilemektedir.

Aşınma ile meydana gelen temas, aşınma mekanizmaları ile doğrudan bağlantılı olduğu için temas noktasındaki koşullar önem kazanmaktadır. Temas şiddeti, elastik veya plastik temasa göre aşınma mekanizmalarını oluşturmaktadır. Aşınma mekanizmalarının başında gelen en temel ve en önemli olanlar;

he International Conference of Materials and Engineering Technology

- 1. Abrasif Aşınma
- 2. Adhesif Aşınma
- 3. Yorulma Aşınması
- 4. Korozif Aşınma olarak ifade edilebilir.

Abrasiv Aşınma; Katı malzemeler ile aynı sertlikte veya daha sert katı malzemeler belli bir yük altında meydana gelir. Sertliği düşük olan bir malzeme, sertliği yüksek bir malzeme karşısında içerisinde bulunan daha sert partiküller sayesinde abrasif aşınmaya neden olabilir [4].

Adhesiv Aşınma; Yük altında kayma yüzeyleri katı faz kaynak şeklinde birbirlerine yapışıp ayrılarak malzeme kaybı oluşturmasıdır. Malzemeler arasındaki yapışma mesafesine bağlı olup sırasıyla, bir materyalin diğerine karşı ilişkisinin yanı sıra, oksitler veya yağlayıcılar gibi yüzey filmlerine de bağlıdır [4].

Yorulma aşınması; Malzemelerin maruz kaldığı tekrarlı yük döngüleri sonucunda kritik sayıda döngüden sonra, büyük çukurlar oluşturmaktadır. Bu çukurların oluşumuyla yüzeyin kırılmasına neden olacak olan yüzey altı veya yüzey çatlaklarının oluşumuna neden olabilir. Bu durum aynı zamanda oyuklaşma (pitting) olarak da bilinir. Bu kritik noktadan önce (yüzlerce, binlerce, hatta milyonlarca devir olabilir), aşınmanın başlangıcından itibaren kademeli bir bozulmaya neden olup, adhesif veya abrasif aşındırıcı mekanizmalarının benzemeyen ihmal edilebilir aşınma meydana gelir. Bu nedenle, aşınması ile aşınan malzeme miktarı kullanışlı bir parametre değildir. Burada önemli olanı, devir sayısı veya yorulma kırılması oluşmadan önceki zaman açısından faydalı ömürdür [3].

Korozif Aşınma; Korozif aşınma, malzemenin korozif bir etkiye maruz kaldığı bir ortamda meydana gelir. Havada en baskın korozif ortam oksijendir. Korozyonun kimyasal ürünleri (örneğin oksitler), yüzeyler üzerinde bir tabaka oluşturur, bu da korozyonu yavaşlatma veya hatta durdurma eğiliminde olur. Ancak bu yüzey ortadan kalkar ve korozif ortam devam ederse malzemenin temas yüzeyi zayıflayarak kırılmaya sebep olabilir. Kimyasal aşınma, madencilik, maden işleme, kimyasal işleme gibi birçok sektörde önemlidir [1].



# 2. Malzeme ve Metod



The International Conference

aterials and Engineering Technology

Şekil 1. Ball-on-Disk aşınma cihazı

Kuru kayma şartları altında, oda sıcaklığında, Aşınma testleri ball-on-disk tipi CSM Instruments marka cihazda yapılmıştır (Şekil 1). Deneylerde; 2 N ve 5 N yük ile 1,0 m/s kayma hızı kullanılmıştır. Kayma mesafesi olarak 500 m belirlenmiştir. Aşınma testlerinin amacı karıştırma döküm yöntemi ile üretilen metal matrisli kompozit malzemelerin içeriğine göre aşınma davranışının incelenmesidir.

# 3. Sonuç ve Tartışma

Kuru kayma şartları altında, oda sıcaklığında, Aşınma testleri; 2 N ve 5 N yük ile 1,0 m/s kayma hızı belirlenmiş, 500 m mesafede gerçekleştirilmiştir. Aşınma testleri sonucunda elde edilen ağırlık kaybı sonuçları Tablo 1'de gösterilmiştir.

AĞIRLIK KAYBI (g)		500 m		
MALZEME ÖZELLİKLERİ	YÜK	Ağırlık kaybı(g)	Ağırlık kaybı ( % )	
Al 2024	2.11	0,0039	-0,0617	
%50 CNT %50 MgO %1	2 N	0,0019	-0,0221	
Al 2024	5 N	0,0018	-0,0307	
%50 CNT %50 MgO %1	ЗN	0,0033	-0,0352	

Tablo 1. Aşınma testi sonucu ağırlık kaybı (veya yüzdesi) (g / %).

Tablo 1 incelendiğinde, Al 2024 ana metalin aşınma yükü arttığında ağırlık kaybının azaldığı ve Al2024 hibrid kompozitin aşınma yükü arttığında ağırlık kaybının arttığı görülmektedir. 2N yük altında ise hibrid etkisiyle ağırlık kaybı azalmaktadır. Bu azalma hibrid etkisiyle malzemenin aşınma davranışın arttığını göstermektedir [5]. 5N yük altında ise ağırlık kaybı matris malzemesinden hibrid kompozite göre artmaktadır. Bu durum beklenen bir durum olmayıp aşınma karekteristiğini azaltmaktadır. Bu malzemenin sertliğinde azalma ve/veya porozite içeriğinde artışın nedn olduğu düşünülmektedir. Aşınma testi sonrası ağırlık kaybının incelendiği benzer çalışmalar literatürde yer almakta ve ağırlık kaybı vasıtası ile aşınma davranışları incelenebilmektedir. Bu çalışmalarda nano boyuttaki partikül takviyelerinin aşınma davranışlarına etkileri incelenmiştir [6]. Gerçekleştirilen çalışmada ise partikül takviyesi aşınma davranışlarını olumsuz olarak eklemiştir [7].



ME

#### 4. Sonuçlar

Bu çalışmada karıştırma döküm yöntemiyel üreitlen alüminyum 2024 matrisli hibrid kompozitlerin aşınma davranışı incelenmiştir. Güçlendirici olarak nano MgO ve çoğul duvarlı CNT kullanılmış olup bu malzemeler ağırlıkça %50 oranında karıştırılmış ve ağırlıkça %1 oranında metal matrise ilave edilmistir. Kompozitlerin asınma davranışları lineer bir görüntü sergilemektedir.

#### Referanslar

- [1] B. Bhushan, «Principles and Applications of Tribology-Second Edition.,» New Delhi, İndia, John Wiley & Sons, Ltd., 2013, p. Vol. 10.
- [2] H. Ian ve P. Shipway, Tribology Friction and Wear of Engineering Materials, United Kingdom: Matthew Deans, 2017.
- [3] DIN 50520 Wear Terms Systems Analysis of Wear Processes Classification Of The Field of Wear, Normung: DIN Deutsohes Institut, 1979.
- [4] J. R. Davis, SURFACE ENGINEERING FOR CORROSION AND WEAR RESISTANCE. ASM International., United States of America, 2001.
- [5] N. Sak, M. Zeren ve R. Yamanoğlu, «Alümina Katkılı ve Nikel ile Alaşımlandırılmış Alüminyum Esaslı Kompozitlerde Özelliklerin Karakterizasyonu,» Kocaeli Üniversitesi Fen Bilimleri Dergisi, cilt 1, no. 1, pp. 1-7, 2018.
- [6] U. Bozan, E. Altuncu ve F. Üstel, «Nano Partikül Takviyeli Teflon Kaplamaların Üretilmesi ve Karakterizasyonu,» SAÜ. Fen Bil. Der., cilt 18, no. 1, pp. 21-30, 2014.
- [7] İ. Topcu, A. N. Güllüoğlu, M. K. Bilici ve H. Ö. Gülsoy, «Karbon nanotüp takviyeli Ti-6Al-4V/KNT kompozitlerin aşınma davranışlarının incelenmesi,» Gazi Üniversitesi Mühendislik-Mimarlık Fakültesi Dergisi, 2018.
- [8] F. Gül ve M. İlivan, «SiO2 Takviye Edilmiş Al Kompozitlerin Abrasiv Aşınma Davranışını Etkileyen Faktörlerin İstatistiksel Analizi,» %1 içinde 4th International Symposium on Innovative Technologies in Engineering and Science, Alanya, 2016.

# MCRALY İÇERİĞİNE SAHİP TERMAL SPREY KAPLAMALARIN AŞINMA DAYANIMI AMAÇLI OLARAK GAZ TÜRBİN MOTORLARINDA KULLANILMASI

The International Conference

aterials and Engineering Technology

# Mehmet KILIÇ<sup>1</sup>, Derviş ÖZKAN<sup>\*2</sup>, Abdullah Cahit KARAOĞLANLI<sup>3</sup>

<sup>1</sup> Bartın Üniversitesi, Mühendislik, Mimarlık ve Tasarım Fakültesi, Metalürji ve Malzeme Mühendisliği, TÜRKİYE.
<sup>2</sup> Bartın Üniversitesi, Mühendislik, Mimarlık ve Tasarım Fakültesi, Makine Mühendisliği, TÜRKİYE.
<sup>3</sup> Mühendislik, Mimarlık ve Tasarım Fakültesi, Metalürji ve Malzeme Mühendisliği, TÜRKİYE.

# Özet

Günümüzde gaz türbin motorlarındaki türbin bıçak ve kanatçıkları gibi komponentlerde yüksek sıcaklıklara bağlı olarak gelişen oksidasyon ve aşınma esaslı hasarları meydana gelmektedir. Bu tip hasarların önüne geçilebilmesi için gaz türbin motorlarında hali hazırda termal bariyer kaplama (TBC) sistemleri kullanılmaktadır. TBC'ler, gelişmiş türbinli motorların yanma odası gibi sıcak bölgelerindeki parçaları sıcak gazların olumsuz etkilerinden korumak amacıyla kullanılan ve iki ya da daha fazla katmandan oluşan koruyucu kaplama malzemeleridir. TBC sistemlerinde, altlık metalik malzeme üzerine etkiyen sıcaklığı düşürmek ve altlık ile üst tabaka arasındaki termal genleşme katsayısını azaltmak için MCrAIY metalik bağ kaplamalar kullanılmaktadır. MCrAIY metalik bağ kaplamalar, TBC sistemlerinde yüksek sıcaklığın beraberinde getirdiği olumsuz etkilerinin bertaraf edilmesinin yanında sistem verimliliğini arttırmak amacıyla da kritik bir role sahiptir. Yüksek sıcaklıklarda kullanılan birçok metalik malzeme atmosferik koşullar ile aralarındaki çeşitli etkileşimler neticesinde aşınarak hasara uğramaktadır. Bu sebepten dolayı, MCrAIY içeriğine sahip metalik ara yüzey kaplamaları, havacılık ve uzay endüstrisinin birçok uygulama alanında kullanım bulmaktadır. Bu çalışmada, MCrAIY metalik bağ kaplamalar, özellikleri ve servis koşullarında oluşan aşınma davranışları incelenerek, sunulmuştur.

Anahtar Kelimeler: MCrAlY, Termal bariyer kaplamalar (TBCs), Metalik bağ kaplama (BC), Aşınma, Tribolojik davranış.

#### 1. Giriş

Termal sprey kaplamalar, aşınma, korozyon, oksidasyon ve erozyon gibi hasar oluşum mekanizmalarını önlemek ve ısı yalıtımı sağlamak amacıyla farklı endüstriyel uygulama alanlarında yaygın bir şekilde kullanılmaktadır [1-2]. Termal sprey kaplama proseslerinde kaplama özellikleri ve performansı; altlık yüzey, kaplama malzemesi ve kaplama parametrelerine bağlı olduğundan dolayı Ar-Ge çalışmaları birçok uygulama alanı ve değişik kompozisyona sahip kaplama malzemeleri üzerine güncel olarak devam etmektedir. Bununla birlikte, optimum kaplama özellikleri, kritik kaplama parametreleri, alternatif kaplama prosesleri ve farklı üretim sınıfları, araştırmaların sürekli güncellenmesi ve geliştirilmesi süreçleri de tüm hızıyla güncelliğini korumaktadır. Termal sprey kaplama prosesleri kendi içerisinde, Plazma Sprey (PS), Atmosferik Plazma Sprey (APS), Vakum Plazma Sprey (VPS), Süpersonik Plazma Sprey (SAPS), Yüksek Hızlı-Oksi Alev Kaplama (HVOF) ve Detonasyon Tabancalı (D-Gun) Sprey ve Soğuk Gaz Dinamik Sprey (CGDS) kaplamalar gibi üretim açısından değişik özelliklere sahip farklı tip proseslere ayrılmaktadır [3].



Termal sprey kaplamalarda sistem bileşenlerinin servis koşullarındaki bütünlüğü açısından, kullanılan kaplamanın altlığa yapışma mukavemeti büyük önem arz etmektedir. Bu nedenle, TBC sistemlerinin kalitesi ve performansı da büyük ölçüde altlık ile kaplama arasındaki yapışmaya bağlıdır [4-5]. Termal sprey kaplama proseslerinde yüksek sıcaklık ve aşınma dayanımını artırmak amacıyla farklı birçok kaplama malzemesi kullanılmaktadır. Şekil 1'de kullanılan kaplama malzemelerinin genel gösterimi verilmektedir.



**Şekil 1.** Termal sprey uygulamalarında kullanım bulan ara yüzey ve üst yüzey kaplama malzemeleri [1-6].

Kaplamaların tribolojik davranışları başlıca uygulanan yük, sıcaklık, hız, tane boyutu, yoğunluk, çevresel koşullar gibi parametreleri içeren temas şartlarından etkilenmektedir. Kaplamaların aşınma ve sürtünme özelliklerinin belirlenmesinde deneysel çalışmalar yapılarak kaplama yüzeylerinin nasıl davranacağı farklı yük ve parametrelerde incelenerek görülebilmektedir [7-8]. Deneysel çalışma sonuçları kaplama kalınlığı, yüzey pürüzlülüğü, kayma hızı, yük, çevre şartları vb. parametrelere göre değişebilmektedir. Bu sayede istenilen hedef değerlere göre kaplama malzemesinin performansı değerlendirilerek, seçim yapılabilmektedir [7-8]. Bu çalışmada, MCrAIY metalik bağ kaplama içeriğine sahip termal sprey kaplamaları ve bu sistemlerin tribolojik özellikleri ayrıntılı olarak açıklanmıştır. Termal sprey kaplamaların tribolojik özelliklerini metalik bağ ile üst yüzey arasındaki yapışma, kaplama üretim prosesleri, yüzey özellikleri ve mikroyapısal özellikler direkt olarak etkilemektedir.

# 2. MCrAlY İçeriğine Sahip Metalik Bağ Kaplamalar

Son yıllarda gaz türbin üreticileri yüksek çalışma sıcaklıklarında verimliliği artırmanın yollarını aramaktadır [5]. İstenilen yüksek sıcaklık koşullarında oksidasyona dirençli MCrAlY (M=Ni, Co) kaplamaları, metalik altlık malzemeyi oksidasyondan korumak ve ayrıca aşınma direnci yüksek bir malzeme olarak farklı uygulamalarda kullanılabilmektedir.



MCrAlY metalik bağ kaplamada bulunan M harfi kobalt (Co) veya nikel (Ni) ya da bunların her ikisinin de yer aldığı bir kombinasyonunu içerir [6]. MCrAlY metalik bağ kaplamanın üzerine yapıldığı Ni esaslı alaşımlar, üstün mekanik özelleri ve yüksek sıcaklık sürünme dayanımları sebebiyle özellikle türbin motorlarının sıcak bölümlerinde, ısıl yorulma, çatlama gibi problemleri önlemek amacıyla kullanılmaktadır [9].

ials and Engineering Technology

MCrAlY metalik bağ kaplamalar genellikle iki fazlı bir yapı özelliği göstermektedir.  $\beta+\gamma$  ve  $\gamma$  fazı kaplamanın sünekliğini arttırarak ısıl yorulma direncini arttırır.  $\beta$ -NiAl fazı içerisindeki alüminyum yüksek sıcaklığa maruz kalmasıyla birlikte altlık malzemeye ve TGO içerisine doğru difüze olur.  $\beta$ -NiAl fazı içerisindeki alüminyumun yüksek sıcaklığa maruz kalmasıyla, üst kaplama yüzeyinden oksijenin ara yüzeye geçişi ile birlikte ısıl olarak büyüyen oksit tabakası (TGO) içerisine doğru difüze olur [10-11]. Şekil 2'de altlık malzeme, metalik bağ kaplama ve üst yüzeyden oksijenin ara yüzeye penetre olması ve Al'un difüzyonu ile birlikte oluşan TGO tabakası gösterilmektedir. İçerisindeki alüminyum miktarı azaldıkça  $\beta$  fazı çözülmeye başlar. Bu yüzden bu faz termal bariyer kaplamalarda önemli bir role sahiptir. Kaplamanın ömrü, faz içerisindeki alüminyum miktarının azalmasına göre belirlenebilir [11].





TGO tabakası, bağ kaplama ile üst kaplama arasında bulunan ince bir arayüzey yapısını ifade etmektedir. Yüksek sıcaklıklara bağlı olarak ara yüzeyde Al oksitlenmeye başlayacaktır. Alüminyumun oksijenle temas halinde olması Al<sub>2</sub>O<sub>3</sub> oluşumunun başlamasına sebep olacaktır. Alüminyumun azalmasıyla, oksijenin miktarında artış meydana gelecektir. Bu da araya yüzeyde oluşan TGO tabakasında kalınlaşmaya ve diğer zayıf mekanik özeliğe sahip diğer oksit yapılarının oluşumuna sebebiyet verecektir [1-12].





# 3. MCrAlY Metalik Bağ Kaplamaların Aşınma Davranışları

Yüksek sıcaklıklarda birçok metalin, altlık malzeme ve atmosferik koşullardaki etkileşimleri ile malzeme yüzeyi üzerinde sürtünmenin de etkisi ile aşınmalar meydana gelmektedir [13]. Bu tür aşınmalar, uçakların çöl koşulları etkisiyle kum tanelerinin türbin motorlarına girmesi ya da katı objelerin kaplama yüzeyinde hasar oluşturmasıyla ortaya çıkabilmektedir.

MCrAlY metalik bağ kaplamaların aşınma davranışlarının geliştirilmesi noktasında, yüksek sıcaklık sürtünme aşınması iyi olan diğer oksit yapılarının potansiyel takviye malzemesi olarak kullanılması alternatif bir çözümdür. HVOF yöntemi kullanarak üretilen kaplamalarda, Al<sub>2</sub>O<sub>3</sub>-B<sub>4</sub>C içeren NiCoCrAlY içerikli kaplamalar, oda sıcaklığından 800°C'ye kadar iyi bir aşınma direnci göstermiştir [14]. Şekil 3a'da NiCoCrAlY kaplamaların oda sıcaklığında yapılan aşınma testi sonrası oluşan hasar yapıları mikro-yapısal olarak verilmektedir. Öncelikle numunelerde ara yüzey pürüzlülüğündeki azalmalar olduğu ve buna bağlı olarak aşınma izlerinin ortaya çıktığı görülmektedir. Aşınma testinde numune üzerinde plastik deformasyona uğrayan noktalar gösterilmiştir.





Şekil 3. NiCoCrAlY bağ kaplamalarda a) oda sıcaklığında yapılan aşınma testi sonrası mikroyapısal değişim, b) yüksek sıcaklıklarda yapılan aşınma testi sonrası oluşan mikro-yapısal değişim [15].



aterials and Engineering Technology

Şekil 4a'da MCrAlY metalik bağ kaplamaların oda sıcaklığında yapılan aşınma testi sonrası oluşan hasar yapıları mikroyapısal olarak verilmektedir. Oda sıcaklığında aşınma testine tabi tutulan kaplamanın aşınma sonrasında öncelikle yüzeydeki pürüzlerinin temizlenerek kompakt bir yapının oluştuğu, sonrasında ise oluşan bu yapının bölgesel kırılmalara bağlı olarak yüzeyden ayrıldığı görülmüştür.



Şekil 4. a) Oda sıcaklığında MCrAlY metalik bağ kaplamaların aşınma davranışının SEM görüntüsü, b)Yüksek sıcaklıklar MCrAlY metalik bağ kaplamaların aşınma davranışının SEM görüntüsü



The International Conference of Materials and Engineering Technology Sekil 4b'de ise, MCrAlY içerikli metalik bağ kaplamaların 500°C sıcaklıkta yapılan asınma testi sonrası oluşan hasar yapısı gösterilmektedir. Yüksek sıcaklıklarda yapılan testlerde ise Şekil 4b'den de anlaşılacağı gibi, yüzeyde oluşan tabakanın çok daha kompakt bir şekilde meydana geldiği görülmektedir. Inconel 718 Nikel (Ni) içerikli süper alaşım altlık malzeme üzerine MCrAIY içerikli metalik bağ kaplamaların yapılması Şekil 4a ve b'den de anlaşılacağı üzere, aşınan yüzeylerde zamanla kırılan tabakanın dökülmesi sonucu meydana gelen plastik deformasyon çatlamalar ve gözenekler tespit edilmiştir. Sıcaklığın artmasına bağlı olarak, yüzeydeki hasar mekanizmasının, ekstrüzyon veya delaminasyona bağlı olarak yüzeyde tabaka şeklinde aşınma partiküllerinin oluştuğu dikkat çekmektedir. Şekil 4b'de yüksek sıcaklıklarda yapılan aşınma testi sonucunda kaplamalarda hacim kayıplarının olduğu da gözlemlenmiştir. Yüksek sıcaklıklarda numunelerin hacim kayıplarının azalmasını ve aşınma sonucunda malzemelerin yüzeyden koparak ayrılması, artan sıcaklık değeri ile birlikte malzemenin plastik deformasyonunun artmasına sebep olacaktır. Ayrıca, sıcaklığın artması ile birlikte kaplama tabakasını oluşturan elementlerin (Cr, Ni) oksitlenme oranın artacağından dolayı, MCrAlY metalik bağ kaplamaların mekanik özelliklerinin artması, aşınma direncini de arttıracaktır.

#### 4. Sonuclar

TBC'ler gaz türbin motorlarının sıcak bölgelerinde bulunan süper alaşım komponentleri yüksek sıcaklık çalışma koşullarına karşı koruma amaçlı yaygın olarak kullanılmaktadır. Konvensiyonel olarak TBC sistemlerinde kullanılan metalik bağ kaplama alaşımları MCrAIY kompozisyonuna sahiptir. MCrAlY metalik bağ kaplamaların TBC sistemindeki sağladığı üstün özellikler sebebiyle gaz türbinlerindeki önem derecesi yüksektir. Konvansiyonel TBC'lerde metalik bağ kaplamanın temel görevi, kullanılan altlık malzemenin oksidasyon, korozyon ve aşınma hasarı gibi oluşumlara karşı korunmasını sağlamaktır. Bu malzemeler, hem yüksek ısı akışını engellemek amacıyla kullanılan TBC sistemindeki üst kaplama yapısının altlık yüzeye yapışmasını sağlayan yapı katmanı olarak hem de agresif yüksek sıcaklık koşullarından altlık yüzeyin korunmasında kullanılmaları yönüyle birincil düzeyde öneme sahiptir. Barındırdıkları üstün mekanik ve tribolojik özellikleri sayesinde farklı birçok alanda kullanım alanı bulmaktadırlar.

#### Kaynaklar

- 1. Meşekıran, N., Farklı Üst Kaplama İçeriğine Sahip Termal Bariyer Kaplamaların(TBC) Oksidasyon Davranışlarının İncelenmesi. Bartın Üniversitesi, 2015.
- 2. Schneider, K. E., Belashchenko V., Dratwinski M., Siegmann S. ve Zagorski A. Thermal Spraying for Power Generation Components, 2006, 271 s. Germany,
- 3. Dorfman, M. R., Thermal Spray Coatings," in Handbook of Environmental Degradation of Materials (Second Edition), Oxford: William Andrew Publishing. 2012, pp. 569–596.
- 4. Lima, C.R.C., Guilemany J.M., Adhesion Improvements of Thermal Barrier Coatings with HVOF Thermally Sprayed Bond Coats, Surface and Coatings Technology, 2007, 201, 4694– 4701.
- 5. Karaoglanli, A. C. "Termal Bariyer Kaplamalarda Bağ Tabakasının Farklı Yöntemlerle Üretilmesi ve Özelliklere Etkisi." (Yayınlanmamış doktora tezi), Sakarya Üniversitesi Fen Bilimleri Enstitüsü, Metalurji ve Malzeme Mühendisliği Anabilim Dalı, Sakarya, 2012.
- 6. Tafralı, M., Tel Püskürtme ile Kaplamada Püskürtme Parametrelerinin Mikroyapı ve Gözenekliliğe Etkisi. Yüksek Lisans Tezi (yayınlanmış), Gazi Üniversitesi Fen Bilimleri Enstitüsü, Metal Eğitimi Anabilim Dalı, Ankara, 2006, 96 s.
- 7. Özkavak, H.V. "Plazma ile Polimer Kaplanmış Metalik Malzemelerin Bazı (Tribolojik) Özelliklerinin Araştırılması. "(Yayınlanmış doktora tezi), Süleyman Demirel Üniversitesi Fen Bilimleri Enstitüsü, Makine Mühendisliği Anabilim Dalı, Isparta, 2013.

8. Dowson, D., Coatings Tribology. Amsterdam, Elsevier Science B.V, 1998. 457s, Nerherland.

The International Conference of Materials and Engineering Technology

- 9. Sivakumar, et al., High Temperature Coatings For gas Turbine Blades, Surf. Coat Tesh. **1989**, 37 139-160.
- Minisker, M.A., Termal Sprey Yöntemiyle Oluşturulan Kaplamaların Mekanik Özelliklerinin İncelenmesi (Yüksek Lisans Tezi). İstanbul Teknik Üniversitesi, Fen Bilimleri Enstitüsü, 2009.
- 11. Richard C. S. et al., The influences of heat-treatments and interdiffusion on the adhesion of plasma-sprayed NiCrAlY coatings, Surf. Coat. Techn. 82, **1996**, 99-109
- 12. Bose, S., High Temperature Coatings. Butterworth-Heinemann: Elsevier Burlington, 2007.
- Dongsheng, W., Zongjun, T., Songlin, W., and Lida, S., Microstructure and wear resistance of lasercladding nano-Al2O3/MCrAlY composite graded coating on TiAl alloy, Appl.Mech. 2012, Mater. 217-1350–1353.
- Tao, C., Wang, L., and Song, X., High-temperature frictional wear behavior of MCrAlYbased coatings deposited by atmosphere plasma spraying, International Journal of Minerals. 2016, Northeastern University, Shenyang 110819, China.
- Pereira, J., Zambrano, J., Licausi, M., Tobar, M., Amigo, V., Tribology and high temperature friction wear behavior of MCrAIY laser cladding coatings on stainless steel. Wear. 2015, 330-331 280–287.
- Kuo, C.M., Yang, Y.T., Bor, H.Y., Wei, C.N., Tai, C.C., Aging effects on the microstructure and creep behavior of Inconel 718 superalloy. Materials Science and Engineering, 2009, A 510–511, 289–294.



ne International Conference

aterials and Engineering Technology

# MUSTAFA BAKKAL<sup>\*1</sup>, ALI TANER KUZU<sup>2</sup>, MAHMUT YILMAZ<sup>3</sup>, ERAY ÇELİK<sup>3</sup>, FİKRET ERTÜRK<sup>3</sup>, BERKAY YUCETURK<sup>1</sup>, İREM DURULMUŞ<sup>1</sup>

 <sup>1</sup> Istanbul Technical University, Mechanical Engineering Faculty, Mechanical Engineering Department, Istanbul, TURKEY
 <sup>2</sup> Yeditepe University, Engineering Faculty, Material Science and Nanotechnology Engineering Department, Istanbul, TURKEY.
 <sup>3</sup> Yılmaz Makina, Istanbul, TURKEY.

#### Özet

PVC ısıtıcı yüzeyleri iyi elektrik iletkenliği, korozyon dayanımı ve hafiflik beklentileri sebebiyle genellikle Al 7075 malzemeden imal edilmektedir. Al 7075 ısıtıcı yüzeyleri kullanım sırasında hızlı bir şekilde aşındığından, yüzeyleri dışarıdan ilave PTFE folyo ile kaplanmakta, zamanla bu folyodaki aşınma sonrasında yeni folyo ile kaplamaya devam edilmektedir. Ancak PTFE folyo hem iletkenliği düşürmesi hem de yüksek maliyeti nedeniyle imalatçı firmalar farklı çözüm yolları bulmaya çalışmaktadır. Bu çalışmada çözüm olarak akımsız nikel kaplama önerilmektedir. Akımsız nikel kaplama yöntemi, sertlik ve korozyona karşı dayanıklılığı nedeniyle endüstriyel ihtiyaçları karşılamak için sayısız değişikliğe uğramıştır. Önerilen çalışmada endüstride ağırlıklı olarak kullanılan Nikel-Fosfor (Ni-P) kaplamaları yerine daha sert ve ısıl işleme daha uygun olan Nikel-Bor (Ni-B) ve Ni-B-PTFE kaplama seçilmiştir. Hazırlanan kaplama banyosu nano kompozit oluşturmak üzere Al 7075 yüzeyine kaplanmıştır. Akımsız Ni-B Al 7075 yüzeyine doğrudan kaplanamadığı için zinkatlama ön işlemleri denenmiş ve başarılı bir kaplama elde edilebilmiştir. Ardından yapılan ısıl işlem sonrasında yüzeyde hem yapışmazlık hem de yüksek sertlik elde edilmiştir.

Keyword: Ni-B, Ni-B-PTFE, Kaplama, Akımsız Nikel Kaplama, Sertlik

#### 1. Giriş

Kimyasal veya oto-katalitik kaplama olarak da bilinen akımsız kaplama, harici bir elektrik akım kaynağı kullanılmayan sulu bir çözeltide aynı anda birkaç reaksiyonun gerçekleştirildiği bir kaplama yöntemidir. En yaygın elektriksiz kaplama yöntemi akımsız nikel kaplamadır. Akımsız nikel kaplama, malzeme yüzeyinde amorf yapıda bir birikinti oluşturur. Bu birikinti, akım dağılımına bağlı olmadığından, kaplanmış yüzeyin büyüklüğü veya şeklinden bağımsız olarak neredeyse homojen kalınlıktadır [1].

Akımsız Nikel kaplamanın malzeme yüzeyine birikmesinde çeşitli faktörler etkidir. Bu faktörler arasında, banyonun kullanım süresi, banyo konsantrasyonu, çalışma sıcaklığı, uygulama zamanı, banyo hacmi, banyonun pH değeri ve banyo katkıları sayılabilir. Banyo konsantrasyonu, banyonun her bir bileşeninin miktarını ile ilişkilidir. Metal kaynağı veya indirgeyici ajandaki bir artış, kaplama hızında ve dolayısıyla indirgenmiş metalde bir artış sağlar. Kaplanacak malzeme banyo çözeltisine temas ettiği sürece ya da çözelti çözünen metal iyonlarını tüketene kadar kaplama işlemi (reaksiyon) devam edecektir [2, 3].

Kaplama işlemi boyunca banyonun kararlı halde kalması önemlidir. Akımsız kaplama banyolarında metal partiküllerinin üretimini kontrol ederek banyo stabilitesi sağlanır [4]. Banyo kararlı olduğu sürece kaplama süresi ile metalin birikmesi arasında lineer bir ilişki vardır.

The International Conference of Materials and Engineering Technology

Çalışma sıcaklığı, akımsız bir banyonun birikme hızını değerlendirmede anahtar bir parametredir. Düşük sıcaklıklar reaksiyona düşük miktarda enerji verir ve bu da düşük biriktirme hızlarına neden olur. Öte yandan, çok yüksek sıcaklıklılardan dolayı banyo çok aktif olacak ve banyo dengesiz olacaktır. Jothi ve diğerleri [5] diğer bütün parametrelerin sabit tutulduğunda, metal biriktirme oranının artan sıcaklıkla üstel olarak arttığını gözlemlemişlerdir. Ni-P banyoları 60 °C ve daha yüksek işletme sıcaklıklarına ihtiyaç duyarken 90 °C'yi aşan bir banyo sıcaklığı çözeltinin bozulmasına yol açabilir [6]. Nİ-B kaplama banyolarında da durum farklı değildir. Bu banyolar genellikle 85 – 95 °C sıcaklık aralığında işlev görürüler ve banyo sıcaklığının artması ile birlikte kaplama hızında da üstel bir artış görülür [7].

Banyonun pH değeri birikintideki fosfor içeriğini etkileyen diğer önemli bir parametredir. Yüksek pH değerleri birikintide daha az fosfor içeriği sağlarken, düşük pH değerleri yüksek fosfor birikintileri oluşturur. Chen ve diğ. [7], 4 pH değerine sahip asidik banyoda kaplama içeriğinde %25 fosfor gözlemlemelerine rağmen alkalin bir banyoda kaplamada % 1'den daha az fosfor içeriği elde edilir. Asitli banyolarda oluşan tortular, çekme gerilmesi oluşturmaya meyilliyken, alkali banyolardan gelen tortular basma gerilmesi oluşturmaya meyillidir. pH'a bağlı olan bir diğer özellik ise asidik banyolar kullanılarak oluşturulan kaplamaların çeliğe iyi yapışma özellikleridir ve bu muhtemelen endüstrideki geniş kullanımını açıklamaktadır [8]. Akımsız Ni-B kaplamanın gerçekleşmesi için ise gerekli reaksiyonların oluşabilmesi için çözelti pH'ı 12'nin üzerinde olmalıdır (tercih edilen değer 14'tür). Kaplama çözeltisinin yüksek alkali doğasından ve prosesin yüksek sıcaklıklarda gerçekleştirilmesinden dolayı kaplama yapılacak malzemelerin de alkali banyolara ve yüksek sıcaklığa karşı dirençli olması gereklidir. Son olarak, talyum bileşenlerinin kurşun tuzlarına oranla çok daha zehirli oldukları ve alternatif proseslerin bulunması durumunda kullanımından elden geldiğince sakınılması gerektiği söylenilebilir. Borhidrür içerikli banyoların dezavantajlarının ve işlem zorluklarının üstesinden gelebilmek ve aynı zamanda Ni-B kaplamaların fiziksel ve kimyasal özelliklerini korumak için özellikle son yıllarda kullanımı artış gösteren aminoborlu banyolar önerilmektedir [7].

Akımsız Ni-B, Ni-P ve Ni-P/Ni-B kaplamaların çok büyük kısmı çelik taban üzerine uygulanmaktadır. Bu kaplamanın sağladığı en büyük yarar korozyon dayanımının gelişmesi, yüksek sertlik seviyelerine çıkılması, aşınma direncinin artırılması başta olmak üzere çeşitli mekanik ve manyetik özelliklerinin değiştirilebilmesidir. Çok geniş uygulama alanı bulunan bu kaplama yönteminde çelik taban üzerine kaplamalar özellikle otomotiv sanayinde, raylarda, uçak-uzay teknolojilerinde kullanılmaktadır. Alüminyum üzerinde yapılan kaplamalar ise çeliğe nazaran daha zordur. Alüminyum üzerine kaplama üç temel sebepten dolayı sorun teşkil etmektedir.

- Alüminyumun oksijene karşı özel bir ilgisi vardır ve saf metal havaya maruz kaldığında neredeyse anında, elektriksel yalıtkan gibi davranan, doğal, ince bir oksit tabakası meydana gelecektir. Böyle bir yüzeye kaplanan tabaka yapışmayacaktır.
- Alüminyumun bu derece aktif olduğunu düşünürsek, üzerine kaplayabileceğiniz metallerin neredeyse hepsi ona karşı katodik davranacaktır. Bu, kaplamanın yüzey üzerinde sürekli olması, gözenek ve çatlak içermemesi gerektiği anlamına gelmektedir. Alüminyumu dış etkiye maruz bırakabilecek en ufak bir hata, kaplamada olacak ileriki uzun süreli zararlarla alüminyumun hızla korozyona uğramasına yol açacaktır.
- Alüminyumun diğer metallere göre kabaca üç-dört kat sıcaklıkla daha fazla genleşmektedir. Bu yüksek sıcaklıklarda kaplamayı metalden kaldıracak ısıl gerilmelere sebep olabilir.


Bu çalışmada Al 7075 malzemenin üzerine Ni-B ve Nİ-B-PTFE nano-kompozit kaplama yapılmıştır. Çalışmada öncelikle alüminyum malzemenin üzeri temizlik ve ön işlemler yapılmış olup daha sonra kaplama gerçekleştirilmiştir. Yapılan kaplamalar optik mikroskop ve sertlik ölçümleri ile analiz edilmiştir.

#### 2. Deney Detayları

Tasarlanması amaçlanan ısıtıcının, elektik rezistansı üzerine döküm ile uygulanması gerektiğinden bu malzemenin erime ısının, çelik rezistansa zarar vermemesi için çelikten daha düşük olması gerekmektedir. Isıl iletkenlik, hafiflik ve erime ısısı açısından en uygun malzeme Al7075 olarak belirlenmiştir. Kaplama 3 adımda gerçekleşmiştir; *i*) ön işlemler (temizleme ve zinkatlama), *ii*) kaplama çözeltisine daldırma *iii*) ısıl işlem.

## 2.1 Ön İşlemler

Kaplamaya başlamadan önce numunenin temiz olduğundan emin olunmalıdır. Her numunenin temiz olmadığı varsayımını yaparak yağ alma işlemi yapmamız gerekir ve yüzeyin kaplamaya uygun hale getirilmesi gerekir. Her malzemenin kendine özgü ön işlemi vardır. Ön işlemlerden önce mutlaka temizlik yapılmalıdır. Temizlik adımları şu şekilde takip edilmiştir;

- 1. Sırasıyla 400, 600, 800 ve 1000'lik zımpara kâğıtları ile zımparalama
- 2. Sıcak yağ alma (60-90°C)
- 3. Methanol ile yıkama
- 4. Asit banyosuna (hacimsel olarak 50% H2SO4 + 1% HF) daldırma

Alüminyum üzerine giriş kısmında bahsedilen nedenlerden dolayı kaplama yapılamamaktadır. Bu çalışmada alüminyum imersiyon yöntemi olarak zinkatlama seçilmiştir. Çalışmada zinkat banyosu hazır olarak temin edilmiştir. Zinkatlama işleminin efektif olması için yüzey 1-2 µm Ni-P kaplanmıştır. Zinkatlama işlemi 2 defa yapılmış olup adımları aşağıda verilmiştir.

1. zinkatlama :Zinkat banyosu (2 dk.)  $\rightarrow$  durulama2. zinkatlama :1. Zinkat banyosu  $\rightarrow$  %30'luk H2SO4 asit banyosu (1 dk.)  $\rightarrow$  durulamazinkat banyosu (1 dk.)  $\rightarrow$  durulama



Şekil 1. Zinkatlama kimyasalları

## 2.2 Banyonun Hazırlanışı

Banyolar iki farklı şekilde hazırlanabilir. Bunlardan ilki, indirgeyici ve dengeleyici içeren tek bir ana çözeltinin hazırlanmasıdır. Diğer yöntem ise indirgeyici ve dengeleyici çözeltilerin ayrıca hazırlanması ve ana çözeltiye sonradan eklenmesidir. Bu çalışmada daha verimli olmasından dolayı ikinci yöntem seçilmiştir. Çalışmada kullanılan, üç aşamalı (Ana çözelti, indirgeme çözeltisi ve dengeleyici çözelti) banyonun hazırlanışı aşağıdaki verilmiştir.

## <u>Ana çözeltinin reçetesi;</u>

- 1. 24 g/L nikel klorür distile suyun içine eklenir ve karıştırılır.
- 2. 60 g/L EDA karışıma eklenir.

3. 26.5 g/L potasyum hidroksit eklenir. Bu sayede banyonun pH değeri 12-14 arasına yükselecektir.

## Şekil 2. Nikel

İndirgeme çözeltisinin reçetesi;

- 1. 100 g sodyum borohidrit distile suya eklenir ve karıştırılır.
- 2. 200 g sodyum hidroksit çözeltiye eklenir.

## Dengeleyici çözeltinin reçetesi;

- 1. 13 g/L sodyum hidroksit distile suya eklenir.
- 2. 2.6 g/L kurşun tungstat eklenir.
- 3. 40 mL/L EDA eklenir.
- 4. 13 g/L etilen diamin tetra asetik asit (EDTA) eklenir. Çözelti berrak görünüme gelene kadar karıştırılır.

Bu noktada, çözeltilerin ana banyoda birleştirilmesi gerekmektedir. Birleştirme işleminin adımları;

1. Ana çözelti manyetik karıştırıcıyla karıştırılarak ısıtılır. Banyo sıcaklığının, kaplamanın yapılabilmesi için gereken optimum değer olan 90 °C 'ye yükselmesi beklenir.

- 2. İndirgeyici çözeltiden 2.6 mL ana çözeltiye eklenir.
- 3. Dengeleyici çözeltiden 2.6 mL ana çözeltiye eklenir.



İndirgeyici eve dengeleyici çözelti eklenmesi, istenilen kaplama kalınlığı elde edilene kadar 30 dakikada bir tekrarlanmalıdır. Banyonun pH değeri pHmetre yardımıyla ölçülerek 12-14 arasında olması sağlanmalıdır. Optimum değer denemeler sonucu 13.5 olarak gözlemlenmiştir. Temel ENi-B banyosu hazırlandıktan sonra nanokompozit kaplama için çözeltiye 15g/L %60 PTFE dispersiyonu eklenir. Kaplama çözeltisini hazırlamak için Teflon PTFE DISP 30LX hazır dispersiyonu kullanılmıştır. Bu dispersyon, PTFE sulu çözeltisidir ve halihazırda surfactant içermektedir. PTFE Dispersiyon Özellikleri Tablo 1'de özetlenmiştir.

Tablo 1 PTFE Dispersiyon Özellikleri					
Özellik	Birim	Tipik Değer			
PTFE partikül içeriği	%	60			
Surfactant İçeriği	%	6			
Dispersiyon Yoğunluğu	g/cm <sup>3</sup>	1.51			
Ortalama Partikül Boyutu	μm	0.23			
pH Değeri		10			



Şekil 2. Teflon partikülleri eklenmemiş ENi-B banyosu

Bütün adımlar uygulanıp, şartlar sağlandıktan sonra temizleme işlemi ve ön işlem yapılmış olan numuneler banyoya daldırılır ve kaplama başlar. Bu çalışmada kaplama süresi 1 saat olarak seçilmiştir.

# 2.3 Isıl İşlem

Kaplamaların yüzey sertliklerinin arttırılması için kaplamalar ısıl işleme tabi tutulmuştur. Çalışmada kaplamalar 400° C derecede 1 saat fırında bekletilerek ısıl işlem gerçekleştirilmiş ve böylece kaplamaların sertlikleri iyileştirilmiştir.



Şekil 3. Kaplama sonrası yüey durumu

# 3. Deney Sonuçları

## 3.1 Kalınlık Ölçümü

Kaplanan numunenin kaplama tabakasının kalınlığının ölçümü numuneden bir kesit alıp, zımparalama ve parlatma işlemlerinden sonra optik mikroskopta kalınlık ölçümleri yapılmıştır. 1 saatlik banyo uygulamasından sonra kaplama kalınlığı ENi-B için ortalama 30µm, ENi-B-PTFE için ortalama 10 µm olarak ölçülmüştür.

## 3.2 Sertlik Ölçümleri

Sertlik değerleri, her bir numunede yapılan en az 3 ölçümün ortalaması alınarak belirlenmiştir. Sertlik ölçümleri "Shimadzu" marka, HMV2 tipi, MicroVickers sertlik ölçüm cihazı ile 3 N kuvvet yük altında yapılmıştır. Kaplama yapılmamış aluminyumda sertlik değeri 150 HV iken Ni-B kaplama ile beraber sertlik değeri 540 HV değere çıkmıştır. Isıl işlem ile kaplamanın sertlik değer 680 HV değerine kadar çıkmıştır. Ni-B kaplamaya nano PTFE eklenerek yapılan kaplamanın sertliği 230 HV ölçülürken ısıl işlem ile beraber nano-kaplamanın sertliği 510 HV olarak ölçülmüştür.

<b>1 abio 2.</b> Kapianiniş yüzeylerin serink degenen						
NUMUNE	Al 7075	Al 7075 ENi-B	Al 7075 ENİ-B+Iİ	Al 7075 ENi-B- PTFE	Al 7075 ENi-B- PTFE+Iİ	
Vickers sertliği	150	540	680	230	510	

Tablo 2. Kaplanmış yüzeylerin sertlik değerleri

## 3.3 Yapışmazlık Testleri

Sıcak silikonun kaplanmış yüzeye tatbik edilmesi ardından yüzeye yapışması gözlemlenerek bu test yapılmıştır. Hem ENi-B hem de ENi-B-PTFE kaplama ile yapışmazlık sağlandığı görülmüştür.

## 4. Sonuçlar

Çalışmada elde edilen sonuçlar aşağıda özetlenmiştir;

- Alüminyum üzerine ENi-B için kaplama için 30µm/saat, ENi-B-PTFE nano kompozit kaplama için 10 µm/saat ortalama kaplama hızı tespit edilmiştir.
- Yapılan ısıl işlem ile ENİ-B kaplamanın sertliği %20 (540 HV'den 680 HV'ye) ve ENi-B-PTFE nano kompozit kaplamaların sertliği % 100'den fazla (230 HV'den 510'ye) arttırılabildiği gösterilmiştir.



The International Conference of Materials and Engineering Technology

• Sıcak silikon ile yapılan yapışmazlık testinde her iki kaplamanında yüzeylerinin yapışma konusunda benzer özellik gösterdiği belirlenmiştir.

## Kaynaklar

[1] Schlesinger, M. 2010. Modern electroplating, 5th edn. John Wiley & Sons Inc

[2] Lobanova, L. L., Batalova, E. V., Khranilov, Y. P. 2008. "Reagent techniques for nickel recovery from spent electroless nickel-plating solutions." Russian Journal of Applied Chemistry, 81(2), 202-206.

[3] Agarwala, R. C., Vijaya, A. 2003. "Electroless alloy/composite coatings: A review." Sadhana, 28.3-4, 475-493.

[4] Yin, X., Hong, L., Chen, B. H., Ko, T. M. 2003. "Modeling the stability of electroless plating bath—diffusion of nickel colloidal particles from the plating frontier." Journal of colloid and interface science, 262(1), 89-96.

[5] Jothi S, Lian J, Wei S (2013) J Alloys Compd 571:183

[6] Guo, R. H., Jiang, S. X., Yuen, C. W. M., Ng, M. C. F., Lan, J. W., Zheng, G. H. 2012. Influence of deposition parameters and kinetics of electroless Ni-P plating on polyester fiber." Fibers and polymers, 13(8), 1037-1043.

[7] Chen Z, Xiaoda X, Chee CW, Subodh M (2003) Surf Coat Technol 167:170

[8] Braun, F., Ana M. T., Laura M. C. 2011. "Optimization and characterization of electroless codeposited PdRu membranes: Effect of the plating variables on morphology." Journal of membrane science 382.1-2, 252-261.



ANTİBAKTERİYEL ÖZELLİKLERİNİN İNCELEMESİ

# DERYA TEKİN<sup>\*1</sup>, DERYA BİRHAN<sup>2</sup>, TANER TEKİN<sup>3</sup>

<sup>1</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, Erzurum, TÜRKİYE.
 <sup>2</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, Erzurum, TÜRKİYE.
 <sup>3</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Kimya Mühendisliği, Erzurum, TÜRKİYE.

# Özet

Son yıllarda, organik – inorganik nanokompozitler piller, sensörler, yakıt hücreleri, süperkapasitörler, güneş pilleri, biyosensörler antibakteriyel özelliklerin incelenmesi gibi farklı uygulamalardaki potansiyel kullanımları nedeniyle polimer - inorganik malzemelere olan ilgi artmaktadır. Bu polimerler arasında, Polivinilalkol (PVA), mükemmel film oluşturucu, emülsifiye edici ve yapışkan özelliklere sahip ticari olarak suda çözünebilen bir polimer türüdür. Bu bildiri kapsamında, TiO<sub>2</sub> nanopartikülleri ve PVA – TiO2 nanokompoziti cast metodu kullanılarak sentezlenmiştir. Sentezlenen nanokompozitlerin morfolojik ve elementel analizi SEM-EDS, kristal yapıları ise XRD kullanılarak incelenmiştir. Titanyum (IV) izopropoksitten yola çıkarak TiO2 nanopartikülleri sentezlenmiş, ikinci aşamada ise, TiO<sub>2</sub> nanopartikülleri PVA ile modifiye edilmiştir. PVA – TiO<sub>2</sub> nanokompozitinin SEM sonuçlarına göre, filmlerinin yapılarında kümelerin ortaya çıktığı görülmektedir. EDS sonuçları oksijen ve titanyumun varlığını kanıtlamaktadır. XRD sonuçları, TiO2'nin anataz fazını doğrulamakta ve TiO<sub>2</sub> nanopartiküllerinin polimer ile modifiye edilmesi TiO<sub>2</sub> piklerini etkilememektedir. Sentezlenen TiO<sub>2</sub> nanopartikülleri ve PVA – TiO<sub>2</sub> nanokompozitinin antibakteriyel aktiviteleri Escherichia coli (ATCC 25922) bakteri türü kullanılarak Time Kill Curve (zamana bağlı yok etme) yöntemi kullanılarak gerçekleştirilmiştir. Antibakteriyel aktivite deneme sonuçlarına göre, TiO2 nanopartiküllerinin %14,42, PVA – TiO<sub>2</sub> nanokompozitinin ise % 19,23 oranında bakteri giderdiği tespit edilmiştir.

Anahtar Kelimeler: TiO<sub>2</sub> nanopartikülleri, PVA – TiO<sub>2</sub> nanokompoziti, Antibakteriyel aktivite

## 1.Giriş

Nanomalzemelerin benzersiz özelliklerini mikro biyolojide uygulama çabalarıyla yapılan materyal araştırması, gelecekteki antimikrobiyal teknolojilerini genişletme konusunda büyük bir potansiyele sahiptir. Antimikrobiyal ajanlar, çevre, gıda, sentetik tekstiller, ambalajlama, sağlık ve tıbbi bakım ürünlerindeki bir dizi endüstriyel uygulama için oldukça önemli olmaktadır [1]. Nanoteknoloji, gıda ürünleri için ambalajlama ve depolama uygulamalarında kullanılan fotokatalitik nanopartiküller ve polimer nanokompozit filmler formunda gelişmiş antimikrobiyal kaplama elde etmeye yardımcı olabilir [2]. Polimer esaslı nanokompozitler, son yıllarda yüksek performanslı hafif malzemeler elde etmek amacıyla önemli ölçüde dikkat çekmektedir [3]. Polivinilalkol (PVA), mükemmel film oluşturma, emülsifiye edici ve yapışkan özelliklere sahip önemli, suda çözünebilen bir polimer türüdür. Polivinil alkol (PVA), kokusuz, toksik olmayan, biyouyumlu özelliklere sahip olup doku mühendisliği, ilaç dağıtımı, eklem kıkırdak ve biyosensörler gibi biyoteknolojik uygulamalarda yaygın olarak kullanılmaktadır [4]. PVA, iyi film morfolojisi nedeniyle mükemmel bir matris malzemesidir. Bununla birlikte, PVA, neme karşı oldukça hassas olmaktadır, buda polimerin dayanıklılığını ve dengesini azaltmaktadır [5].

Bu probleme çözüm olarak, döküm yöntemi kullanılarak TiO<sub>2</sub> nanopartikülleri ile birlikte, PVA matriksli nanokompozit malzemeler sentezlenmiştir.

The International Conference of Materials and Engineering Technology

Titanyum dioksit (TiO<sub>2</sub>), kimyasal kararlılık, mükemmel optik geçirgenlik, iyi elektriksel özelliklere sahip olduğundan nanokompozitlerin geliştirilebilmesi için en önemli dolgu maddesi olmaktadır [6]. TiO<sub>2</sub> boyaya duyarlı güneş hücreleri, fotokatalizörler, su arıtma ve antimikrobiyal çalışmalarda yaygın olarak kullanılmaktadır [7]. TiO<sub>2</sub> tozları sol – jel tekniği kullanılarak ince film üretiminde kullanılmaktadır.

Bu çalışmada, TiO<sub>2</sub> nanopartikülleri sentezlenip PVA matriks üzerine dopp edilmiştir. Sentezlenen TiO<sub>2</sub> nanopartikülleri ve PVA- TiO<sub>2</sub> nanokompoziti SEM, XRD teknikleri kullanılarak karakterize edilmiştir. Antibakteriyel aktiviteleri için ise, *E.coli* bakterisi kullanılmıştır.

#### 2. Materyal Ve Yöntem

#### 2.1. Materyaller

Titanyum (IV) izopropoksit (Sigma Aldrich, %97), etanol (Sigma Aldrich, %99,8), Polivinilalkol (Mw 9,000-10,000,80% hydrolyzed,PVA, Sigma Aldrich) ve distile su kullanılmıştır.

#### 2.2. Yöntem

#### 2.2.1. TiO<sub>2</sub> nanopartiküllerinin sentezi

TiO<sub>2</sub> nanopartiküllerini sentezlemek için ilk önce 20 mL Titanyum (IV) izopropoksit 140 mL etanol içerisinde 15 dakika için manyetik olarak karıştırıldı. Karıştırma işlemi tamamlandıktan sonra solüsyon 80°C'de 1 saat süre ile reflux işlemine tabi tutuldu. Reflux işleminden sonra Titanyum solüsyonu süzülüp 60°C'de kurutuldu. Kurutma işleminin ardından 500°C'de 3 saat süre ile kalsinasyon işlemi gerçekleştirildi. Saf Titanyum nanoparçacıkları elde edilmiştir.

#### 2.2.2. PVA – TİO<sub>2</sub> nanokompozitinin sentezi

1 g polivinil alkol (PVA) tozları 25 mL iyonize su içerisinde 70°C'de 1 saat süre ile karıştırıldı. Diğer tarafta 25 mL iyonize su içerisinde sentezlenen saf TiO<sub>2</sub> nanopartikülleri 30 dakika boyunca ultrasonikasyon işlemine tabi tutuldu. Sonikasyon işleminin ardından PVA solüsyonu titanyum solüsyonunun içerisine damla damla ilave edildi ve 3 saat boyunca karıştırıldı. Karışma işlemi tamamlandıktan sonra solüsyon cam petri kaplarında 40°C'de 2 gün süre ile kurutulmuştur.

#### 2.2.3. Antibakteriyel Test

Sentezlenen TiO<sub>2</sub> nanopartikülleri ve PVA-TiO<sub>2</sub> nanokompozitinin antibakteriyel aktiviteleri *Escherichia coli* (ATCC 25922) bakteri türü kullanılarak Time Kill Curve (zamana bağlı bakteri sayısında azalma) yöntemi kullanılarak gerçekleştirilmiştir. Sentezlenen nanokompozitlerin optik yoğunluğu 600 nm'lik dalga boyu (OD600) ile absorbans değerleri okunarak nanokompozitlerin bakteri gideriminde ne kadar etkili olduğu incelenmiştir.





3. Sonuçlar ve Tartışma 3.1. SEM ve XRD Analizi





Şekil 1. TiO2 nanopartikülleri (a) SEM görüntüsü, (b) EDS spektrumu

Şekil 1'de TiO<sub>2</sub> nanopartiküllerinin 100 nm'deki SEM görüntüsü gösterilmektedir. Buna göre, TiO<sub>2</sub> nanopartiküllerinin aglomera olduğunu gözlemliyoruz. TiO<sub>2</sub> nanopartiküllerinin küresel şeklini ve gözenekli yapısını SEM görüntüleri açıkça ortaya koymaktadır [8].EDS spekturumları Oksijen ve Titanyumun varlığını doğrulamaktadır.



Şekil 2. PVA-TiO<sub>2</sub> nanokompoziti (c) SEM görüntüsü, (d) EDS spektrumu

Şekil 2'de PVA-TiO<sub>2</sub> nanokompozitine ait 1 µm'deki SEM görüntüsü görülmektedir. Buna göre, PVA-TiO<sub>2</sub> filmlerinin yapılarında kümelerin ortaya çıktığı görülmektedir. Kompozit filmler için elde edilen morfoloji, literatürde de bildirildiği gibi tozla doldurulmuş polimer kompozit malzemelere benzer özellikler göstermektedir [9]. EDS spekturumları Oksijen ve Titanyumun varlığını doğrulamaktadır.



Sekil 3. TiO<sub>2</sub> nanopartikülü ve PVA-TiO<sub>2</sub> nanokompozitine ait XRD diyagramı

Şekil 3'de sentezlenen saf TiO<sub>2</sub> nanopartiküllerinin XRD sonuçlarında TiO<sub>2</sub> nanopartikülünün anataz formu için JCPDS (JCPDS No. 21-1272) kartları ile uygun yoğun kırınım pikleri göstermektedir. TiO<sub>2</sub> nanopartiküleri, 3 saat boyunca 500°C'de tavlama sonrasında polikristalin anataz fazına dönüşmektedir. Sentezlenen PVA-TiO<sub>2</sub> nanokompozitinin XRD sonuçlarında PVA – TiO<sub>2</sub> nanokompozitinde, bir anataz TiO2 fazı sergilenmektedir ve JCPDS (No. 21-1272) kartı ile de uyumluluk gösterdiği görülmektedir. PVA -TiO2 nanopartiküllerinin kristalleşme sağladığı ve sadece anataz fazının gözlemlenebildiğini görülmektedir.

# 3.2. Antibakteriyel Aktivite

Hazırlanmış olan numunelerin başlangıç ve 24 saat sonundaki absorbans ve E. Coli değerleri Tablo 1'de verilmiştir.

Maddeler	Başlangıç		24 saat sonunda		
	Absorbans	Absorbans CFU/mL Absorbans		CFU/mL	
Kör Numune	0.125	$1,0*10^8$	0,936	9,36*10 <sup>7</sup>	
TiO <sub>2</sub>	0.125	1,0*10 <sup>8</sup>	0,801	8,01*10 <sup>7</sup>	
PVA/TiO <sub>2</sub>	0.125	1,0*108	0,756	7,56*107	

Tablo 1. Na	nokompozitlerin e	eklemiş olduğu ve re	ferans E. Coli	i bakterilerinin ab	sorbans ve CF	U değerleri
	Maddeler	Başlaı	ıgıç	24 saat so	onunda	
		Absorbons	CEU/mI	Abcorbong	CEII/mI	



Şekil 4. Nanokompozitlerin 24 saat sonunda bakteri konsantrasyonunda meydana getirdiği azalma grafiği



#### 4. Sonuç

Yapılan çalışma sonucunda PVA matrisi üzerine dopp edilen TiO<sub>2</sub> nanopartikülleri bakteri konsantrasyonunda % 19,23 oranında bir giderim göstermiştir. TiO<sub>2</sub> nanopartikülleri modifiye edilerek antimikrobiyal özelliklerinin iyileştirildiği tespit edilmiştir.

#### Referanslar

- Rajeshkumar, S. and C. Malarkodi, In vitro antibacterial activity and mechanism of silver nanoparticles against foodborne pathogens. Bioinorganic chemistry and applications, 2014. 2014.
- 2. Dhanasekar, M., et al., Ambient light antimicrobial activity of reduced graphene oxide supported metal doped TiO2 nanoparticles and their PVA based polymer nanocomposite films. Materials Research Bulletin, 2018. **97**: p. 238-243.
- 3. Zhou, T., et al., The preparation of high performance and conductive poly (vinyl alcohol)/graphene nanocomposite via reducing graphite oxide with sodium hydrosulfite. Composites Science and Technology, 2011. **71**(9): p. 1266-1270.
- 4. Zhu, Y., et al., Nanoindentation and thermal study of polyvinylalcohol/graphene oxide nanocomposite film through organic/inorganic assembly. Applied Surface Science, 2015. **349**: p. 27-34.
- 5. Hdidar, M., et al., Influence of TiO2 rutile doping on the thermal and dielectric properties of nanocomposite films based PVA. Journal of Alloys and Compounds, 2018. **750**: p. 375-383.
- 6. Rathod, S.G, et al., Pressure sensitive dielectric properties of TiO 2 doped PVA/CN-Li nanocomposite. Journal of Polymer Research, 2015. **22**(2): p. 6.
- 7. Ahmadpoor, P., A.S. Nateri, and V. Motaghitalab, The optical properties of PVA/TiO2 composite nanofibers. Journal of applied polymer science, 2013. **130**(1): p. 78-85.
- 8. Singh, R., S.G. Kulkarni, and S.S. Channe, Thermal and mechanical properties of nanotitanium dioxide-doped polyvinyl alcohol. Polymer bulletin, 2013. **70**(4): p. 1251-1264.
- 9. Li, S., et al., Facile green synthesis of Degraded-PVA coated TiO2 nanoparticles with enhanced photocatalytic activity under visible light. Journal of Physics and Chemistry of Solids, 2019. **129**: p. 92-98.

# r-GO /TiO2 NANOKOMPOZİTİNİN ÜRETİMİ, KARAKTERİZASYONU VE SERTLİK ÖZELLİKLERİNİN İNCELENMESİ

he International Conference

aterials and Engineering Technology

# DERYA TEKİN<sup>\*1</sup>, DERYA BİRHAN<sup>2</sup>, TANER TEKİN<sup>3</sup>

<sup>1</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, Erzurum, TÜRKİYE.
<sup>2</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, Erzurum, TÜRKİYE
<sup>3</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Kimya Mühendisliği, Erzurum, TÜRKİYE.

#### Özet

Yarı iletken fotokatalizler, farklı katalitik reaksiyonlarda çok önemlidirler. TiO<sub>2</sub>, WO<sub>3</sub>, CdS, ZnS, GaN, ZnO, In<sub>2</sub>O<sub>3</sub> ve BiVO<sub>4</sub> gibi farklı tiplerde fotokatalizörler mevcut olmaktadır. Titanyum dioksit (TiO<sub>2</sub>), maliyet etkinliği, düşük toksisite, yüksek stabilite, mükemmel biyouyumluluk, yüksek kimyasallık ve benzersiz fotokatalitik özellikler gösterdiği için birçok uygulama alanında kullanılan bir yarı iletken fotokatalizör olmaktadır. Son yıllarda grafen esaslı nanokompozitler iki bileşenin avantajlı özelliklerini bir araya getiren yüksek katma değere sahip değerli malzemeleri üretmek için bir araya getirilmiştir. Grafen bal peteği şeklinde 2D kafes yapısına sahip sp<sup>2</sup> hibritleşmesi yapan, karbon atomları arasındaki mesafe 0.142 nm olan bir karbon tabakasıdır. İki boyutlu olarak indirgenmiş GO'nun (rGO) kenar ve bazal düzlemleri içerisinde var olan fonksiyonel grupların içerdiği az miktardaki oksijen miktarının varlığı büyük önem taşımaktadır. TiO<sub>2</sub> nanopartiküllerinin rGO ile hibritleşmesi, yapıya birçok avantaj sağlamaktadır. Bunun nedeni ise r-GO elektron deliği çiftlerinin rekombinasyonunu azaltmakta ve yük transfer oranını artırmaktadır. Bu bildiri kapsamında r-GO/TiO<sub>2</sub> nanokompozit katalizörü sentezlenmiştir. Sentezlenmiş olan nanokompozitin yapısal özelliklerini incelemek için SEM-EDS ve XRD analizleri kullanılmıştır. Sertlik özelliklerinin incelenmesi için Vickers sertlik cihazı kullanılmıştır. r-GO/ TiO2 nanokompozitinin sentezinde, ilk aşamada destek malzemesi Hummer metodu ile sentezlenmiş ve ikinci aşamada da sol-jel yardımı ile r-GO/TiO<sub>2</sub> nanokompoziti sentezlenmiştir. Elde edilen r-GO/TiO<sub>2</sub> nanokompozitinin SEM görüntülerinde, TiO<sub>2</sub> nanopartiküllerinin ortala çapının ~ 15 nm olduğu ve çeşitli şekil ve boyutlara sahip olduğu gözlemlenmiştir. rGO tabakalarının tamamen TiO<sub>2</sub> nanopartikülleri ile kaplandığı tespit edilmiştir. XRD sonuçlarında, TiO2'nin sahip olduğu anataz kristal yapısının kırınım pikleri görülmektedir.

Sertlik ölçümü sonrası, sol-jel yöntemi ile sentezlenmiş r-GO/TiO<sub>2</sub> nanokompozitinin ortalama sertlik değeri 74,34 HV olarak ölçülmüştür. Yapının sahip olduğu karbon – karbon (C=C) kovalent bağı en güçlü bağlardan olmaktadır ve yapı içerisine katıldığında oldukça iyi mekanik özellikler gösterdiği görülmektedir.

Anahtar kelimeler: İndirgenmiş grafen oksit (r-GO), TiO<sub>2</sub> nanopartikülü, Hardness Vickers

#### 1. Giriş

İyi bilinen fotokatalitik bir malzeme olan Titanyum oksit (TiO<sub>2</sub>), maliyet etkinliği, düşük toksisitesi ve yüksek kimyasal kararlılığı nedeniyle birçok alanda büyük ilgi görmektedir [1]. TiO2, yüksek oksitleme kapasitesi, orta bant aralığı, fotokorozyon ve kimyasal aşınmaya karşı toksik olmayan, ucuz ve yüksek kimyasal kararlılığasahip olması gibi üstün özellikleri nedeniyle, araştırılmış bir yarı iletken fotokatalisttir [2].

Bununla birlikte, TiO<sub>2</sub>'nin pratik uygulamaları, bazı sahip olduğu dezavantajlar nedeniyle büyük ölçüde engellenmiştir. Foton üreten elektron – holl çiftlerinin hızlı bir şekilde rekombinasyon olması, görünür ışık bölgesini yeterli derecede kullanamaması, UV bölgesine yakın absorbsiyon göstermesi gibi dezavantajlara sahip olmaktadır [1, 2]. Bu problemleri çözebilmek için metal iyon katma, karbon veya azot katkılama, metal yükleme gibi birçok yöntem kullanılmaktadır. Bunlar arasında katalizörün verimliliğini artırmanın en iyi yolu, karbon ve türevlerinin kullanılmasıdır. Bu karbon materyaller arasında en iyi olanı iki boyutlu grafenin kullanılmasıdır [3]. Son zamanlarda grafen, TiO<sub>2</sub>'nin mekanik, fotokatalitik, termal ve elektrik iletkenlik özelliklerini artırmak için yaygın olarak kullanılanı

The International Conference of Materials and Engineering Technology

Grafen, altıgen bir ağa bağlanan ve bu ağın karbon atomlarından oluştuğu tek tabakalı bir malzemedir. Grafen, karbon nanotüplerin (CNT) ve büyük fullerenlerin imalatında yapı taşı olarak kullanılmaktadır. Grafen, oda sıcaklığında kuantum Hall etkisinin gözlemlenmesi, yüksek mekanik mukavement, uygun ısıl iletkenliği, yüksek Young modülü, kırılma direnci gibi birçok üstün fiziksel özelliklere sahip olmaktadır. Grafen oksitin indirgenmesi birden fazla indirgeyici ajanlar kullanılmaktadır. Hidrazin hidrat, dimetil hidrazin, dimetil formaldehit gibi indirgeyici ajanlar termal yöntemler ve ultraviyole ışınlamaları kullanılarak indirgenmektedir [5]. Esas olarak, grafen grafen oksitin (GO) kimyasal olarak indirgenmesi redüklenmiş grafen oksitin (rGO) kenar ve bazal düzlemlerinde yer alan az miktarda oksijen içeren fonksiyonel gruplar büyük öneme sahip olmaktadır [6]. Redüklenmiş grafen oksit, karbon ailesinin yeni allotropu olarak; rGO'yu destekleyen nanopartiküller ile iyi bir temas sağlayabilmesi için mükemmel bir destek malzemesi, sp<sup>2</sup> hibritleşmesi yapmaktadır.Karbon yapısının düzlemsel formunu oluşturma ve r-GO/TiO<sub>2</sub> nanokompozitlerinde elektron – holl çiftlerinin rekombinasyonunun etkili bir şekilde engelleme gibi yapı içerisinde birçok benzersiz işleve sahip olmaktadır [7].

Sıkıştırma, bükülme mukavemeti ve sertliği gibi birçok mekanik özelliği geliştirmek adına reçinelere dolgu maddeleri ilave edilmektedir. Geliştirilmiş fiziksel özellikler arasında, polimerizasyon büzülmesinde bir azalma ve elastiklik modülünde bir artış ortaya çıkmaktadır. Kompozit yapıda ki reçinelerin fiziksel ve mekanik özellikleri, yapı içerisine katkılanan dolgu maddelerinin özelliklerinden etkilenmektedir. Bir grafen katmanındaki karbon – karbon (C=C) kimyasal bağı muhtemelen doğada bilinen genişletilmiş bir sistemdeki en güçlü kimyasal bağdır. Grafen allotropları sorunsuz bir şekilde toplanmış grafen katmanları olduğu için, keşfedildiği günden itibaren bu nanoyapıların mükemmel mekanik özelliklere sahip olduğu ve bu özelliklerin miktarının ölçülmesi nanoteknoloji alanında büyük ilgi görmektedir. Nanokompozit malzemelerin sertliği, kalıcı girinti veya penetrasyona direnç olarak tanımlanır. Sertlik test ölçüm yöntemlerinde, belirli bir yük altında hareketli prop yüzeye batırılarak ve bir girinti oluşturularak ölçüm yapılır.

Bu çalışmada,TiO<sub>2</sub> nanopartikülleri ve r-GO/TiO<sub>2</sub> nanokompozit yapıları sentezlenip SEM,XRD kullanılarak karakterize edilmiştir. Nanokompozitlerin sertlikleri Vickers sertlik cihazı kullanılarak belirlenmiştir.

#### 2. Materyal ve Yöntem

#### 2.1. Materyaller

Grafit tozları (powder<45 $\mu$ m $\geq$ 99.99%, Sigma Aldrich), Sülfürik asit (H<sub>2</sub>SO<sub>4</sub>,95-97%, Merck), Sodyum Nitrat (NaNO<sub>3</sub>), Potasyum Permanganat (KMNO<sub>4</sub>), DMF(N-N-Dimethylformamide, $\geq$ 99%, Sigma Aldrich) ve Hidrojen peroksit (H<sub>2</sub>O<sub>2</sub>, % 30, Sigma Aldrich), Titanyum (IV) izopropoksit (Sigma Aldrich, %97), Etanol (Sigma Aldrich, %99,8) temin edilmiştir.





# 2.2. Yöntem

## 2.2.1. TiO2 nanopartiküllerinin sentezi

TiO<sub>2</sub> nanopartiküllerini sentezlemek için ilk önce 20 mL Titanyum (IV) izopropoksit 140 mL etanol içerisinde 15 dakika için manyetik olarak karıştırıldı. Karıştırma işlemi tamamlandıktan sonra solüsyon 80°C'de 1 saat süre ile reflux işlemine tabi tutuldu. Reflux işleminden sonra Titanyum solüsyonu süzülüp 60°C'de kurutuldu. Kurutma işleminin ardından 500°C'de 3 saat süre ile kalsinasyon işlemi gerçekleştirildi. Saf Titanyum nanoparçacıkları elde edilmiştir.

## 2.2.2. Grafen Oksitin Sentezi

Grafen oksit grafit tozu Hummer Metoduna göre sentezlenmektedir. İlk önce 69 ml sülfirik asit, 3 *g* grafit tozu, 1.5 *g* NaNO<sub>3</sub> bir 250ml erlen içerisinde karıştırıldı. Daha sonra çözelti sıcaklığının 0°C'ye inmesi için bir buz banyosu içerisine yerleştirilmiştir. Sıcaklık 0°C düştükten sonra 9 *g* potasyum permanganat çözelti içerisine ilave edildi. Potasyum permanganatın ilavesi bittikten sonra çözelti buz banyosu içerisinden çıkarılarak sıcaklığının oda sıcaklığına çıkması için beklemeye alındı. Daha sonra 138 ml iyonize su ilavesi yapıldı ve 98°C'de 15 dakika karıştırıldı ve 480 ml su ilavesi yapıldıktan sonra % 30'luk H<sub>2</sub>O<sub>2</sub> damlatılarak mevcut kahverengi rengin sarıya dönüşmesi sağlanarak oksitleme işlemi tamamlandı. Elde edilen grafen oksit birkaç kez etanol ve iyonize su ile yıkanarak pH dengesi sağlandı. Daha sonra süzüldükten sonra 60°C'lik fırında tamamen kuruması gerçekleşene kadar bekletilmektedir.

## 2.2.3. rGO Sentezi

0.8~g GO tozları 400 ml DMF içerisinde ultrasonikasyonda çözündürülerek 6 saat reflux işlemi gerçekleştirildi. 6 saatlik işlemin sonunda rGO tozları süzülerek etanol ve su ile yıkanarak 60°C'lik firinda kurutuldu.

## 2.2.4. rGO-TiO2 Nanokompozit Yapısının Sentezi

0.1 g rGO tozu 10 ml etanol ve 20 ml su ile karıştırılarak 1 saat sonikasyon işlemine tabi tutuldu. 1 saatlik işlemin sonunda 0.2 g anataz formundaki TiO<sub>2</sub> nanotozları rGO solüsyonunun içerisine ilave edildi ve 1 saatlik karışma süresinin sonunda solüsyon petri kaplarına dökülerek 60°C'lik fırında kurutuldu. Ve rGO-TiO<sub>2</sub> nanotozları elde edildi.



Şekil 2. r-GO/TiO2 nanokompozitinin (c)SEM görüntüsü, (d)EDS analizi

Şekil 2'de görüldüğü gibi, rGO tabakaları tamamen TiO<sub>2</sub> nanopartikülleri ile kaplanmıştır. Bu iki malzeme arasında mükemmel bir elektrik iletkenliği istiyorsak rGO levhaları ve TiO<sub>2</sub> nanopartikülleri arasındaki bu güçlü etkileşim kurmak oldukça önemlidir [9]. EDS spektrumları C,O ve Ti varlığını doğrulamaktadır.



Şekil 3. TiO2 nanopartikülü ve r-GO/TiO2 ait XRD diyagramları

314

Şekil 3'de görüldüğü gibi, Sentezlenen saf TiO<sub>2</sub> nanokompozitinin XRD sonuçlarına göre, TiO<sub>2</sub> nanopartikülü anataz formu için JCPDS (JCPDS No. 21-1272) kartları ile uygun yoğun kırınım pikleri göstermektedir. TiO<sub>2</sub> nanopartiküleri, 3 saat boyunca 500 ° C'de tavlama sonrasında polikristalin anataz fazına dönüşmektedir [10]. r-GO/TiO<sub>2</sub> nanokompozitinin kırınım pikleri, anataz formundaki TiO2'nin kırınım pikleri ile benzerlikler göstermektedir [11].

## 3.2. Sentezlenen Nanokompozitlerin Sertlik Ölçümü

Sentezlenen TiO<sub>2</sub> nanopartikülleri ve rGO-TiO<sub>2</sub> nanokompozit yapısının mikrosertlik analizleri 245 mN (0,025) yük altında 10 saniye tutularak Hardness Vickers değerinde 5 sertlik değerinin ortalaması alınarak yapılmıştır. Şekil 4'de numunelerin sertlik değerleri grafik halinde gösterilmektedir.



Şekil 4. Nanokompozitlerin sertlik grafiği



TiO<sub>2</sub> nanopartikülüne ait sertlik değerlerinin ortalaması 53,96 HV olarak ölçülmüştür. 500°C'de kalsine edilen TiO<sub>2</sub> nanopartikülleri iyi mekanik özellikler ile sonuçlanmaktadır. Literatürde yapılan çalışmalarda, sıcaklığın artırılması ile sertliğin daha da arttığı tespit edilmiştir [12].

Tablo 2. r-GO/TiO <sub>2</sub> nanokompozitinin sertlik değerleri							
1	2	3	4	5			
79 (HV)	70 (HV)	79,2 (HV)	71,2 (HV)	72,3 (HV)			

r-GO /TiO<sub>2</sub> nanokompozitinin ortalama sertlik değeri 74,34 HV olarak ölçülmüştür. Bir karbon allotropu olan grafen, grafit ve pırlantaya yakın mekanik özellikler göstermesi beklenmektedir. Yapının sahip olduğu karbon – karbon (C=C) kovalent bağı en güçlü bağlardan olmaktadır ve yapı içerisine katıldığında oldukça iyi mekanik özellikler gösterdiği görülmektedir [13].

Technology



# 4. Sonuçlar

Sentezlenen TiO<sub>2</sub> nanopartikülü ve r-GO/TiO<sub>2</sub> nanokompozit yapılarının sertlik ölçümünde TiO<sub>2</sub> nanopartillerinin içerisine destek maddesinin ilave edilmesi ile yapının önemli ölçüde mekanik olarak iyileştiği tespit edilmiştir.

# Referanslar

- 1. Zhao, W., et al., Synthesis of Ag/TiO2/graphene and its photocatalytic properties under visible light. Materials Letters, 2016. **171**: p. 182-186.
- 2. Maruthamani, D., D. Divakar, and M. Kumaravel, Enhanced photocatalytic activity of TiO2 by reduced graphene oxide in mineralization of Rhodamine B dye. Journal of Industrial and Engineering Chemistry, 2015. **30**: p. 33-43.
- 3. Galińska, A. and J. Walendziewski, Photocatalytic water splitting over Pt- TiO2 in the presence of sacrificial reagents. Energy & Fuels, 2005. **19**(3): p. 1143-1147.
- 4. Luo, L., et al., Hydrothermal synthesis of fluorinated anatase TiO2/reduced graphene oxide nanocomposites and their photocatalytic degradation of bisphenol A. Applied Surface Science, 2015. **353**: p. 469-479.
- 5. Didehban, K., L. Mohammadi, and J. Azimvand, Preparation of RGO/Fe3O4/poly (acrylic acid) hydrogel nanocomposites with improved magnetic, thermal and electrochemical properties. Materials Chemistry and Physics, 2017. **195**: p. 162-169.
- 6. Sohail, M., et al., Synthesis of well-dispersed TiO2@ reduced graphene oxide (rGO) nanocomposites and their photocatalytic properties. Materials Research Bulletin, 2017. **90**: p. 125-130.
- 7. Wang, D., et al., Enhanced photoelectrocatalytic activity of reduced graphene oxide/TiO2 composite films for dye degradation. Chemical engineering journal, 2012. **198**: p. 547-554.
- 8. Singh, R., S.G. Kulkarni, and S.S. Channe, Thermal and mechanical properties of nanotitanium dioxide-doped polyvinyl alcohol. Polymer bulletin, 2013. **70**(4): p. 1251-1264.
- 9. Morais, A., et al., Nanocrystalline anatase TiO 2/reduced graphene oxide composite films as photoanodes for photoelectrochemical water splitting studies: the role of reduced graphene oxide. Physical Chemistry Chemical Physics, 2016. **18**(4): p. 2608-2616.
- 10. Vishwas, M., et al., Effect of tio2 nano-particles on optical, electrical and mechanical properties of poly (vinyl alcohol) films. Procedia Materials Science, 2014. **5**: p. 847-854.
- 11. Yang, Y., et al., One-step hydrothermal synthesis of surface fluorinated TiO2/reduced graphene oxide nanocomposites for photocatalytic degradation of estrogens. Materials Science in Semiconductor Processing, 2015. **40**: p. 183-193.
- 12. Oktar, F., Hydroxyapatite–TiO2 composites. Materials Letters, 2006. **60**(17-18): p. 2207-2210.
- 13. Lau, K.-T., et al., Micro-hardness and flexural properties of randomly-oriented carbon nanotube composites. Journal of composite materials, 2003. **37**(4): p. 365-376.



# ADEM KURT<sup>1</sup>, H.İBRAHİM TOFAN<sup>2</sup>

<sup>1</sup>Gazi University Faculty of Technology Metallurgycal and Materials Engineering Dep., Ankara, TURKEY.

In this study, the effect of friction and wear properties of pine cone powder by adding a certain amount of asbestos-free organic additives into brake pads composition was investigated. With this experimental formulation, in addition to this composition, the organic-based balata composition in commercial use was adopted as a reference and 4 different sample groups were formed by using powder metallurgy method by adding 3%, 5% and 10% pumice cocci powder by weight, in turbula brand mixer 10 minutes. 1200kg at 180°C after mixing. pressed under pressure. The samples produced were tested on the chase type wear and friction test device. As a result of the test, according to the reference sample, the pine cone had an oil effect by reducing the coefficient of friction in the samples with powder added.

#### 1. Introduction

Friction materials in industrial and automotive applications are divided into three main groups. These are organic, semi-metallic and asbestos-free pads. All these varieties are grouped with similarities of the substances in them while considering their performance characteristics. The materials that make up the friction materials used in the automotive industry can be categorized into four classes according to their similar properties and intended use [1]. These; reinforcing materials, fillers, binding materials and friction adjusting materials.

The application of friction materials is mainly due to the high coefficient of friction and good sliding properties. However, in order to perform the functions of the friction materials as expected, the high coefficient of friction is not sufficient. It should maintain a constant coefficient of friction as much as possible under stresses and particularly at high temperatures, rather than a high coefficient of friction. Furthermore, the amount of wear should be low, the counter material should not be scratched and damaged, and the mechanical strength should be sufficient to withstand the stresses [2,3].

The tribological behavior of the lining depends on the applied load, environmental conditions and parameters such as microstructure [4,5]. In some cases, it is known that the frictional force is not evenly distributed across the brake and lining interfaces. Especially during long braking, the temperature and braking force on the friction surface change periodically. This change causes the coefficient of friction to be reduced and increased [6].

In this study, 3, 5 and 10% red pine cone powder was added to the organic brake lining composition and its effect on friction and abrasion properties was investigated.

#### 2. Material And Method

In this study, asbestos free organic brake lining composition was created and used as a reference. In addition to the reference sample, 4 different samples were produced by powder metallurgy by adding 3, 5 and 10% pine cone powder. The effect of added pine cone powder on the friction and wear characteristics of the lining was observed by the test.



Brake lining samples were prepared by adding pine cone powder in different proportions on the basis of mass ratio to the organic lining composition which we accept as reference. Powder from pine cone was also produced by mechanical alloying machine (Figure 1) in Gazi University Metallurgical and Materials Engineering Department's Exactive Metallurgical Laboratory.



Figure 1. Mechanical Alloying Machine



Figure 2. a) Pinecone, b) pinecone particle c) Pinecone powders

Pine cone and powders are shown in Figure 2. 300gr. weight (B0) sample composition and in addition to this composition 3% (B1), 5% (B2) and 10% (B3) four different sample groups were obtained by adding pine cone powder (Table 1).

		The inte		
Materials	B0(Reference)	B1	B2	B3
Rosin	Х	Х	Х	X
Steel wool	X	Х	X	X
Rock wool	Х	Х	Х	Х
Graphite	Х	Х	Х	Х
Brass shavings	Х	Х	X	Х
Vermiculite	Х	Х	Х	Х
Zrsio <sub>3</sub>	X	Х	Х	Х
Barite	X	Х	Х	Х
Rubber	X	Х	Х	X
Calcite	X	Х	Х	X
Coloring	X	Х	Х	X
Copper shavings	X	Х	X	X
Friction dust	Х	Х	Х	Х
Pinecone	0	3	5	10

ials and Engineering Technology

Precision weighing lining powder mixes they mixed in Turbula for 10 min. Mixed powders in die for 30 mm diameter of 2 MPa at 180°C temperature pressed under pressure.

Pressed lining materials are shown in Figure 3.

UWE dithe Bristol Bristol



Figure 3. Pressed lining materials a) Reference sample, b) with 3% pinecone powders, c) with 5% pinecone powders, d) with 10 % pinecone powders.

319



Pressed densities of pine cone dusty and reference samples are given in the Table 2.

Sample no	Pressed density (g/cm <sup>3</sup> )
ВО	1.45
B1	1.38
B2	1.46
B3	1.41

#### Wear testing:

The test device is based on the measurement of the friction force and temperature formed by contacting the appropriately sized lining sample to the gray cast iron drum and transferring the data to the computer environment. The compressor consists of a compressor that applies a braking force to the sample at the values specified in the standards. The test device is shown in Fig.4.



Figur 4. Wear test machine

## **3.Results And Discussion**

UWE Bristol

In this study, the results of the Burnish and Resetting stages in the Chase test experiment were obtained and evaluated. Try the 280 mm rotating sample at a speed of 308 rpm for 1200 seconds at the Burnish section. diameter gray cast iron drum, 45kg. contact with the sample. Temperature and friction coefficients ( $\mu$ ) of the lining samples B0, B1, B2, and B3 are shown in Table 3.

The International Conference of Materials and Engineering Technology

	<b>B0</b>		B1	]	B2	]	B3
T( <sup>0</sup> C)	μ	$T(^{0}C)$	μ	$T(^{0}C)$	μ	$T(^{0}C)$	μ
25	0.332	84	0.413	82	0.298	26	0.375
28	0.336	76	0.412	74	0.315	30	0.382
30	0.357	61	0.431	65	0.317	35	0.396
33	0.387	55	0.450	59	0.319	37	0.403
37	0.415	51	0.442	52	0.327	43	0.411
39	0.441	51	0.434	53	0.341	45	0.420
43	0.476	52	0.451	53	0.345	46	0.438
45	0.494	52	0.471	52	0.348	47	0.440
46	0.502	52	0.477	54	0.351	48	0.449
49	0.523	52	0.484	54	0.353	49	0.455
51	0.533	55	0.492	54	0.354	51	0.460
53	0.531	57	0.497	54	0.357	52	0.475
54	0.533	58	0.501	56	0.380	52	0.483
54	0.526	58	0.510	57	0.412	53	0.495
54	0.528	59	0.517	57	0.421	54	0.506
55	0.526	55	0.522	58	0.438	54	0.508
55	0.529	55	0.525	59	0.441	54	0.507
56	0.530	55	0.527	63	0.460	54	0.508



**Figure 4**.Firiction coefficient of samples

According to the graph obtained at the end of the experiment, it was observed that the friction coefficient of the pine cone powder lining samples added to the B0 coded reference sample was reduced compared to the reference sample. The pine cone powder added to the reference sample (B0) continued to reduce the friction coefficient by up to 5% and increased by 5% (B2) to 10% (B3).

Wear rates calculated based on the weight of the lining samples obtained before and after the wear test showed a decrease in the wear rates of the B1 and B2 coded samples compared to the B0 coded reference sample and an increase was observed in the B3 coded sample. Probably increases the amount of wear by reducing wear resistance in the pad as pinecone powder increases.

## 4. Results And Recommendations

Pine cone doped brake lining samples; 3%, 5% and 10% of the composition prepared as a reference to the results obtained by adding 4 different samples obtained as a result of experimental studies were compared and the following results were reached:

1.Pine cone powder added to brake lining samples in 3%, 5% and 10% ratios decreased the friction coefficient according to the sample we accept as reference.

2. Pine cone powder can be said to have a lubricating effect as it decreases the coefficient of friction according to the data reached.

3. At the end of the experimental studies, even though it was observed that the friction coefficient of pine cone powder added to the brake lining sample composition was generally reduced, an increase of 10% was observed while the decrease continued up to 5%.

4.When we look at the wear rate of samples, B1 and B2 coded samples compared to the reference sample (B0), while an increase in B3 coded sample was observed.

5. After the tests of the lining samples produced, it is possible to investigate to what extent the reduction in the coefficient of friction ceases after 5% of the cone powder added to the composition of the reference sample.



#### References

1. Washabaugh, F. J., EMCOR 66 Ultra-Short Fibers for Asbestos-Free Friction Materials, SAE 860630, 1987.

2. Reinsch, E. W., Sintered metal brake linings for automotive applications, Delco-Moraine division, General motors corp dayton, Ohio, cilt 2 sayfa 9-21, 1970.

3. Gediktaş, M., Sürtünme malzemelerinin deneysel tayini, İstanbul Teknik Üniversitesi Matbaası, Gümüşsuyu, İstanbul, 1968.

4. Habig, K. H. and Woydt, M., Sliding Friction and Wear of Al2O3, ZrO2 Si3N4, Proc. 5th International Cong. on Tribology, Finland, Cilt 3, sayfa 106-113, 1989.

5. Habig, K. H., Sliding Wear of Ceramic-Ceramic, Ceramic-Steel and Steel-Steel Pairs in Lubricated and Unlubricated Contact, Wear, Cilt 133, sayfa 1-22, 1989.

6. Lee, K. and Barber, J. R., An Experimental Investigation of Frictionally Excited Thermoelastic Instability in Automotive Disc Brakes under a Drag Brake Application, Trans. ASME J. Tribology, Cilt 116, sayfa 409- 414, 1993.

# AN EXPERIMENTAL INVESTIGATION ON THE CHARPY IMPACT RESPONSE OF GLASS/EPOXY COMPOSITES AGED IN SEAWATER

1

The International Conference of Materials and Engineering Technology

#### Ahmet ERKLİĞ<sup>1</sup>, Zeynal Abidin OĞUZ<sup>2</sup>, Ömer Yavuz BOZKURT<sup>3</sup>

<sup>1</sup> Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY.
 <sup>2</sup> Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY.
 <sup>3</sup> Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY.

#### Abstract

This study highlights the influence of seawater absorption on Charpy impact properties of glass/epoxy composites. For this study, two groups of glass/epoxy composites that were produced by using vacuum infusion method were immersed in seawater at 25°C and 70°C temperatures and one group was immersed any condition. Specimens were keep in water until getting saturation point. The immersed specimens reached saturation at the end of sixth weeks (1000 h). Charpy impact test (having 15 J impact capacity) were conducted three groups that are dry samples, saturated samples and redried samples after saturation. From the Charpy impact test result it was observed that impact properties degradation occured due to hydrothermal aging. Compared to their non-immersed state, maximum impact stress energy of immersed specimen have dropped seriously, regardless of temperature. Also, as temperature increased, the reduction rate of maximum impact stress energy increased. Similar results occured at re-dried group. That is the decrease rate in aged samples at 25°C is 27.3%, whereas this rate is 30.03% in aged samples at 70°C. However, when compared to their dry state, the decrease in maximum impact stress in the re-dried group is considereably lower than in the satureted group. The decrease rate in re-dried samples aged samples at 25°C is 2.6%, whereas this rate is 7.77% in re-dried samples aged at 70°C.

Keywords: Hydrothermal Aging, Seawater, Charpy Impact Test

#### **1.Introduction**

Lately, the use of composite materials has become one of the most popular research subjects as they provide weightlessness, high strength, high modulus of elasticity and a wide range of application area. Various types of polymer-matrix composites are being considered for use in different fields of application, ranging from sporting goods to structural materials for automotive, marine, and aerospace industries. In such applications, the composites are exposed to harsh and variable environments ranging from variations in temperature and exposure to moisture, including elevated temperature immersion and "hot-wet" exposures. Hydrothermal ageing research on composites has increased over the last years because these materials have been intensively used for many applications in humid environments. Water absorption and mechanical loading have been recognised as the major factors which affect the mechanical composite properties through a wide range of chemical and physical phenomena. The major environmental ageing factors such as heat, moisture, salt water and varied loads, or their combinations may degrade the material properties [1]. The behavior of composite structures under transient impact loading and the ways to enhance their characteristics to withstand this type of dynamic loading might be of specific significance in the aerospace sector and other applications. Several questions must be addressed, however, before such a materials substitution can be made. First, the long-term durability of polymeric composite materials in sea water must be determined. While polymeric composite materials are not subject to galvanic corrosion as are metals, the polymeric matrix can absorb 1%-2% water at ambient temperature (with the equilibrium level of moisture content increasing with increasing temperature), which may degrade the mechanical properties of either the matrix itself or the fibre/matrix interface [2].

Many investigations have focused on the influence of either chemical or environmental effects on the various composite system properties. Numerous works have been studied the fiber modification effect on the mechanical properties of fiber-reinforced polymers (FRP).

The International Conference of Materials and Engineering Technology

The effect of moisture coupled with temperature on Glass/Epoxy composite in the presence of the dissolved salts in sea water has been reported by several workers [3-8]. Sea water absorption causes changes in the thermo-physical, mechanical and chemical characteristics of the matrix by the processes of both plasticization and hydrolysis. It is reported [9] that the effect of sea water on GFRP composites differs with matrix material and conditioning environment. In Glass/Epoxy composite, in particular, the mode of failure is altered from a brittle matrix and ductile fibre to ductile matrix and brittle fibre.

Hawa et al. [10] studied the burst strength and impact behaviour of hydrothermally aged GRE pipes under impact levels of 5, 7.5, and 10 J. The results revealed that the aged and impacted specimens had low burst strength.

The topic of repeated impact response of glass/epoxy composites subjected to thermal aging was investigated by Atas et al. [1]. At the end of study they have found that for both single impact and repeated impact cases, it seems that the overall damage area increases with aging duration, especially for smaller impact energies. Also they have noted that the variations of the impact characteristics such as absorbed energy, peak contact force, maximum deflection and contact time for successive impacts until perforation of the samples are affected significantly by thermal aging.

The effects of seawater and impact loading on the fatigue life of glass/epoxy composite pipes under cyclic internal pressure were investigated by Deniz et al. [11, 12]. The pipes produced by a filament-winding technique were immersed in seawater for periods of 3, 6, and 9 months. Impact tests with three different energy levels (5, 7.5, and 10 J) were conducted on the specimens, and fatigue damage such as perspiration, leakage, and eruption were observed.

Strait at, al [13] studied the effects of seawater immersion on the impact resistance of glass fiber reinforced epoxy composites. systems following seawater immersion. Results indicate that moisture-induced degradation can significantly reduce the impact resistance of glass fiber reinforced epoxy composites due to energy after peak load decreased and total energy absorbed was reduced significantly.

Vijay at. al. [14] investigated the effect of moisture absorption on tensile properties of  $SiC_p$  filled glass fiber reinforced epoxy composite material. At the end of study they have found that the tensile strength degradation occurs due to immersion in seawater and normal water.

This study focuses on the Charpy impact behavior of glass/epoxy composite kept in seawater at two different temperatures (25°C and 70°C). The glass/epoxy composites were immersed hydrothermal aging conditions until specimens reach saturation point. After saturation, the composite samples were removed the ambient conditions and one group of samples subjected immediately to Charpy impact test, one group of samples subjected to Charpy impact test after samples re-dried and the last group conducted to test as dry state. The effects of temperature on the impact strength of glass/epoxy were investigated in accordance with experimental results.

# 2. Experimental Procedure

#### **2.1 Material Production**

Woven S-glass fibre/epoxy composite plates were produced by using of method of vacuum -assisted resin-infusion method (VARIM) as twelve ply laminates. An epoxy system consisted of MGS L 285 resin and MGS H 285 hardener was used as matrix material in manufacturing with mass ratio 100:40. The properties of S-glass woven fabric are 202 g/m2, 3000-5000 MPa,72-82 GPa and 0.15 mm respectively for density, tensile strength, elastic modulus and thickness. The production process was carried out on a vacuum device and control unit-connected heater table. At the first step, a release film was unrolled on the heater table then twelve-layer glass-fibre fabrics and peel ply were laid on top of it. A distribution network was embedded in order to gain quick and homogenous resin spread. A vacuum-bagging film was pasted from the edges by a sealant tape to wrap the layers. A vacuum duct and a resin inlet were connected to the vacuum bag's two opposite sides with air tight (Fig.2). Making sure that there is no leakage in the vacuum bag, the mixture of resin and fast hardener was impregnated. The leakage was controlled from vacuum bag that is the pump vacuum was kept constant as 0.12 MPa. Therefore, the mixture diffused into the layers gently. After the impregnation process was fully completed, the valves of resin inlet and vacuum tubes were turned off in order to keep the pressure constant in the vacuum bag. Then the laminate was left for curing for 2 h at the temperature 45°C. Once the curing was done, the laminate was left for cooling at room temperature throughout 12 hours.

The International Conference

aterials and Engineering Technology



Figure 1. S-glass woven fabric



Figure 2. Production of glass fibre/epoxy composite plates by VARIM



# 2.2 Specimen Preparation

Charpy impact test specimens were cut with the help of the CNC rooter according to ISO 179/92 [15] as can be seen in figure 3. The samples that would be immersed water before test was not sandpapered due to the sandpaper process may prevent water absorption. Instead of sanding the samples, fringes of samples were cut by using an electronic cutter. After cutting of fringes, samples were numbered and prior to placing specimens into the environmental chambers all specimens were dried on the vacuum tables with 50°C until take a constant weight to make samples completely dry. By this way samples became moisture free. Before immersed samples to sea water, samples were weighed to obtain the dry weight of them. The thickness of samples was taken by a digital caliper and recorded.

The International Conference

aterials and Engineering Technology



Figure 3. Cutting Process of Glass/Epoxy Plates with CNC rooter

# 2.3 Hydrothermal Aging Process

Immersing process was done by cabins made of heat resistant glass and silicon.  $25^{\circ}$ C temperature was used for room temperature, and 70°C temperature was used for hot condition. The hydrothermal aging was carried out based on ASTM D5229/D5229M-14 [16]. According to standard a conditioning chamber is required that shall be capable of maintaining the required temperature to within  $\pm 3^{\circ}$ C. To be able to obtain constant heat inside of the whole cabins, adjustable resistance has been installed to cabins. Due to the position of resistance in the cabins, there are different temperatures in the cabin at different points. To avoid this problem, a small circulation pump was placed to cabin. By this way the change of heat could be achieved as nearly zero. The change of heat inside of the cabin may lead to misinterpretation of water absorption of composite laminates due to it changes the density of fluid and thus fluidity of water. All equipment's of cabin are shown in figure 4. The temperature control was done every day by a digital thermometer.



Figure 4. Aging Cabin





Figure 5. Refractometer

The groups are named as SW25 for specimens at 25°C, SW70 for specimens at 70°C. For each groups of composite, fifteen specimens were prepared to immerse hydrothermal aging environments, five of them for to saturate, and five of them for re-dried and five of them for dry (non-immersed).

# 2.4 Impact Test

To be able to understand the low velocity impact behaviour of glass/epoxy composites immersed in sea water at different temperature, Charpy impact test was conducted. Test was done with three groups. The first group consist of wet samples. This group was immersed 1000 h in a seawater cabin at 25°C and 70°C. After saturation point, samples were taken out and conducted to Charpy impact test immediately. The second group consist of re-dried samples. Samples of this group were removed after waiting 1000 h in a seawater cabine and heated until they reached constant weight on a heat table at 50°C. The last group is dry samples. These samples were not immersed hydruthermal aging condition. Tests were performed by ISO 179/92 standard [15]. Impact tester Köger 3/70 device with pendulum energy of 15 J shown in Fig.6 a) schematic illustration and b) test machine was used to investigate the low velocity impact behavior of composite samples. Test samples with dimensions of 55 x 10 mm as seen in Fig. 8 were subjected to impact loads flatwise. Charpy impact test device consists of a pendulum with a mass attached to a rotating arm connected to the device body. Pendulum falls from a certain height and strikes the test sample and sample absorbs part of pendulums kinetic energy. The absorbed impact energy and impact strength of material were calculated from Eq.1.



Figure 6. a) Schematic Illustration of Charpy Impact Test b) Test Machine



$$K = \frac{W}{A} \qquad (1)$$

Where *W* is the absorbed energy (J), *A* is the area (m<sup>2</sup>) and *K* is the impact resistance (J/m<sup>2</sup>)

terials and Engineering Technology

 $W = E_1 - E_2 \quad (2)$ 

Where  $E_1$  and  $E_2$  are initial and final potential energies respectively.



Figure 7. Test Samples

# 3. Results

Five specimens of the glass/epoxy composite materials were conducted to Charpy impact test as dry state at room temperature to obtain the control impact properties which include impact strength, and absorbed energy at failure. Replicas of five specimens for each condition of the conditioned samples that are saturated and re-dried groups were tested to determine the combined effects on the impact properties. Average of five samples was evaluated finally for each conditions. The schematic illustration of impact test of glass/epoxy composites is shown in figure 8.



Figure 8. Impact Resistance of Glass/Epoxy Composites

It can be observed from figure 8 impact strength of samples which were immersed until saturation (wet state) in seawater and re-dried after saturation point at 25°C and 70°C decreased when compared to the control specimen. Impact strength is considerably influenced by temperatures. This decrease is also valid for the absorbed energy that can be seen in figure 9.



Figure 9. Absorbed Energy Values for Glass/Epoxy Composites

The Charpy impact test results showed that both absorbed energy values and impact strength values are much lower in wet state compared to their dry state for both temperatures. This varied trend can be attributed to several competing phenomena occurring simultaneously. The prolonged sea water immersion causes damages in the composite material due to plasticization and swelling which affect the stress and strain values at failure and the tolerance capacity of the composite is severely affected [17,18].

It can be seen from the figure 10, there is no completely break for control samples, however there is separation between the laminates on the tension side of samples. The wet and re-dried groups of samples that immersed at 25°C indicate similiar fracture type with control group.



Figure 10. Dry Impact Sample a) tension side b) lateral side c) compression side

In contrast to control samples and samples aged 25°C, samples subjected to aging at 70°C either completely or almost completely fracture occured. This type of fracture is valid both wet state and redried state of glass/epoxy composites that can be seen in figure 11 and 12.



Figure 11. Wet Impact Samples Immersed at 70°C a) tension side b) compression side



Figure 12: Re-dried Impact Samples Immersed at 70°C a) tension side b) lateral side

## 4. Conclusion

By this study the effect of seawater aging condition on tensile properties of glass-epoxy composites was investigated experimentally. Results have shown that;

- ➤ The mechanical properties of glass/epoxy composites exposed to hydrothermal ageing decrease generally due to material degradation. Impact strength decreases are higher for wet satate of samples. The decrease of impact properties is higher in the wet samples that immersed at 70°C seawater. That is impact strength decrease is 30,03% at 70°C and 27,3% at 25°C.
- ➢ Furthermore, the decrease of impact strength for re-dried groups are 2,60% and 7,73% for 70°C and 25°C respectively.
- ➤ While there is no complete breakage at control samples and samples immersed at 25°C, complete breakage occured in aged samples at 70°C for both wet and re-dried groups.
- From the Charpy impact test results it can be said that regardless of temperature, immersion in seawater causes mechanical deterioration when compared to their dry state in glass/epoxy composites. Also, as temperature increase, decrease of impact resistances increase for both wet and re-dried state of samples. In the case of submerged samples, the wet state samples has higher mechanical degeneration.

## Acknowledgements

This work was supported by the Gaziantep University Scientific Research Project Governing Unit (BAPYB) with a MF.DT.19.08 project number.

#### References

1. Atas, C., Dogan, A. An experimental investigation on the repeated impact response of glass/epoxy composites subjected to thermal ageing. Composites Part B 75 (2015) 127-134.

The International Conference of Materials and Engineering Technology

- 2. Bradley, W.L., Grant, T.S. The effect of the moisture absorption on the interfacial strength of polymeric matrix composites. Journal of Materials Science 30;5537-5542 (1995)
- 3. Ellyin, F., Rohrbacher, F. Effect of aqueous environment and temperature on glass-fibre epoxy resin composites. Journal of Reinforced plastics and composites (2000) 19(17):1405-1427 doi: 10.1106/AAKT-65LE-R7LG-6XX1
- 4. Aktas A., Uzun I., Sea water effect on pinned-joint glass fibre composite materials 2008 Composite Structures 85/1/59-63 Elsevier
- Catherine, A., Bradley, W.L. Determination of the effect of seawater on the interfacial strength of an interlayer E- glass/graphite/epoxy composite by in situ observation of transverse cracking in an environmental SEM. Composite Science and Technology Volume 57, Issue 8, 1997 1033-1043
- Ray, B.C. Temperature effect during humid ageing on interfaces of glass and carbon fibres reinforced epoxy composites. Journal of Colloid and Interface Science, Volume 298, Isuue 1, 2006 111-117
- 7. Chakraverty, A.P., Mohanty, U.K., Biswal, B.B. Thermal shock behaviour of hydrothermally conditioned e-glass fiber/epoxy composite. Material Research (2012) 1 263
- 8. Rutowska, M., Krasowska, K., Heimwoska, A., Steinka, E. and Janik, H. Degradation of polyurethanes in sea water 2002 Polym. Degrad. Stab.76 233-239
- 9. Mourad, A.I., Abdel-Magid, B.M., EI-Maaddawy, T. and Grami, M.E. Effect of Seawater and Warm Environment on Glass/Epoxy and Glase/polyurethane Composites. Applied Composite Material 17(5), 557-573 (2010)
- Hawa, A., Abdul Majid, M.S., Afendi, M., Marzuki, H.F.A., Amin, N.A.M., Mat, F., Gibson, A.G. Burst strength and impact behaviour of hydrothermally aged glass fibre/epoxy composite pipes. Mater Des 2015; 89(5):455–464.
- 11. Deniz, M.E., Ozen, M., Ozdemir, O., Karakuzu, R., Icten, B.M., Environmental effect on fatigue life of glass-epoxy composite pipes subjected to impact loading. Composites Part B 2013; 44(1): 304-312.
- 12. Deniz, M.E., Karakuzu, R., Seawater effect on impact behaviour of glass-epoxy composite pipes. Composites Part B 2012; 43 . 1130-1138.
- Strait, L.H., Karasek M.L., Amateau M.F. Effects of Seawater Immersion on the Impact Resistance of Glass Fiber Reinforced Epoxy Composites. Journal of Composite Materials, Vol. 26, No. 14/1992
- 14. Vijay, R.B., Shivdarshan, B., effect of moisture absorption on tensile properties of SiC<sub>p</sub> filled glass fiber reinforced epoxy composite material. International Journal of Scientific and Engineering Research Volume 9, Issue 11, ISSN 2229-5518
- 15. ISO 179–181 Plastics Determination of Charpy impact properties Part 1: non-instrumented impact test (2010)
- 16. ASTM D5229 Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials.
- 17. Erkliğ A., Oğuz Z.A., Doğan N.F., Effect of Seawater Aging Condition on Tensile Properties of Glass-Epoxy Composites 6th International Multidiciplinary Studies Congress (2019)
- Chakraverty A.P., Mohanty U.K., Mishra S.C., Satapathy A., (2015). Sea Water Ageing of GFRP Composites and the Dissolved salts. Materials Science and Engineering 75; 012029 doi:10.1088/1757-899X/75/1/012029.

# **ON THE LOW VELOCITY IMPACT BEHAVIOUR OF HYDROTHERMALY** AGED GLASS/EPOXY COMPOSITES

NI E

The International Conference of Materials and Engineering Technology

#### Ahmet ERKLİĞ<sup>1</sup>, Zeynal Abidin OĞUZ<sup>2</sup>,Ömer Yavuz BOZKURT<sup>3</sup>

<sup>1</sup>Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY. <sup>2</sup>Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY. <sup>3</sup>Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY.

#### Abstract

In this study, the low velocity impact behaviour of hydrothermally aged glass/epoxy composite has been studied as experimentally. The kind of fluid was sellected as distilled water and two different aging temperatures that are 25°C and 70°C were used as environmental conditions. The glass/epoxy composites were immersed hydrothermal aging conditions until specimens reach saturation point. The immersed specimens reach saturation at the end of sixth weeks (1000 h). End of the saturation point, samples were conducted to Charpy impact test to be able to define the low velocity impact properties at the impact energy of 15 J. Charpy impact test results of three groups that are dry samples, saturated samples and re-dried samples after saturation point were compared each other. Results have revealed that the damage in glass/epoxy composite under distilled water environment is entirely different. The impact energy and impact strength of three groups have shown changes in their magnitudes with the variation in immersion temperature. That is, from the Charpy impact test result it was seen that material properties degradation occur due to hydrothermal aging. Compared to their non-immersed state, maximum impact stress of immersed specimen have dropped significantly, regardless of temperature. Also, as temperature increase, the reduction rate of maximum stress increase. The decrease rate in aged samples at 25°C is 31.74%, whereas this rate is 45.43% in aged samples at 70°C. Also, the degradation rate of re-dried groups showed similar result that the decrease rate in re-dried samples immersed at 25°C is 29.49%, whereas this rate is 39.22% in re-dried samples immersed at 70°C. The whole results showed that the impact strength reduction in wet samples are greater than the re-dried samples when compared to dry samples.

Keywords: Hydrothermal Aging, Distilled Water, Charpy Impact Test

#### **1. Introduction**

The materials and their properties in the structures which is required high technology change continuously, new material types develop and using areas increase due to the their light weight, high strength, stiffness and resistance to high temperature. The composite materials are this type material class. Polymer-matrix composites used in aerospace and other applications may be exposed to environments usually involving temperature and humidity. In most cases, the deterioration of a composite material during wet aging is caused by a water absorption phenomenon depending on temperature, hygrometric/hydrometric rate and the nature of the composite. The transport of water can be facilitated by diffusion inside the matrix, by imperfections within the matrix, created during the fabrication (micro space, pores or cracks) or by capillarity along the fiber/matrix interface. The damage of polymer matrix composite materials induced by impact load is one of the most critical factors that restrict extensive use of these materials [1].

Many investigations have focused on the influence of water absorption or environmental effects on the various composite system properties.

The effects of thermal ageing and impact loading on tensile properties of adhesively bonded fibre/epoxy composite joints was studied by Akderya at al. [2]. Three different ageing temperatures were determined as -18°C, 25°C and 70°C for a week. At the end of study, it is found that load carrying capacities of specimens were affected by aging temperatures. The load carrying capacities of impacted specimens increase with the effect of thermal ageing process at -18°C in comparison with the specimens under other conditions. At the tensile testing temperatures of -18 and 70°C, reductions have been observed in the break load of the joints and the maximum break load occurred at room temperatures.

Boukhoulda at al.[3] investigated the aging-impact coupling based analysis upon glass/polyester composite material in hygrothermal environment. Impact tests were performed with dry and aged composite plates at 13.6 J and 9 J energy. As a result of the study, it was observed that with aging, the impact strength decreased, the contact time increased, the delamination area decreased, and the flexibility increased in the composite plates.

The topic of the influence of long term water immersion ageing on impact damage behaviour and residual compression strength of glass fibre reinforced polymer (GFRP) has been investigated by Berketis at. al. [4]. At the end of study, researchers have observed that the water immersion ageing affected the microstructural integrity of the laminated plates causing numerous defects and the impact tests caused multiple through thickness damage mechanisms depending on the immersion time leading to reduced elastic properties of the GFRP.

Becky [5] and his co-workers studied the combined effects of load, moisture, and temperature on the properties of E-glass/epoxy composites. They developed a method to evaluate the combined effects of mechanical and environmental conditioning on fibre reinforced polymer composites. The results emphasised that the reduction in modulus was due to the plasticization of the absorbed water.

Erklig [6] at. al. studied the effect of distilled water aging condition on tensile properties of glass-epoxy composites. From the tensile test result it was seen that material properties degradation occur due to hydrothermal aging. Compared to their non-immersed state, maximum tensile stress of immersed specimen have dropped significantly, regardless of temperature. Also, as temperature increase, the reduction rate of maximum stress increase.

The effect of aqueous environment and temperature on glass-fibre epoxy resin composites was studied by Ellyin at. al. [7]. At the end of study they observed that the degree of damege strongly depends on the immersion temperature. The threshold strain for the matrix crack initiation increases when the laminated are immersed in ambient water in comparison to that of a dry environment.

The topic of interface damage study of hydrothermally aged glass-fibre-reinforced polyester composites was investigated by Gautier at. al. [8]. The decrease of interlaminar shear strength (ILSS) was explained by the researcher that mainly by interfacial debonding, induced by di  $\Box$  erential swelling, and by osmotic cracking at the interphase but the matrix also contributes to the decrease.

This study focuses on the Charpy impact behavior of glass/epoxy composite kept in distilled water at two different temperatures (25°C and 70°C). The glass/epoxy composites were immersed hydrothermal aging conditions until specimens reach saturation point that occurs at 1000<sup>th</sup> h. After saturation, the composite samples were removed the ambient conditions. Charpy impact test applied to three groups. These are wet group, re-dried group and dry group. The effects of temperature on the impact strength of glass/epoxy were investigated in accordance with experimental results.

# 2. Experimental Procedure

#### **2.1 Material Production**

Twelve ply laminates woven S-glass fibre/epoxy composite plates were produced by method of vacuum -assisted resin-infusion method (VARIM). An epoxy system consisted of MGS L 285 resin and MGS H 285 hardener was used as matrix material in manufacturing with mass ratio 100:40. The properties of S-glass woven fabric are 202 g/m2, 3000-5000 MPa,72-82 GPa and 0.15 mm respectively for density, tensile strength, elastic modulus and thickness The production process was carried out on a vacuum device and control unit-connected heater table. At the first step, a release film was unrolled on the heater table then twelve-layer glass-fibre fabrics and peel ply were laid on top of it. A distribution network was embedded in order to gain quick and homogenous resin spread. A vacuum-bagging film was pasted from the edges by a sealant tape to wrap the layers. A vacuum duct and a resin inlet were connected to the vacuum bag's two opposite sides with air tight (Fig.2). Making sure that there is no leakage in the vacuum bag, the mixture of resin and fast hardener was impregnated. The leakage was controlled from vacuum bag that is the pump vacuum was kept constant as 0.12 MPa. Therefore, the mixture diffused into the layers gently. After the impregnation process was fully completed, the valves of resin inlet and vacuum tubes were turned off in order to keep the pressure constant in the vacuum bag. Then the laminate was left for curing for 2 h at the temperature 45°C. Once the curing was done, the laminate was left for cooling at room temperature throughout 12 hours.

The International Conference of Materials and Engineering Technology



Figure 1. S-glass woven fabric



Figure 2. Production of glass fibre/epoxy composite plates by VARIM

## **2.2 Specimen Preparation**

Charpy impact test specimens were cut with the help of the CNC rooter according to ISO 179/92 [9] as can be seen in figure 3. The samples that would be immersed water before test was not sandpapered due to the sandpaper process may prevent water absorption. Instead of sanding the samples, fringes of samples were cut by using an electronic cutter. After cutting of fringes, samples were numbered and prior to placing specimens into the environmental chambers all the specimens were dried on the vacuum tables with 50°C until take a constant weight to make samples completely dry.



By this way samples became moisture free. Before immersed samples to distilled water, samples were weighed to obtain the dry weight of them. The thickness of samples were taken by a digital caliper and recorded.



Figure 3. Cutting Process of Glass/Epoxy Plates with CNC rooter

#### 2.3 Hydrothermal Aging Process

Hydrothermal aging process has been done by cabins made of heat resistant glass and silicon were prepared for use.  $25^{\circ}$ C temperature was used for room temperature, and  $70^{\circ}$ C temperature was used for hot condition. The hydrothermal aging was carried out based on ASTM D5229/D5229M-14 [10]. According to standard a conditioning chamber is required that shall be capable of maintaining the required temperature to within  $\pm 3^{\circ}$ C. To be able to obtain constant heat inside of the whole cabins, adjustable resistance has been installed to cabins. Due to the position of resistance in the cabins, there are different temperatures in the cabin at different points. To avoid this problem, a small circulation pump was placed to cabin. By this way the change of heat could be achieved as nearly zero. The change of heat inside of the cabin may lead to misinterpretation of water absorption of composite laminates due to it changes the density of fluid and thus fluidity of water. All equipments about cabin are shown in figure 4. The temperature control was done every day by a digital thermometer.



Figure 4 : Aging Cabin

The groups are named as W25 for specimens at 25°C, W70 for specimens at 70°C. For each groups of composite, ten specimens were prepared to immerse hydrothermal aging environments, five of them for to saturate, and five of them for dry (non-immersed).


## 2.4 Impact Test

Charpy impact test was carried out to evaluate of hydrothermal aging effect on low velocity impact behaviour of glass/epoxy composites. The test was performed per ISO 179/92 standard [9]. Impact tester Köger 3/70 device with pendulum energy of 15 J shown in Fig.5 was used and tests were conducted at room temperature. Charpy impact test device consists of a pendulum with a mass attached to a rotating arm connected to the device body. Pendulum falls from a certain height and strikes the test sample at the anvil and sample absorbs part of pendulums kinetic energy. 55 x 10 mm unnotched test samples were used and the impact load was applied on the flat surface of the samples. Five samples were used in tests for each produced composite types. Average absorbed energy values were calculated using five samples test results. The absorbed impact energy and impact strength of material were calculated from Eq.1 and 2 as follows:

aterials and Engineering Technology

$$K = \frac{W}{A} \qquad (1)$$

Where W is the absorbed energy (J), A is the area (m<sup>2</sup>) and K is the impact resistance (J/m<sup>2</sup>) <sub>2</sub>

 $W = E_1 - E_2 \quad (2)$ 

Where  $E_1$  and  $E_2$  are initial and final potential energies respectively.



Figure 5. a) Charpy impact test set-up, b) Composite test samples.

## **3.Results**

To be able to evaluate the low velocity impact behaviour of hydrothermaly aged glass/epoxy composites, Charpy impact test was conducted to three groups with two different temperature that are 25°C and 70°C. The first group is control group and this group was immersed any hydrothermal condition and tested as dry state. The second group was immersed at 25°C and 70°C until saturation point and tested as wet state. The last group was immersed distilled water as second group. The last group re-dried after saturation point and was tested as dry state. Five specimens of the glass/epoxy composite materials were conducted to Charpy impact test for each groups to obtain the control impact properties which include impact strength, and absorbed energy at failure. Average of five samples was evaluated finally for each conditions. The schematic illustration of impact test of glass/epoxy composites is shown in figure 6.



Figure 6. Impact Resistance of Glass/Epoxy Composites

It is clear that distilled water immersion of glass/epoxy composites shows negative affect on impact resistance regardless of temperature. Compared to the control group, samples immersed at both temperatures show lower impact strength results when tested in wet form than re-dried samples. The decrease of absorbed energy for all hydrothermal conditions shows similiar trend with impact resistance as shown in figure 7.



Figure 7. Absorbed Energy for Glass/Epoxy Composites



Materials and Engineering Technology

As shown in Figure 8 and Figure 9, the temperature also has an effect on the fracture type of the glass/epoxy composites in the Charpy impact test applied at the end of hydrothermal aging. Unlike the control group and samples immersed at 25°C (Figure 9), the samples that immersed at high temperature glass/epoxy composites were completely broken (Figure 10). This type of fracture shows that the high temperature causes poor matrix/resin adhesion. In other words, high temperature disrupts the matrix/resin bond.



**Figure 8**. Types of fracture a) tension side of dry sample b) lateral side of dry sample c) compression side of dry sample d) tension side of W25 sample b) lateral side of W25 sample c) compression side of W25 sample



Figure 9. a) tension side of W70 (wet) b) compression side of W70 (wet) c) lateral side of W70 (redried) d) tension side of W70 (re-dried)

## 4.Conclusion

Hydrothermal effect on the low velocity impact behaviour of glass/epoxy composites was investigated in this study experimentally. Two different temperatures with distilled water were used as hydrothermal aging condition. The Charpy impact test results revealed the following conclusions.

 Experimental results showed that hydrothermal aging has a direct effect on the impact strength of glass/epoxy composites.

The International Conference of Materials and Engineering Technology

- The greatest decrease in impact strength is in the wet-tested group that immersed in 70°C with a rate of 45,43 percent, while the least decrease is in the re-dried group after being exposed to aging at 25°C with 29,49 percent.
- ✤ Results showed that for both temperatures, decrease of impact strength is higher when the samples are tested as wet state. While the decrease of impact strength of wet state tested samples immersed at 25°C in distilled water is 31.74%, this ratio is 29.22% for re-dried group. Also this trend is similiar for 70°C condition. That is, the decrease of impact strength of wet state tested samples immersed at 70°C in distilled water is 45.43%, this ratio is 39.23% for re-dried group.
- All this results show that as temperature increase, the decrease of impact resistance increase. This is valid for both wet form and re-dried form.
- ✤ While there is no complete breakage at control samples and samples immersed at 25°C, complete breakage occured in aged samples at 70°C for both wet and re-dried groups.

#### Acknowledgements

This work was supported by the Gaziantep University Scientific Research Project Governing Unit (BAPYB) with a MF.DT.19.08 project number.

## References

- Andrew, J.J., Srinivasan, S.M., Arockiarajan, A., Dhakal, H.N. Parameters influencing the impact response of fiber-reinforced polymer matrix composite materials: A critical review. Composite Structures 224 (2019) 111007
- 2. Akderya, T., Kemiklioğlu, U., Sayman, O., Effects of thermal ageing and impact loading on tensile properties of adhesively bonded fibre/epoxy composite joints. Composites Part B 95,117-122 (2016).
- 3. Boukhoulda, F.B., Guillaumat, L., Lataillade, J.L., Adda-Bedia, E., Lousdad, A. Agingimpact coupling based analysis upon glass/polyester composite material in hygrothermal environment, Materials and Design 32, 4080–4087 (2011)
- Berketis, K., Tzetzis, D., Hogg, P.J. The influence of long term water immersion ageing on impact damage behaviour and residual compression strength of glass fibre reinforced polymer (GFRP) Materials and Design 29 (2008) 1300–1310
- Beckery, A.M., Saed, Z., Katrina, G., Marcus, S., The combined effects of load, moisture and temperature on the properties of E- glass/epoxy composites. Composite Structure 2005; 320-326



The International Conference of Materials and Engineering Technology

- Ellyin F., Rohrbacher C., Journal of Reinforced Plastics and Composites. 2000 19:1405. doi: 10.1177/073168400772678518
- 8. Gautier, L., Mortaigne, B., Bellenger, V. Interface damage study of hydrothermally aged
- 9. glass-fibre-reinforced polyester composites. Composites Science and Technology 59 (1999) 2329–2337
- 10. ISO 179–181 Plastics Determination of Charpy impact properties Part 1: non-instrumented impact test (2010)
- 11. ASTM D5229 Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials.
- 12. Ellyin, F., Maser, R. Environmental effects of the mechanical properties of glass fiber epoxy composite tubular specimens. Compos Sci ; 64 (12): 1863–1874 .(2004)
- 13. Gautier, L., Mortaigne, B., Bellenger, V. Interface damage study of hydrothermally aged glass-fibre-reinforced polyester composites. Compos Sci Technol;59:2329–37 (1999)



# COMPARISON OF AN INTERVAL TYPE-2 FUZZY SETS AND AHP METHODS FOR MATERIAL SELECTION PROBLEM ON LITHIUM-ION BATTERIES

# AHMET AKTURK<sup>\*1</sup>, SEDA TURK<sup>2</sup>

<sup>1</sup>Igdir University, Engineering Faculty, Mechanical Engineering Department, Igdir, TURKEY. <sup>2</sup>Igdir University, Engineering Faculty, Industrial Engineering Department, Igdir, TURKEY.

#### Abstract

Lithium-ion batteries have become one of the most commercially preferred energy storage devices because of their high energy density, low self-discharge rate, and the ability to be cycled many times with slow capacity fading in comparison with other rechargeable batteries. They have been applied on a wide variety of electrical devices and systems such as consumer electronics, power tools, electric vehicles and aerospace equipment. The characteristics of Li-ion batteries are mainly determined by the materials used for its components which can be categorized into four parts: anode and cathode electrodes, separator, and electrolyte. Over the last decade, there has been a significant increase in the number of studies evaluating battery performance based on various materials used in each battery component. However, few attempts have been made to evaluate materials of Lithium-ion batteries. Thus, in this study, we aim to evaluate different materials for cathode electrode in terms of four main criteria: cost, performance, safety and service life using two methods; AHP and interval type-2 fuzzy sets. It is shown that more reliable results are obtained for selecting the best cathode material of Li-ion battery and based on comparison of two methods, same rank is achieved for both approaches.

Keyword: Material selection problem, AHP, Fuzzy Logic, Interval type-2 fuzzy sets, Li-ion battery.

## **1. Introduction**

In view of the current and predictable energy needs, the utilization of lithium-ion batteries has become more essential because of its accommodation of high energy and power density, with a low selfdischarge rate [1]. In addition, one of remarkable features is able to work under wide temperature range of operation [2]. The high energy efficiency of Li-ion batteries also makes them the leading source of energy storage in many application areas such as renewable sources, the electronic device and electric vehicle industry [3]. Thus, researches on Li-ion batteries are become a critical and essential issue in both industry and government agencies [1].

Li-ion batteries comprise of the anode and cathode electrodes, separator, and electrolyte and these components determine the characteristics of Li-ion batteries [4]. In order to increase the performance of a Li-ion battery along with a decrease on cost, most of researches have focused on electrode materials which provide higher rate capability, higher charge capacity [1].



There are several commercially available electrode materials that can be found in different works which compare them based on their performance, power, weight, energy storage capability, volume, life time and cost. However, in this study, we will focus on five cathode electrodes namely; lithium cobalt oxide (LCO), lithium manganese oxide (LMO), lithium iron phosphate (LFP), nickel manganese cobalt (NMC) and, nickel cobalt aluminium (NCA).

Due to the complexity of the material selection problem, there has been a range of studies applying multi-criteria decision making methods which provide a structure to solve a problem considering multiple criteria at the same time. Panday and Bansal addressed a battery selection problem for hybrid electric vehicle applications using TOPSIS and VIKOR [5]. Kaa et al. pointed out that the factors affecting technology success in the residential grid storage market could be related to success in battery technologies. The problem is solved using the best worst method (BWM) evaluating five battery materials: lithium, lead, nickel, sodium and flow batteries [6]. Sangwan and Jindal proposed an integrated fuzzy multi-criteria decision model to evaluate different recycling alternatives for Lithium-Ion batteries to show the importance of recycling processes [7]. Based on the literature, it is clearly revealed that although multi-criteria decision making methods are used in many engineering applications to select the most suitable materials, few attempts have been made to evaluate materials of Lithium-ion batteries and to the authors' knowledge, there is no research found on material selection on cathode materials with considering variety of criteria selected at the same time.

The aim of this study is to select the best materials for cathode electrodes of Lithium-ion batteries used in electric scooter battery system using both AHP and interval type-2 fuzzy sets. Due to easy to understand and successfully applied to different decision making problems in literature, AHP is selected to solve this problem. Because of the uncertain nature of material selection problems of Lithium-ion batteries, in the study, a fuzzy approach is preferred as a second method to apply to the problem evaluating alternatives proposed for cathode electrodes. First, we solve the problem using AHP and then in order to see the impact of uncertainties revealed into the linguistic evaluation of decision makers, the same problem is solved using interval type-2 fuzzy sets. Finally, the results of two methods are compared to conclude with more reliable results.

The rest of the study is as follows. Section 2 gives basic concepts of Lithium-ion batteries and an introduction to methods used in this study. In Section 3, the definition of criteria and numerical experiments are provided to introduce the case study with a problem definition. Section 4 presents results and discussion. Finally, Section 5 concludes the study and discusses some potential future research directions.

## 2. Preliminaries

This section introduces basic concepts of lithium-ion batteries and the fundamentals of the techniques used in this work with an overview of related work in the area.

## 2.1 Basic concepts of lithium-ion batteries

A Lithium-ion battery is a general term used to refer to the batteries of various chemistry with different performance and characteristics [8]. Lithium-ion batteries composed of a positive (cathode) and negative (anode) electrodes, and ionically conductive and electrically insulating an electrolyte and a separator. The electrolyte containing lithium conductive salt connects the positive and negative electrodes. The anode and the cathode are electrically isolated by the separator consisting of a microporous polymer membrane.

This membrane allows the movement of lithium ions between the anode and the cathode, but does not allow electron movement. During the charge / discharge process, aluminium acts as a current collector on the cathode side and copper on the anode side [9, 10]. The operating principle of lithium-ion batteries is as the following: During charging lithium-ion batteries, two electrodes are first connected to an external power source. Thus, the electrons released from the cathode move towards the anode through the external circuit, the positively charged lithium ions move through the electrolyte towards the anode and intercalation of lithium ions into active material takes place in anode side. After the intercalation process is completed, the external electrical energy is chemically stored in the battery [10]. The discharge process is in the opposite direction of the charging process. Lithium ions de-intercalated from the anode move through the electrolyte and electrons move through the outer circuit towards the cathode. Due to the electron movement, the external circuit becomes a usable electrical circuit [9, 10].

The International Conference of Ma

aterials and Engineering Technology

Although this study focuses on the evaluation of fully commercialized cathode materials, before clear understanding of the cathode, it is useful to give brief information about anode and electrolyte materials used in Lithium batteries. The anode is the negative terminal of the battery cell and is usually coated on current collectors such as aluminium and copper. Today, a mixture of graphite and soft or hard carbons are generally used to form anode for lithium-ion batteries [8]. One of the outstanding features of the graphite anode is that it has a specific capacity of  $300-350 \text{ mAhg}^{-1}$  [11]. Another remarkable anode material is lithium titanite due to its low temperature operation and high power density. Batteries using this anode material are called lithium-ion titanite cells (LTO). The LTO cell operates between -40 and + 60 Celsius and has the advantage of having a good power density of 1400  $WL^{-1}$ , but the nominal voltage is between 2.2 V and 2.3 V and is low. Another vital part of lithium batteries is the electrolyte, which plays a crucial role in electrochemical behavior and ion transfer between the two electrodes [12]. In view of their advantages and disadvantages, liquid, gel and solid electrolytes are selected by the manufacturers for different purposes. Although liquid electrolytes cause safety concerns such as solid electrolyte interphase (SEI), dendrite growth, leakage and thermal runaway, it has high ionic conductivity, making it attractive. Compared to liquid electrolytes, the gelpolymer electrolytes have lower ionic conductivity but offer improved safety. Due to the suppression of dendrite growth and higher electrochemical and thermal stability, solid electrolyte is safer than the other two electrolytes. The disadvantage of solid electrolyte is that it has lower ionic conductivity compared to liquid and gel electrolyte [12, 13].

Cathode is an electrode that accepts electrons during cell discharge [14]. In order to benefit from the advantages of different chemistries and to achieve significant performance results, cathode materials containing different chemistries in various proportions are preferred by cell manufacturers. In this study, five of the most commercially accepted cathode materials are evaluated to be used in electric scooter battery system. These cathode materials can be listed as lithium cobalt oxide (LCO), lithium manganese oxide (LMO), lithium iron phosphate (LFP), nickel manganese cobalt (NMC) and, nickel cobalt aluminum (NCA) [8]. Due to good cycling performance, low self-discharge rate, high theoretical specific capacity of 274 mAhg<sup>-1</sup>, high theoretical volumetric capacity of 1363 mAhcm<sup>-3</sup>, and high discharge voltage, the layered LiCoO<sub>2</sub> has been a revolutionary part of lithium-ion batteries used in portable electronics for decades. However, due to chemical and thermal instability, high cost and safety concerns, the use of this cathode material in electrical vehicles and energy storage from renewable energy sources is quite low. In addition, only 50% of the theoretical capacity can be utilized using this cathode material [15, 1, 16].



Furthermore, the LCO cathode experiences severe capacity fading at high current rates and deep cycles. The main reason why LCO cathode material is expensive is the high cost of Cobalt. Because of using  $LiCoO_2$  cathode with low thermal stability, the cell may be at risk of thermal runaway and consequently explosion due to exothermic oxygen release after heating the cell above certain point [1]. In order to achieve a more affordable cost and higher energy density, efforts have been made to replace cobalt with cheaper nickel. As a result, a  $LiNiO_2$  cathode material with similar theoretical specific capacity of 275 mAhg<sup>-1</sup>, higher reversible capacity of 200 mAhg<sup>-1</sup>, and layered structure was formed [1, 9, 14]. During the production of NCO, Nickel ions substitute some of the lithium positions. Ni<sup>2+</sup> ions, which have similar radii as the Li<sup>+</sup> ions, are the main cause of this mixed occupation, and the Ni<sup>2+</sup> ions block lithium diffusion, resulting in an irreversible capacity loss in the cathode material. Basically, LNO is more thermally unstable than LCO due to Ni<sup>3+</sup> ions and the safety concerns as a result of oxygen release persist in this cathode material [9].

The International Conference

It was found that the partial substitution of Ni with Co is a more effective method, thereby avoiding the negative effects of complete mixing. Adding a small amount of Al to this partial substitution both improves electrochemical performance and leads to a more thermally stable cathode. LiNi<sub>0.8</sub>Co<sub>0.15</sub>Al<sub>0.05</sub> (NCA) cathode material formed by the addition of a small amount of Al has become commercially widespread. NCA batteries manufactured by Panasonic are used in Tesla electric vehicles. Compared to the other four batteries, NCA batteries have a high usable discharge capacity of 200 mAhg<sup>-1</sup>. However, Due to the formation of SEI and micro-crack growth at the grain boundaries, they suffer from significant capacity loss even at temperatures between 40 and 70 Celsius degree [1]. The reason for using manganese (Mn) in the batteries is that it is cheap, environmentally friendly, has high electronic and ionic conductivity, excellent rate capability and offers an ideal level in terms of safety [15]. Spinel LiMn<sub>2</sub>O<sub>4</sub> cathode has been considered one of the serious options by the electric vehicle industry and has been used in Chevy Volt. Disadvantages of batteries using  $LiMn_2O_4$  cathode are that it has a severe capacity loss at temperatures exceeding 55 Celsius degree, it has a short cycle life [15, 1]. In addition, it has a gravimetric energy density of 410-492 Whkg<sup>-1</sup>, which is less than the energy density of other four cathode materials [14]. The short cycle life is mainly because of irreversible reactions with electrolyte, oxygen loss due to de-lithiation of  $LiMn_2O_4$ , dissolution of Mn and formation of Li<sub>2</sub>Mn<sub>2</sub>O<sub>4</sub> at high discharge rates [1].

Oxygen production does not occur in cells where LiFePO<sub>4</sub> cathode material is used, even at high temperatures and as a result of fully decomposition. For this reason, this cathode material provides a vital safety advantage with the lowest heating rate during thermal runaway. In LFP cells, thermal runaway is dominated by the anode and electrolyte pair [17]. LiFePO<sub>4</sub> with olivine structure is nontoxic, has excellent safety and is cost-effective. Having such features made LFP batteries one of the most prominent candidates for transportation applications. The LFP battery manufactured by A123 was used in Chevrolet Spark EV for transportation purposes [18]. On the other hand, low ionic and electronic conduction are an obstacle to be overcome for LiFePO<sub>4</sub> cathode materials. Electronic and ionic conductivity are generally enhanced by making LiFePO<sub>4</sub> as nanoparticles and coating these particles with conductive carbons. Nanoparticle making and carbon coating process creates extra processing costs [15]. Current batteries using LiFePO<sub>4</sub> cathode are only able to deliver garvimetric energy density of 90-110 Whkg<sup>-1</sup>, and in this respect, it lags behind competitors like NCA and NMC [19]. Also, LFP has lower energy density than the other four chemistries on the market which means larger volume is needed to fit battery pack into vehicles [17]. Although different NMC cathode materials can be formed using cobalt, nickel and manganese in various proportions, the most common NMC in the market is in the form  $\text{LiNi}_{0.33}\text{Co}_{0.33}\text{Mn}_{0.33}\text{O}_2$  (NMC-111) [1].

Technology



Compared to LiCoO2, The LiNi<sub>0.33</sub>Co<sub>0.33</sub>Mn<sub>0.33</sub>O<sub>2</sub> cathode, which offers improved thermal, structural and chemical stability and has more satisfactory electrochemical properties, has become an attractive option [14]. NMC batteries have good cycle stability even at 50 Celsius. As it has an average specific capacity of 160-170 mAhg<sup>-1</sup> and 1000-2000 charge/discharge cycles, the interest in NMC batteries increases over time and NMC has a growing market share [14, 19]. Cylindrical and prismatic NMC batteries produced by different companies are used in the electrical vehicles of various brands such as Nissan, Renault, Chevrolet, Honda and Volkswagen [18]. Based on literature, it could be said that each material has some advantages and disadvantages. Thus, in order to evaluate them, considering several criteria at the same time could be the best way to provide fair evaluation.

he International Conference of Materials and Engineering Technology

## 2.2 Multi-criteria decision making

Conflicting criteria are evaluated in order to make decisions with a consideration of different objectives such as minimizing the cost of Li-ion battery while maximizing specific capacity. The multi-criteria decision making (MCDM) is widely used method to evaluate explicitly multiple conflicting criteria in the discrete decision spaces. In a MCDM problem, alternatives proposed are examined to compare, rank and order them based upon criteria [20]. In literature, a number of MCDM methods can be found as Analytic Hierarchy Process (AHP), Analytic Network Process (ANP), Technique for Order Preference by Similarity to Ideal Solution (TOPSIS) and Multi Attribute Utility Technique (MAUT), Fuzzy set theory. Analytic Hierarchy Process (AHP) is one of the most outstanding MCDM approaches to solve complex decision problems using hierarchical levels along with a process of paired comparison [21]. However, this method is not capable where imprecise human thinking is present in linguistic judgment [22, 23]. In order to cope with these types of uncertainty, fuzzy sets are preferred by researchers [24, 25]. Thus, in this study, we used both AHP and interval type-2 sets to handle a material selection problem for Li-ion batteries.

## 2.2.1 Analytic Hierarchy Process

Saaty introduced the AHP method which compares alternatives in a hierarchical structure with respect to perception and thinking of decision makers [26]. AHP uses pairwise comparison of elements in the same hierarchy for both criteria and alternatives giving different weight for each element [27]. In literature, the AHP method has been used in many different real-word problems. In addition, some researchers have applied in material selection problems owing to a number of conflicting criteria affecting decision makers. For instance, Dweiri and Al-Oqla stated that the AHP method is useful to evaluate materials under competition on quality and cost [27]. In addition, Kiong et al. addressed a material selection problem in screw manufacturing to minimize environmental impacts using AHP and pairwise comparison among materials provided useful information to decrease harmful environmental impact of screw manufacturing [28]. Kühn et al. proposed an AHP approach to select materials for automated dry fiber placement and this method helps to minimize cost and preparation time decreasing iterative manufacturing trials [29]. Through the review of several papers chosen, it is clearly seen that AHP is applied into a number of material selection problems and it is capable of providing useful information for decision makers. Fort this reason, in this work, an AHP approach is used to deal with cathode materials for Li-ion batteries. In addition, due to ambiguity in subjective judgments and the lack of information, the same problem proposed is solved using interval type-2 fuzzy sets and results of two methods are compared in order to obtain more reliable result.



## 2.2.2 Interval Type-2 Fuzzy Sets

Zadeh introduced fuzzy set theory which allows varying degrees of membership values in a given set to consider both tangible and intangible knowledges [30]. Also Zadeh proposed type-2 fuzzy sets to handle problems which type-1 fuzzy sets cannot cope with [31]. According to Hagras, the wider coverage of uncertainties are provided using type-2 fuzzy sets while using less rules [32]. However, due to the computational complexity on type-reduction and defuzzification processes of type-2 sets, the most researchers have focused on interval type-2 fuzzy sets [33]. Thus, in this study, we preferred to use interval type-2 fuzzy sets.

The International Conference

For type-2 fuzzy systems, a membership function (the degree of membership) shown as  $\mu_{\tilde{A}}(x, u)$  characterized as fuzzy set  $\tilde{A}$  and this fuzzy sets are shown as:

$$\tilde{A} = \{ (x, u), \ \mu_{\tilde{A}}(x, u) | \forall x \in X, \forall u \in J_x \subseteq [0; 1] \}$$

$$\tag{1}$$

where  $x \in X$  and  $u \in J_x \subseteq [0; 1]$  in which  $0 \leq \mu_{\tilde{A}}(x, u) \leq 1$ . If all  $\mu_{\tilde{A}}(x, u) = 1$  then  $\tilde{A}$  is named as an interval type-2 fuzzy set [34]. In addition, membership functions can have different shapes such as triangular, trapezoidal, Gaussian and in this study, trapezoidal interval type-2 fuzzy sets (IT2FSs) are used and shown as follows

$$\tilde{A}_{i} = \left(\tilde{A}_{i}^{U}, \tilde{A}_{i}^{L}\right) = \left(\left(\tilde{a}_{i1}^{u}, \tilde{a}_{i2}^{u}, \tilde{a}_{i3}^{u}, \tilde{a}_{i4}^{u}; h_{1}(\tilde{A}_{i}^{U}), h_{2}(\tilde{A}_{i}^{U})\right), \left(\tilde{a}_{i1}^{l}, \tilde{a}_{i2}^{l}, \tilde{a}_{i3}^{l}, \tilde{a}_{i4}^{l}; h_{1}(\tilde{A}_{i}^{L}), h_{2}(\tilde{A}_{i}^{L})\right)\right)$$
(2)

where  $\tilde{A}_{i}^{U}$  and  $\tilde{A}_{i}^{L}$  are type-1 fuzzy sets while  $\tilde{a}_{i1}^{u}, \tilde{a}_{i2}^{u}, \tilde{a}_{i3}^{u}, \tilde{a}_{i4}^{l}, \tilde{a}_{i2}^{l}, \tilde{a}_{i3}^{l}$  and  $\tilde{a}_{i4}^{l}$  represent the reference points of the interval type-2 fuzzy set,  $\tilde{A}_{i}$  [34]. The height of each constituent membership function shown as  $h_{k}(\tilde{A}_{i}^{U}), h_{k}(\tilde{A}_{i}^{U})$  for  $1 \le k \le 2$ 

is assumed to be equal to 1. Thus, it is not explicitly defined. Algebraic operations used in this work are addition and multiplication as depicted respectively as follows [34]:

$$\tilde{A}_{1} \bigoplus \tilde{A}_{2} = \left(\tilde{A}_{1}^{U}, \tilde{A}_{1}^{L}\right) \bigoplus \left(\tilde{A}_{2}^{U}, \tilde{A}_{2}^{L}\right)$$

$$\tilde{A}_{1} \otimes \tilde{A}_{2} = \left(\tilde{A}_{1}^{U}, \tilde{A}_{1}^{L}\right) \otimes \left(\tilde{A}_{2}^{U}, \tilde{A}_{2}^{L}\right)$$
(3)
(4)

## 3. Methodology

This study aims to provide an evaluation of five cathode materials for Li-ion batteries namely  $LiCoO_2$  (LCO),  $LiMn_2O_4$ (LMO),  $LiMn_2O_4$ (LFP),  $LiNi_{0.33}Mn_{0.33}Co_{0.33}O_2$ (NMC),  $LiNi_{0.8}Co_{0.15}Al_{0.05}$  (NCA) with respect to 9 criteria under 4 main criteria as shown in Table 1. The performance of 5 alternatives are investigated using two different methods; AHP and interval type-2 fuzzy sets. In AHP, the problem is solved considering 9 criteria that have same importance weight for decision maker while in fuzzy approach, the problem is examined using 4 main, 9 sub-criteria. Thus, although thermal abuse and thermal stability is similar in general, in this study, thermal stability is considered under safety while thermal abuse is examined under service life in order to obtain more reliable results.

Main Criteria	Abb.	Sub-criteria	Abb.
Performance	$M_1$	Specific Capacity	$C_1$
		Gravimetric Energy Density	$C_2$
		Discharge (C- Rate)	C <sub>3</sub>
		Working voltage	$C_4$
Cost	$M_2$	Cell Cost	C5
Safety	$M_3$	Cathode Safety	$C_6$
		Thermal Stability	$C_7$
Service Life	$M_4$	Cycle life	$C_8$
		Thermal Abuse	C <sub>9</sub>

Table 1. Main and Sub-criteria used for evaluation of alternative cathode materials

## 3.1 Definition of Criteria

**Table 2.** Criteria for Cathode materials and their changes based on five materials namely  $LiCoO_2$  (LCO),  $LiMn_2O_4$ (LMO),  $LiMn_2O_4$ (LFP),  $LiNi_{0.33}Mn_{0.33}Co_{0.33}O_2$ (NMC),  $LiNi_{0.8}Co_{0.15}Al_{0.05}$  (NCA), respectively.

Criteria	LCO	LMO	LFP	NMC	NCA
Specific Capacity [14]	140	100-120	150-170	160-170	180-200
Gravimetric Energy Density [2	14] 546	410-492	518-587	610-650	680-760
Discharge (C- Rate) [19]	<1C	1C	1C-3C	0.7-1C 1	С
Working Voltage [9]	3.9	4.1	3.4	3.7	3.7
Cost [35,36]	High	Low	Low	Low	Low
Safety [15,9]	Very Low	Medium	Very High	High	Low
Thermal Stability [15,37]	Very Low	Medium	High	Medium	Low
Cycle life [37,19]	500-1000	300-700	1000-2000	1000-2000	) 500
Thermal Abuse [5, 37]	High	Low	Low	Medium	High

For decades, there has been a growing interest on lithium-ion battery systems. Since the first launch of lithium batteries, it has always been a key objective to obtain high-performance, inexpensive, perfectly safe and durable batteries. Obtaining desirable cathode materials and using these cathodes in the cell play a significant role to achieve this key objective. Nowadays, cathode materials consisting of different chemistries, having various performances and varying costs are frequently encountered in commercial lithium ion batteries as seen in Table 2. Therefore, it has become extremely important to select proper lithium ion batteries that use different cathode materials depending on the place of use. In this study, in order to select cathode suitable for electric scooter battery system, nine criteria are taken into consideration as explained in the followings:

1. Specific Capacity; is defined as the amount of charge the cathode contains per unit weight and is expressed as mAhg<sup>-1</sup> or Ahkg<sup>-1</sup>. Also, it is a measure of how long the energy can be supplied to a device under a certain discharge current. Materials with higher specific capacity are often preferred because they provide energy for longer [14].

Technology

e International Conference of Materials and Engineering

- Gravimetric energy density; is described as the energy density of a material's unit weight and expressed as Whkg<sup>-1</sup>. Energy density is the common product of specific capacity and voltage. Cathode materials generally have a lower specific capacity than graphite, the most commonly used anode material and the energy density of the cell is determined by the electrode having a lower energy density. Therefore, the main determinant of the energy density of the cell is cathode. As a result, selecting cathode material with a high gravimetric energy density is extremely important as it will increase the energy density of the cell and provide more energy per unit weight [14].
- 3. Discharge (C-Rate); is a term used to refer to the rate at which a battery can discharge all its energy or power. The 1C discharge rate delivers the nominal capacity of the battery within 1 hour. In other words, 1C rate means that a battery can discharge in one hour and 3C rate means that the same battery can discharge in 20 minutes (60 min / 3C = 20 min). High C-rate is a desirable feature since cells with high C-rate rates can provide higher discharge current [8].
- 4. Working Voltage (V); is the potential difference between the anode and cathode during the operation of the cell and it is called either working voltage or operating voltage. In addition, it varies with the state of charge. Since the energy density of the cell is determined by capacity and voltage, it is important to select cathode material that can provide higher voltage [35].
- 5. Cell Cost; is the sum of cost of battery components and processing cost. Obviously, the cost of cells using various cathode chemistries will be different than each other. In addition, some cathode materials need to undergo extra processing to perform at the desired level, which results in extra processing costs [15]. Low-cost cathode materials are preferable to create cheaper cells.
- 6. Cathode Safety; concerns include thermal instability, chemical instability, toxicity, and short circuit caused by various reasons. The safety is one of the key factors in determining the areas where different lithium ion batteries can be applied. As a component of the cell, the choice of safer cathode material is advantageous for whole cell [16].
- 7. Thermal Stability; specifies to what extent the battery loses its stability with increasing or decreasing temperature. Thermal instability of the cathode material causing overheating, thermal runaway and even explosion can be said to be the main safety problem in the cell. Therefore, the thermal properties of the cathode material must be carefully examined to ensure that the cell remains thermally stable as desired [16].
- 8. Cycle Life; is defined as the total number of cycles that can the battery reach. One time discharging and then charging the battery is called a cycle. The cycle can be carried out at various power and voltage ratings or at constant charge or discharge rates as required. Cathode materials capable of reaching higher number of cycles will lead to cells with long cycle life [8].
- 9. Thermal Abuse; relates to thermal stability and examines how cells with different cathode materials behave over a wide temperature range. Cells are more prone to thermal abuse may encounter different modes of failure such as thermal runaway due to internal gas and heat generation, short-circuit due to electrode displacement and contact of two electrodes. It is obvious that thermal abuse shortens the life of the cell due to failures it may cause. Therefore, it has become essential to select thermally stable cathode materials to create cells that resist thermal abuse [17].

349

## 3.2 Applying AHP method

Table 3. Numerical Scale for Pairwise Comparison [38].									
Linguistic variable	Numeric value								
Extremely important	9-8								
Very Strongly more important	7-6								
Strongly more important	5-4								
Moderately more important	3-2								
Equally important	1								

The International Conference of Materials and Engineering Technology

The basic concept of AHP is to decide objective, alternatives that we evaluate and criteria used for evaluation of alternatives. After deciding all constraints and objective, a pairwise comparison matrix is generated to compare alternatives over criteria for all alternatives. As a next step, a numeric scale shown in Table 3 is used to calculate the relative importance of two criteria and it is carried on with until all criteria are compared to each other to generate the matrix. For instance, if C1 is compare to C2 and it is assigned as 5, the comparison of C2 to C1 becomes 1/5 as its reciprocal. After that, in order to obtain the required relative criteria weights, the matrix is normalised. And then, percentage importance distribution of the alternatives is calculated to get a 1×m matrix where m is the number of alternatives. Finally, to achieve the rank of alternatives, the matrix of option scores (n×m) is computed where n is the number of criteria (detailed in [39]).

## **3.3 Applying Interval Type-2 Fuzzy Sets**

In this study, the decision maker selected four main, nine sun-criteria. After that, all criteria are defined in a linguistic way such as "very high" in order to represent the importance of each criterion. Linguistic weights of attributes are shown in Table 4. Then, the linguistic definitions for all criteria are converted into fuzzy weights using fuzzy membership functions. For example, let one criterion defined as "Medium", it is assigned as ((0.3, 0.5, 0.5, 0.7) (0.4, 0.5, 0.5, 0.6)) as shown in Table 4. Next, cathode materials are identified in the same manner using linguistic terms such as 'very good', 'good', 'poor'. Linguistic terms and their corresponding fuzzy sets are demonstrated in Table 5. After that, these terms are converted into fuzzy performance rating. And then, the aggregate fuzzy score is calculated using Centroid type-reduction and defuzzification methods and the rank of materials are obtained as shown in Table 11 (detailed in [39]).

<b>Trapezoidal ITFSs</b>	$\tilde{a}_{i1}^u$	$\tilde{a}_{i2}^{u}$	$\tilde{a}_{i3}^u$	$\tilde{a}_{i4}^u$	$h_1$	$h_2$	$\tilde{a}_{i1}^l$	$\tilde{a}_{i2}^{l}$	$\tilde{a}_{i3}^l$	$\tilde{a}_{i4}^{l}$	$h_1$	$h_2$
Vey Low (VL)	0	0	0	0.1	1	1	0	0	0	0.05	0.9	0.9
Low (L)	0	0.1	0.1	0.3	1	1	0.05	0.1	0.1	0.2	0.9	0.9
Medium Low (ML)	0.1	0.3	0.3	0.5	1	1	0.2	0.3	0.3	0.4	0.9	0.9
Medium (M)	0.3	0.5	0.5	0.7	1	1	0.4	0.5	0.5	0.6	0.9	0.9
Medium High (MH)	0.5	0.7	0.7	0.9	1	1	0.6	0.7	0.7	0.8	0.9	0.9
High (H)	0.7	0.9	0.9	1	1	1	0.8	0.9	0.9	0.95	0.9	0.9
Very High (VH)	0.9	1	1	1	1	1	0.95	1	1	1	0.9	0.9

**Table 4.** Linguistic variable to evaluate each criterion [40]



## 4. Results and Discussion

#### 4.1. Results of AHP

In Table 6, the pairwise comparison matrix filled by a decision maker is demonstrated. After that, matrices are generated and normalised as shown in Table 7 which gives only one criterion as an example because of the page restriction.

**Table 6.** Pairwise comparison matrix for Li-ion cathode materials.

Comparison	<b>C1</b>	<b>C2</b>	<b>C3</b>	<b>C4</b>	C5	<b>C6</b>	<b>C7</b>	<b>C8</b>	<b>C9</b>
LCO/LMO	7	7	1	1/3	1/7	1/5	1/5	3	1/3
LCO/LFP	1/3	1	1/3	7	1/7	1/7	1/5	1/7	1/5
LCO/NMC	1/5	1/7	1	3	1/7	1/7	1/3	1/7	1/5
LCO/NCA	1/9	1/9	1	3	1/7	1/3	1	3	1/3
LMO/LFP	1/5	1/7	1/3	9	1	1/5	1	1/9	1
LMO/NMC	1/7	1/7	1	5	1	1/3	3	1/9	1
LMO/NCA	1/9	1/9	1	5	1	3	5	1	3
LFP/NMC	1/3	1/3	3	1/5	1	1	3	1	1
LFP/NCA	1/5	1/7	3	1/5	1	5	5	9	3
NMC/NCA	1/3	1/5	1	1	1	5	3	9	3

	Matri	ix gene	1		Matri	_					
Comparison	LCO	LMO	LFP	NMC	NCA	LCO	LMO	LFP	NMC	NCA	Mean
LCO	1	7	0.33	0.20	0.11	0.06	0.24	0.03	0.04	0.06	0.088
LMO	0.14	1	0.20	0.14	0.11	0.01	0.03	0.02	0.03	0.06	0.031
LFP	3	5	1	0.33	0.20	0.17	0.17	0.10	0.07	0.11	0.126
NMC	5	7	3	1	0.33	0.28	0.24	0.31	0.21	0.19	0.247
NCA	9	9	5	3	1	0.5	0.31	0.52	0.64	0.57	0.508

Technology



At the same time, the performance importance of each criterion is compared to each other and demonstrated as Table 8. Finally, the location is ranked by calculating the score matrix and results are shown in Table 11. Based on the results, it is clearly seen that NMC is the best material among five alternatives by a small margin while NCA is placed as the second preferable material.

Criteria	<b>C1</b>	C2	C3	C4	C5	C6	<b>C7</b>	<b>C8</b>	<b>C9</b>	Mean
C1	1	3	1	1	1	3	5	1	5	0.186
C2	0.33	1	3	1	1	1	3	1	5	0.125
C3	1	0.33	1	0.33	0.33	0.33	1	0.20	1	0.054
C4	1	1	3	1	1	1	5	1	3	0.141
C5	1	1	3	1	1	1	3	1	5	0.137
C6	0.33	1	3	1	1	1	1	1	3	0.107
<b>C7</b>	0.20	0.33	1	0.20	0.33	1	1	3	3	0.085
C8	1	1	5	1	1	1	0.33	1	5	0.133
С9	0.20	0.20	1	0.33	0.20	0.33	0.33	0.20	1	0.032

 Table 8. Percentage importance of potential criteria.

## 4.2. Results of interval type-2 fuzzy sets

In order to rank cathode materials, interval type-2 fuzzy sets are also used. First main and sub-criteria are defined as shown Table 1. Their importance is decided as depicted in Table 9. The main criterion and its corresponding sub-criterion is multiplied to convert linguistic terms to fuzzy sets and this is done for each sub-criterion. Let consider C1 as an example:

C	$C1 = MH \otimes VH = ((0.5, 0.7, 0.7, 0.9; 1, 1), (0.6, 0.7, 0.7, 0.8; 0.9, 0.9)) \otimes$	
(	((0.9,1,1,1;1,1),(0.95,1,1,1;0.9,0.9)) = ((0.45,0.7,0.7,0.9;1,1),(0.57,0.7,0.7,0.7;0.9,0.9))	(5)

The linguistic terms for performance of alternatives are determined as shown in Table 10. These terms are converted into fuzzy sets in the same manner as explained for importance of criteria. And then, aggregate fuzzy scores are calculated by multiplying each performance by fuzzy importance weight of criteria. In order to obtain crisp scores, fuzzy set values are converted into crisp values using Centroid type-reduction and defuzzification. Finally, these crisp values are ranked to obtain the rank of alternatives as depicted in Table 11.

Table 9. Importance of main and sub-criteria according to decision maker.													
	<b>M1</b>	M2	<b>M3</b>	<b>M4</b>	<b>C1</b>	<b>C2</b>	<b>C3</b>	<b>C4</b>	C5	<b>C6</b>	<b>C7</b>	<b>C8</b>	<b>C9</b>
Decision Maker	MH	VH	Μ	Н	VH	VH	Μ	VH	VH	Н	М	VH	MH

|--|

Alternatives	<b>C1</b>	<b>C2</b>	<b>C3</b>	<b>C4</b>	C5	<b>C6</b>	<b>C7</b>	<b>C8</b>	<b>C9</b>
LCO	F	MG	F	MG	G	VP	G	MG	Р
LMO	MP	MP	F	G	Р	F	Р	MP	MG
LFP	MG	MG	MG	F	VP	G	Р	G	G
NMC	G	G	F	MG	Р	MG	MP	G	MG
NCA	VG	VG	F	MG	Р	Р	MG	F	MP



NMC is found as the best cathode material for Li-ion batteries used for Scooter. In comparison to examining materials, it is found that the performance of NMC and NCA is quite similar both AHP and fuzzy approaches. Although, AHP assumes that decision makers provide precise information to examine alternatives, ambiguity can arise in judgements of the decision maker. For this reason, comparison of two methods is done and it is found that precise information is provided to examine alternatives according to same results achieved by AHP and interval type-2 fuzzy sets.

				<u>, , , , , , , , , , , , , , , , , , , </u>
Materials	Score	Rank	Score	Rank
LCO	0.098	5	11.661	5
LMO	0.180	4	21.374	4
LFP	0.233	3	23.264	3
NMC	0.245	1	24.444	1
NCA	0.243	2	23.342	2

## **Table** 11. Results for both AHP and fuzzy approaches.

## **5.** Conclusions

In this study, we addressed material selection problem for Li-ion batteries considering both qualitative and quantitative factors and solved the problem using two methods; AHP and interval type-2 fuzzy sets. The aim of this work is to examine different cathode materials under variety of criteria selected with respect to both subjective and objective thoughts. The motivation behind this study is also that there has been a lack of extensive research in the field of material selection for Li-ion batteries considering several criteria at the same time. Due to uncertainties raised in judgements, two methods are preferred to provide more appropriate results and it is found that both methods achieved same results. As a future study, this work can be extended to cover all materials which form Li-ion battery.

## References

- 1. Nitta, N., Wu, F., Lee, J. T., and Yushin, G., Li-ion battery materials: present and future. Materials Today, 2015, 18(5):252–264.
- 2. Nishi, Y., Lithium ion secondary batteries; past 10 years and the future, Journal of Power Sources, 2001, 100(1-2): 101-106.
- 3. Sarkar, A., Shrotriya, P., Chandra, A., Parametric Analysis of Electrode Materials on Thermal Performance of Lithium-Ion Battery: A Material Selection Approach Batteries and Energy Storage, J. Electrochem. Soc., 2018, 165(9): A1587-A1594.
- Mishra, A., Mehta, A., Basu, S., Malode, S. J, Nagaraj P. Shetti, N. P., Shukla, S., Nadagouda, M. N., Aminabhavi, T. M., Electrode materials for lithium-ion batteries, Materials Science for Energy Technologies, 2018, 1(2): 182-187.
- 5. Panday, A., and Bansal, H. O., Multi-Objective Optimization in Battery Selection for Hybrid Electric Vehicle Applications. Journal of Electrical Systems, 2016, 12(2):325–343.
- 6. Kaa, G., Fens, T., Rezaei, J., Residential grid storage technology battles: a multi-criteria analysis using BWM, Technology Analysis and Strategic Management, 2019, 31(1):40-52.
- 7. Sangwan, S. K., Jindal, A., An integrated fuzzy multi-criteria evaluation of lithium-ion battery recycling processes, International Journal of Sustainable Engineering, 2013, 6(4): 359-371.

8. Warner, J. T., The Handbook of Lithium-Ion Battery Pack Design: Chemistry, Components, Types and Terminology, Elsevier, Grand Blanck, MI, 2015, 1–80.

OMET

The International Conference of Materials and Engineering Technology

- 9. Graf, C., Cathode materials for lithium-ion batteries, Lithium-ion batteries: basics and applications. Edited by Korthauer, R., and Wuest, M., Springer, 2018, 29-40.
- 10. Deng, D., Li-ion batteries: Basics, Progress, and Challenges, Energy Science & Engineering, 2015, 3(5): 385–418.
- 11. Scrosati, B., and Garche, J., Lithium Batteries: Status, Prospects and Future. Journal of Power Sources, 2010, 195(9):2419–2430.
- 12. Gwon, H., Hong, J., Kim, H., Seo, D.-H., Jeon, S., and Kang, K., Recent Progress on Flexible Lithium Rechargeable Batteries. Energy Environ. Sci., 2014, 7(2):538-551.
- 13. Yuan, M., Erdman, J., Tang, C., and Ardebili, H., 2014, "High Performance Solid Polymer Electrolyte with Graphene Oxide Nanosheets," RSC Adv., 4(103), pp. 59637–59642.
- 14. Doeff, M. M., Chapter 2: Battery Cathodes. Batteries for Sustainability: Selected Entries from the Encyclopedia of Sustainability Science and Technology. Edited by Brodd J. R., Springer, New York, NY, 2013, 5-49.
- 15. Manthiram, A., Materials Challenges and Opportunities of Lithium Ion Batteries. The Journal of Physical Chemistry Letters, 2011, 2(3):176–184.
- 16. Wang, Y., and Huang, H.-Y. S., An Overview of Lithium-Ion Battery Cathode Materials. MRS Proceedings, 2011, 1363.
- 17. Doughty, D. H., and Roth, E. P., A General Discussion of Li Ion Battery Safety. The Electrochemical Society Interface, 2012, 21(2):37-44.
- 18. Schmuch, R., Wagner, R., Hörpel, G., Placke, T., & Winter, M., Performance and cost of materials for lithium-based rechargeable automotive batteries. Nature Energy, 2018, 3(4): 267-278.
- 19. Al-Hallaj, S., Wilk, G., Crabtree, G., and Eberhard, M., Overview of distributed energy storage for demand charge reduction. MRS Energy & Sustainability, 2018, 5(1): 1–18.
- 20. Lazim, A., Norsyahida Z., Integration of fuzzy AHP and interval type-2 fuzzy DEMATEL: An application to human resource management, Expert Systems with Applications, 2015, 42(9): 4397-4409.
- 21. Ying-Chyi C., Chia-Chi S., Hsin-Yi Y., Evaluating the criteria for human resource for science and technology (HRST) based on an integrated fuzzy AHP and fuzzy DEMATEL approach, Applied Soft Computing, 2012, 12(1):64-71.
- 22. Marbini A.H. and Tavana M., An extension of the Electre I method for group decision-making under a fuzzy environment, Omega, 2011, 39 (4): 373-386.
- 23. Sen, C. G., Cinar, G., Evaluation and pre-allocation of operators with multiple skills: A combined fuzzy AHP and max-min approach, Expert Systems with Applications, 2010, 37 (3): 2043-2053.
- 24. Ling W., Jian C., Jun W., Selection of optimum maintenance strategies based on a fuzzy analytic hierarchy process, International Journal of Production Economics, 2007, 107(1): 151-163.
- 25. Yeap, J.A.L., Ignatius J., Ramayah, T., Determining consumers' most preferred eWOM platform for movie reviews: A fuzzy analytic hierarchy process approach, Computers in Human Behavior, 2014, 31: 250-258.
- 26. Saaty, R., The analytic hierarchy process-what it is and how it is used, Math. Model.,1987,9:161–176.
- 27. Dweiri, F., Al-Oqla, F. M., Material selection using analytical hierarchy process. Int. J. Comput. Appl. Technol., 2006, 26 (4):182-189.

354



The International Conference of Materials and Engineering Technology

- 29. Kühn, F., Rehra, J., May, D., Schmeer, S., Mitschang, P., Dry fiber placement of carbon/steel fiber hybrid preforms for multifunctional composites, Advanced Manufacturing: Polymer & Composites Science, 2019,5(1):37-49.
- 30. Zadeh, L., Fuzzy sets. Information and Control, 1965, 8:338-353.
- 31. Zadeh, L., The consept of a linguistic variable and its applications to approximate reasoning. Inform Science, 1975, 8:199- 249.
- 32. Hagras, H., Type-2 flcs: A new generation of fuzzy controllers. IEEE Computational Intelligence Magazine, 2007, 2:30-43.
- 33. Greenfield, S., Chiclana, F., John, R., Coupland, S., The sampling method of defuzzification for type-2 fuzzy sets: Experimental evaluation, Information Sciences, 2012, 189: 77-92.
- 34. Mendel, J.M., John, R., Liu, F., Interval type-2 fuzzy logic systems made simple. IEEE T. Fuzzy Systems, 2006, 14:808- 821.
- 35. Young, K., Wang,, C., Wang, L. Y., and Strunz, K., Electric Vehicle Battery Technologies. Electric vehicle integration into modern power networks. Edited by R. Garcia-Valle, and J.A.P. Lopes, SPRINGER-VERLAG NEW YORK, 2013, 15–56.
- 36. W. Reaugh, Larry. Re-Cycling Spent Electric Vehicle Batteries Potentially Recovers Significant Amounts of Lithium, Cobalt and Other Cathode Metals. American Manganese Inc., 19 Jan. 2017, https://americanmanganeseinc.com/re-cycling-spent-electric-vehiclebatteries-potentially-recovers-significant-amounts-of-lithium-cobalt-and-other-cathodemetals/.(accessed September 10, 2019).
- Sen, C. G., Cinar, G., Evaluation and pre-allocation of operators with multiple skills: A combined fuzzy AHP and max–min approach, Expert Systems with Applications, 2010, 37 (3): 2043-2053.
- 38. Bhushan, N., & Rai, K. (2004). Strategic Decision Making: Applying the Analytic Hierarchy Process. Decision Engineering. Springer London.
- 39. Türk, S. John, R. and Özcan, E., Interval type-2 fuzzy sets in supplier selection, 14th UK Workshop on Computational Intelligence (UKCI), Bradford, 2014, pp. 1-7.
- 40. Chen, S., Lee, L., Fuzzy multiple attributes group decision-making based on the interval type-2 TOPSIS method, Expert Systems with Applications, 2010, 37 (4): 2790-2798.

# ŞEBEKE KALKIŞLI DAİMÎ MIKNATISLI SENKRON MOTORUN SCADA TABANLI ONLİNE DURUM İZLENMESİ VE RULMAN ARIZASI TESPİTİ

The International Conference

of Materials and Engineering Technology

## SAADET GULSUM GOZUOGLU \*1, ZAFER DOGAN 2

<sup>1-2</sup> Tokat Gaziosmanpaşa Üniversitesi, Elektrik ve Elektronik Mühendisliği, Tokat, TÜRKİYE.

#### Özet

Yüksek verim ve yüksek güç faktörü gibi avantajlara sahip Şebeke kalkışlı daimî mıknatıslı senkron motor (SKDMSM) bant sistemleri, fan sistemleri vb. endüstriyel alanlarda günden güne asenkron motorların yerini almaktadır. ŞKDMSM'ler ağır işletme koşulları nedeniyle zamanla arızalanmaktadır. Bu motorların arızalanması üretim kayıpları oluşturmasının yanı sıra yüksek bakım ve onarım masraflarını da beraberinde getirir. Bu nedenle ŞKDMSM'de oluşacak arızaların hızlı bir şekilde tespiti çok önemlidir. Son yıllarda motorların çalışma durumlarının denetiminde online durum izlemesi yapılmaktadır. Bu çalışmada ŞKDMSM'nin çalışma durumunun uzaktan kontrol edilmesi, motora ait analog-dijital akım ve gerilim bilgilerinin bir merkezde toplanması ve bu bilgilerin analiz edilerek rulman arızası tespiti için, Supervisory Control and Data Acquisition (SCADA) tabanlı durum izleme otomasyonu gerçekleştirilmiştir. Önerilen arıza tespiti, motor akım sinyal bilgileri üzerinde istatistiksel proses kontrol yöntemine dayalıdır. Durum izleme amacıyla gerçekleştirilen donanımsal bölüm merkezinde mikro denetleyici olarak Arduino Mega yer almaktadır. Kullanılan mikro denetleyici düşük maliyetli olarak seçilmesi herkes tarafından erişilebilir olmasını sağlarken düşük örnekleme frekansı (~1000 samples per second) bir dezavantaj olarak ortaya çıkmaktadır. Düşük frekans değerine sahip olan örnekleme değerlerinden arıza tespiti yapabilmek için Exponentially Weighted Moving Average (EWMA) grafik yöntemi kullanılmıştır. SCADA ara yüzünde görüntülenen ve veri tabanına kaydedilen tüm veriler sayesinde ŞKDMSM online durum izlemesi yoluyla başarılı bir şekilde rulman arızası tespit edilebilmektedir.

Anahtar Kelimeler: Şebeke kalkışlı daimî mıknatıslı senkron motor, Rulman arızası, Ewma grafik yöntemi, Scada, Online durum izleme.

#### Giriş

Teknolojideki gelişmeler her alanda sürekli bir iyileşme ve hayatı kolaylaştırma olarak ortaya çıkmaktadır. Bu gelişmeler, endüstride çok yaygın olarak kullandığımız motorlarda da görülmektedir. Endüstriyel uygulamalarda en yaygın olarak kullanılan tahrik elemanı asenkron motorlar (ASM) olarak görülmektedir [1]. ASM' nin tercih edilme nedenlerinden bazıları, yapısal olarak basit ve ekonomik olmaları, periyodik bakım gerektirmemeleri ve güvenilir olmalarıdır. Ancak bu önemli avantajlarının yanında hacimsel olarak büyüklükleri, verimlerinin senkron motora göre düşük olması, gürültülü çalışmaları ASM' lerin önemli dezavantajlarındandır.



ASM'lerin verimlerinin düşük olması, motor araştırmacılarını son yıllarda verimli motor arayışlarına yöneltmiştir. Yapılan çalışmalarda Daimi Mıknatıslı Senkron Motor (DMSM)'un rotor yapısına ASM'nin kafes sistemi eklenerek Şebeke Kalkışlı Daimi Mıknatıslı Senkron Motor (ŞKDMSM) elde edilmiştir. Eklenen bu yapı sayesinde ŞKDMSM'ler şebekeden direk beslenerek kalkış yapabilmektedirler [2]. ŞKDMSM'nin rotor yapısında bulunan mıknatıslar sayesinde sürekli haldeki performans artırılmıştır [3]. ŞKDMSM yüksek verim kabiliyeti, hacim ve ağırlığına göre yüksek güç sağlaması, hassas ve kararlı kontrol imkânı, yüksek moment gibi pek çok avantajlarından ötürü endüstrinin birçok alanında en fazla tercih edilen motor türlerinden biri olmaya adaydır [3].

The International Conference of Materials and Engineering Technology

Tüm motorlarda olduğu gibi SKDMSM'lerin de çalışma ömrü sağlıklı çalışmalarına bağlıdır. Sanayinin birçok ortamında bulunan sıcaklık, nem, toz vb. etkiler ŞKDMSM'lerde arızaların meydana gelmesine neden olmaktadır. Bu arızalar genel olarak elektriksel, mekanik ve manyetik arızalar olarak gruplandırılmaktadır. Elektriksel arızalar; sargı arızaları, rotor çubuk kırığı arızası ve rotor cubuk halkası kırığı arızasıdır. Mekanik arızalar; eksenden kaçıklık ve rulman arızalarıdır. Manyetik arızalar ise demagnetizasyon arızası ve mıknatıs kırığı arızası olarak sınıflandırılabilir [4]. Arızalı olan motorun çalışma ömrü de kısadır. Arızanın henüz başlangıç aşamasında iken tespit edilmesi ve buna müdahale edilmesi sistemin devamlılığı açısından ve motor çalışma ömrü açısından büyük öneme sahiptir [5]. Günümüzde online izleme yoluyla motorların işletme koşullarındaki durumları denetlenmektedir. Online durum izleme sayesinde, motora ait arızalar önceden tespit edilebilmekte, arızanın sebep olacağı motorun tamamen kullanılamaz hale gelmesi durumun önüne geçilebilmekte ve etkin bir koruma sağlanabilmektedir. ŞKDMSM'lerin online durum izlemelerinde birçok yöntem kullanılmaktadır: termal izlemeler, hava aralığı moment izleme, akustik ses ölçümü, titreşim izleme, motor akımlarının izlenmesi, manyetik akının izlenmesi, indüklenen gerilimin izlenmesi, kısmi deşarjın izlenmesi, gaz analizi, ani elektriksel gücün izlenmesi, ani açısal hızın izlenmesi, darbe testi ve gerilim izleme. Bu yöntemler içerisinde en pratik olan ve en yaygın kullanılan yöntem motor akımlarının izlenmesidir.

Arıza tespit amaçlı sinyal analizlerinin en önemli noktası özellik çıkarmadır. Özellik çıkarma işlemlerinde sinyalleri temsil eden en baskın özellikler hesaplanarak sinyaller sayısallaştırılır. [6]. Elektrik makinalarında oluşan arızaları temsil eden özellikleri elde etmede en çok kullanılan yöntemler frekans boyutunda analiz, zaman-frekans boyutunda analiz, zaman boyutunda analiz, model tabanlı analizler vb. olarak sınıflandırılmaktadır [7]. Bu analizler içerisinde zaman boyutunda analizler en hızlı arıza tespiti yapması ve pratik olması yönleriyle diğerlerine nazaran daha çok kullanılmaktadır. Zaman boyutunda analiz yöntemlerinde, çoğunlukla sinyallerin istatistiksel özelliklerinin elde edilmesi amaçlanmaktadır. Bu amaç için kullanılan yöntemlerden biri de İstatistiksel proses kontrol (İPK) yöntemidir. İPK, sinyallerin istatistiksel özellikleri yoluyla süreç takibi, arıza belirleme, kalite kontrol vb. uygulamalarda kullanılmaktadır.

Literatür incelendiğinde İPK yönteminin, birçok çalışmada kullanıldığı görülmüştür. ASM'nin kullanıldığı [8] ve arıza tespiti için İPK yönteminin kullanıldığı çalışma offline olarak değerlendirilmiştir. Veriler motordan alındıktan sonra Matlab uygulaması kullanılarak arıza tespit edilmiştir. İPK [9] yöntemi kullanılarak hazırlanmış olan başka bir çalışmada ise bir askeri kuruluşa ait araçların motor yenileştirilmesi süreci üzerinde durulmuştur. Kullanılan yöntem ile yenileştirme sürecinde hataların önlenmesi, işçilik ve malzeme kayıplarının düşürülmesi sağlanmıştır. Djmal ve ark. [10] Optimize (Exponentially Weighted Moving Average) EWMA yöntemini kullanarak atık su arıtma tesisinde suların temizlenmesini kontrol etmişlerdir. Bu yöntem en ufak değişiklikleri ortaya çıkarabildiği için arıtma sürecindeki verimi arttırmıştır.

Bu çalışmada ŞKDMSM'nin rulman arızası tespiti için Scada tabanlı durum izleme otomasyonu gerçekleştirilmiştir. Önerilen arıza tespit yöntemi, ŞKDMSM stator akım sinyalleri üzerinden İPK yoluyla özellik çıkarmaya dayalıdır ve bu alandaki literatürde bu motor için ilk kez kullanılmıştır.

aterials and Engineering Technology

Durum izleme amacıyla gerçekleştirilen donanımsal bölüm merkezinde mikro denetleyici olarak Arduino Mega kullanılmıştır. Arduino Mega Remote Terminal Unit (RTU) gibi görev yapmaktadır. Donanımsal bölümü; sensör verilerini toplayan, düzenleyen ve Scada ara yüzüne aktaran yapıdır. Ayrıca Scada ara yüzünden gelen komutları donanımsal bölümde uygun yere gönderilmesini sağlamaktadır. Donanımsal böümden alınan akım sinyalleri SCADA ortamında izlenmekte ve EWMA İPK yöntemi kullanılarak arıza tespiti yapılmaktadır.

Bu makale 6 bölümden oluşmaktadır. Makalenin devam eden ikinci bölümünde ŞKDMSM matematiksel modeli, Üçüncü bölümde İstatistiksel Proses Kontrol, Dördüncü bölümde SCADA Sistemi ve RTU, Beşinci bölümde deneysel çalışmalar ve sonuçları hakkında bilgi verilmiştir. Altıncı bölüm sonuçlar bölümüdür.

## 1. Şebeke Kalkışlı Daimi Mıknatıslı Senkron Motor Modeli

ŞKDMSM'ler ASM'lerin ve DMSM'lerin en üstün özelliklerini taşıyan hibrit motorlardır. Yapısal olarak ASM stator yapısına sahip olup, rotorda ASM'nin kafes yapısına ilaveten DMSM rotorunda bulunan daimi mıknatıslar eklenmiştir. Bu motor kalkış anında yaklaşık asenkron tork ile yol alırken sürekli durumda mıknatıs torku ile senkron çalışır. Bu nedenle ŞKDMSM'ler arada evirici olmadan doğrudan şebekeyle beslenebilir. Sürekli halde çalışırken klasik senkron motorun performansına sahiptir [11].

Rulman arızaları gibi mekanik arızalar motorda titreşimlere, yük değişimlerine ve hava aralığında akı değişimlerine sebep olurlarken, motor akımlarında da harmonikler oluştururlar. Bütün bunların sonucunda ise motor torkunda salınımlar ortaya çıkar. Motorların arıza durumlarının motor parametreleri üzerine etkisini incelemede matematiksel modelleri kullanmak etkin bir yaklaşımdır. Matematiksel modeller motorların fiziksel davranışlarının belirlenmesinde ve arıza tespitinde kullanılabilirler. Rulman arızası durumundaki bir ŞKDMSM modeli için sağlam durum matematiksel model öncelikle bilinmelidir. Bu motorun matematiksel modeli d-ekseni, q-ekseni ve sıfır bileşen dikkate alınarak çıkartılabilir [12]. **Şekil 1**'de ŞKDMSM'ye ait eşdeğer devreler gösterilmiştir.



d – ekseni



(1)



Sıfır dizi bileşen devresi

Şekil 1. Üç fazlı ŞKDMSM eşdeğer devresi [12]

ŞKDMSM'de sürekli mıknatıslarında oluşan rotor eddy akım kayıpları ihmal edilerek, motorun eşdeğer mıknatıslanma akısı  $\lambda_m$  Formül 1 ile hesaplanabilir.

$$\lambda_m = L_{md} i'_m$$

Burada, i mıknatıslanma akımını ve L mıknatıslanma indüktansını göstermektedir. Kirşoff mdm

gerilim yasası dikkate alınarak yazılan, ŞKDMSM'de d-q ve 0 eksenleri için stator gerilimi  $v^r$ ,  $v^r$ ,  $v^r$ , rotor gerilimi  $v^{'r}$  ve  $v^{'r}$ , sırasıyla formül 2 ve formül 3'de,

$$\begin{array}{l}
\boldsymbol{v}_{ds}^{\prime} = \boldsymbol{r}_{s}\boldsymbol{l}^{\prime} - \boldsymbol{\omega} \,\boldsymbol{\lambda}^{\prime} + \boldsymbol{p}\boldsymbol{\lambda}^{\prime} \\
\boldsymbol{d}_{s}^{\prime} = \boldsymbol{r}_{ds}^{\prime} \boldsymbol{\lambda}^{\prime} + \boldsymbol{p}\boldsymbol{\lambda}^{r} \\
\boldsymbol{v}_{qs}^{r} = \boldsymbol{r}_{s}^{\prime} \boldsymbol{i}_{s}^{r} + \boldsymbol{p}\boldsymbol{\lambda}^{r} \\
\boldsymbol{v}_{0s}^{r} = \boldsymbol{r}_{s}\boldsymbol{i}^{r} + \boldsymbol{p}\boldsymbol{\lambda}^{r} \\
\boldsymbol{v}_{qr}^{r} = \boldsymbol{r}_{dr}^{\prime} \boldsymbol{i}_{qr}^{r} + \boldsymbol{p}\boldsymbol{\lambda}^{\prime r} = \mathbf{0} \\
\boldsymbol{v}_{dr}^{\prime} = \boldsymbol{r}_{dr}^{\prime} \boldsymbol{i}_{dr}^{r} + \boldsymbol{p}\boldsymbol{\lambda}^{\prime r} = \mathbf{0} \\
\boldsymbol{v}_{dr}^{\prime} = \boldsymbol{r}_{dr}^{\prime} \boldsymbol{i}_{dr}^{\prime r} + \boldsymbol{p}\boldsymbol{\lambda}^{\prime r} = \mathbf{0} \\
\end{array}$$
(2)

olarak yazılabilir. Bu ifadelerde  $r_s$ , stator direnci, r'rotor direnci, L ve L ve r

> dr qrтq md

mıknatıslanma indüktansı, L' rotor kaçak indüktansı,  $L_{ls}$  stator kaçak indüktansı ve  $\omega_m$  açısal hızdır. Motorun stator akısı  $\lambda^r$ ,  $\lambda^r$ ,  $\lambda^r$ ,  $\lambda^r$ , rotor akısı  $\lambda'^r$ ,  $\lambda'^r$  sırasıyla formül 4 ve formül 0r

ds 0*s* qs qr dr 5'te ifade edilmiştir.

ds



$$\lambda_{qs}^{r} = L_{ls}i_{qs}^{r} + L_{mq}(i_{qs}^{r} + i_{qr}^{r})$$

$$\lambda_{ds}^{r} = L_{ls}i_{ds}^{r} + L_{md}(i_{ds}^{r} + i_{qr}^{r}) + L_{md}i_{m}^{r}$$

$$\lambda_{ds}^{r} = L_{ls}i_{ds}^{r} + L_{mq}(i_{qs}^{r} + i_{qr}^{r})$$

$$\lambda_{qr}^{r} = L_{lr}i_{qr}^{r} + L_{mq}(i_{qs}^{r} + i_{qr}^{r})$$

$$\lambda_{dr}^{r} = L_{lr}i_{dr}^{r} + L_{md}(i_{ds}^{r} + i_{qr}^{r}) + L_{md}i_{m}^{r}$$

$$\lambda_{dr}^{r} = L_{lr}i_{dr}^{r} + L_{md}(i_{ds}^{r} + i_{dr}^{r})$$

$$\lambda_{0r}^{r} = L_{lr}i_{dr}^{r}$$
(5)

Burada;  $i_{ds}$ ,  $i_{qs}$ ,  $i_{0s}$  stator akımları,  $i'^r$ ,  $i'^r$ ,  $i'^r$  rotor akımlarıdır.

qr dr 0r

Sonuç olarak motoru arıza durumunu incelemek için kullanılacak tork ifadesi; *P* kutup sayısı olmak üzere tork ifadesi formül 6 ile hesaplanabilir.

$$T_{e} = \frac{3P}{22} \begin{bmatrix} L_{md} i_{dr}^{'r} i_{qs}^{r} - L_{mq} i_{qr}^{'r} i_{ds}^{r} + \lambda_{m}^{'r} i_{qs}^{r} \\ + (L_{md} - L_{mq}) i_{ds}^{r} i_{qs}^{r} \end{bmatrix}$$
(6)

## 2. İstatistiksel Proses Kontrol

İPK süreç içerisinde oluşan en ufak değişikliklere bağlı olarak farklılığı ortaya çıkartan bir yöntemdir[13-15]. Normal durum içerisinde hesaplanan değerler limit değerlerini içerisinde salınım yapmaktadır. Ancak limit değerlerini aşmaları, durumunda oluşan değişikliği belirtmektedir.[16, 17].

Proses kontrol yöntemlerinden biri olan Ewma'da, merkez çizgi (MÇ) ile bunun altına ve üstüne çizilen, alt kontrol limit (AKL) ve üst kontrol limit (ÜKL) çizgileri bulunmaktadır. MÇ salınımın hangi düzlemde olması gerektiğini belirtirken AKL ve ÜKL ise salınım alanını ifade etmektedir. Bu alan dışına çıkılması durumunda arıza veya normal olmayan durum teşhis edilmiş olur [18, 19]. Örnek bir İPK grafiği **Şekil 2**'te görülmektedir.



Technology



kullanılan hesaplama aşamaları aşağıdaki formüllere göre yapılır.

k alt grup sayısı, n h er bir alt grupta bulunan ölçüm sayısı, xij değeri i alt grubundaki j numaralı örnekleme değeri olmak üzere, alt grubunun ortalaması xi Formül 8'de gösterilmiştir.

$$\overline{x} = \frac{\sum_{j=1}^{n} x_{ij}}{n}$$
(7)

Tüm alt grupların genel ortalaması aşağıdaki şekilde hesaplanmaktadır.

$$\overline{x} = \frac{\sum_{k=1}^{k} \overline{x}}{k}$$
(8)

Her bir alt grubun standart sapma değeri  $(s_i)$  ve genel ortalaması formül 9 ve formül 10'daki ifadelerle hesaplanır.

$$s_{i} = \sqrt{\frac{\sum_{i=0}^{n} (x_{i} - \bar{x})^{2}}{n-1}}$$
(9)

$$\overline{s} = \frac{1}{k} \sum_{i=1}^{k} s_i \tag{10}$$

Hesaplama sonucu grafikteki her bir nokta aşağıdaki şekilde elde edilir. Zo değeri olarak genel ortalama değeri kullanılmıştır.

$$z_0 = \overline{x} \tag{11}$$

$$z_i = \lambda \overline{x} + (1 - \lambda) z_{i-1} \tag{12}$$



Çalışmada Ewma grafik analizi gerçekleştirilirken sistemin all ve üst kontrol limitleri (AKL, UKL) de belirlenmektedir. Formül 13'te belirtilen değerler bu limitleri aşarsa arıza tespit edilmiş olur. Bu denklemlerde L sabit değerdir.

$$AKL_{i} = \overline{x} - (L\overline{s})\sqrt{\frac{\lambda}{(2-\lambda)}} \left[1 - (1-\lambda)^{2i}\right]$$
(13)

$$UKL_{i} = \overline{x} + (L\overline{s})\sqrt{\frac{\lambda}{(2-\lambda)}} [1 - (1-\lambda)^{2i}]$$
(14)

#### **3.SCADA Sistemi ve RTU**

SCADA sistemi bütünleşik endüstriyel ortamlarda tüm alt birimleri kontrol eden ve izleyen merkezi sistemdir. Bu sistemlerde denetim kontrolü ve veri toplama işlemleri yapılır. Günümüzde havaalanları, fabrikalar, elektrik üretim tesisleri vb. birçok alanda kullanılmaktadır. SCADA sistemlerinde sistem kontrolleri yapılabildiğinden, endüstriyel ortamlardaki motorların online izlenmesi ve arıza tespitinde de etkin bir şekilde kullanılabilirler. Motor arızalarında erken arıza teşhisi sayesinde önemli donanım hasarlarının önüne geçilebilir. Bu nedenle motorda ortaya çıkan arıza kritik bir noktada ise SCADA sistemi ile motor işletmeden alınabilir. Motor korumasında olduğu gibi elektrik şebekelerinin kontrolü ve arıza takibinde de SCADA sistemleri kullanılmaktadır [20]. Şekil 3'te SCADA için tasarlanan sisteminin genel şeması görülmektedir.



Şekil 3. SCADA için tasarlanan sisteminin genel şeması

Sistem, bilgileri toplayan RTU bölümünden, bilgilerin analiz edilmesini sağlayan SCADA ara yüzünden ve veri tabanından oluşmaktadır [20].

Bu çalışmada tasarlanan motor online durum izleme ve koruma sisteminin donanımsal bölümünün temelini Arduino Mega oluşturmaktadır. Arduino Mega içerisinde ATmega2560 çip barındıran, 54 dijital giriş-çıkış, 16 analog giriş pinine sahip olan ve 256kB'lık geniş bir hafızası bulunan bir mikro denetleyicidir [21]. Sistemde kullanılan akım algılayıcı Zeming marka ZMCT103C model en fazla 5A rms ölçüm yapabilmektedir. Algılayıcı çalışma şekli 5A/5mA şeklinde olup çıkış pinlerine RB=200 $\Omega$  örnekleme direnci bağlanmıştır.



Bu sayede hem akım trafosu açık uçlu kalmamış hem de algılayıcı çıkış işareti ölçümü değerlendirecek olan Arduino analog giriş yapısına uygun olarak gerilim biçimine dönüştürülmüştür [22]. Gerilim algılayıcı olarak, ZMPT101B model en fazla 250V rms ölçüm yapabilen 2mA/2mA gerilim sensörü kullanılmıştır. Gerilim ölçümü üç fazlı sistemde faz-nötr arası gerçekleştirilmiştir. Donanımsal kontrol birimi Şekil 4'te görülmektedir.



Şekil 4. Donanımsal Kontrol Birimi genel yapısı

Donanımsal bölümde 16x2 LCD ekran, DS3231 gerçek zaman modülü ve röle modülü yer almaktadır. LCD ekran ölçüm ve değerlendirme sonuçlarının SCADA ara yüzünün yanı sıra donanımsal bölümde de görüntülenmesini sağlamaktadır. DS3231 Real Time Clock (RTC) modülü I2C iletişim protokolü kullanarak mikro denetleyici ile iletişim sağlamakta ve ara yüze gönderilen verilerin zaman etiketi ile aktarılmasını sağlamaktadır. Zaman ile ilgili uygulanabilecek senaryolarda bu modül kullanılabilmektedir. Röle modülü mikro denetleyici dijital çıkışlarından birine bağlanmış durumdadır. Bu modül motor koruması sağlanırken gerekli durumda motorun enerjisiz kalmasını sağlamak için kullanılmıştır. Kullanmış olduğumuz kontrol yönteminde arızanın teşhis edilmesine bağlı olarak motorun durdurulması gerektiğinde röle modülü bu görevi yerine getirmektedir.

Mikro denetleyici içerisinde yer alan yazılım iki formatta çalışmaktadır. Ara yüzden gelen talebe göre birinci çalışma modunda elde edilen örnekleme verilerini kullanarak pozitif tepe noktalarını yazılım ortamına gönderir ve EWMA kontrol yöntemi için veri sağlar. İkinci çalışma modunda ise yine örnekleme verilerini ve "*Emonlib*" kütüphanesini kullanarak akım, gerilim ve güç değerleri gibi sistemin anlık durumunu kullanıcıya sunmaktadır.

Çalışmanın yazılımsal bölümünde ise Processing 3 ve Wamp Server kullanılmıştır [20]. Processing ortamı özellikle mikro denetleyici çalışmalarında entegreye gömülü halde bulunan kullanıcı tarafından görülemeyen verilerin kullanıcı tarafından görüntülenmesini ve kolay bir şekilde kontrol etmesini sağlayan Java tabanlı bir ortamdır.



Çalışmada kullanılan Scada ara yüzü seri iletişim yolu ile mikro denetleyiciden gelen veri paketlerini alıp değerlendirecek şekilde Processing ortamında hazırlanmıştır. Şekil 5'te görüldüğü gibi ara yüz uygun Port Adı ve Baud Rate seçilerek mikro denetleyici ile online olarak iletişimde olduğu gibi Wamp Server yazılımı kullanılarak MySQL veri tabanıyla da iletişimini sağlamaktadır [22].



Şekil 5. SCADA ara yüzü

Processing ortamında hazırlanmış olan SCADA ara yüzünün, donanımsal bölümden verileri alabilmesi için belirli kurallar oluşturulmuştur [20]. Port adı ve baud rate gibi seri iletişim parametrelerinin seçilmesinin ardından "*Connect*" butonuna basılmasıyla ara yüz mikro denetleyiciye başlama komutu göndermektedir. Bu komutu bekleyen Arduino veriyi doğrulayarak seri iletişimi başlatır. Ara yüze String formatında "*Data#*" ile başlayan devamına sensör verilerinin virgül "," karakteriyle art arda sıralanmış değerlerinin eklenmesiyle oluşan uzun bir veri aktarılır. Veri yapısı "*Data#Sensor1,Sensor2,Sensor3,…*" şeklindedir [22]. Ara yüz, gelen verinin "*Data#*" ile başladığını onayladıktan sonra "*Split*" fonksiyonunu kullanarak tüm veri değerlerini virgül "," karakterine göre ayırır. Önceden tanımlanmış bir dizi elemanları olarak atar. Scada ara yüzü donanımsal bölümdeki yapılarda elde edilen verileri bu yolla güncellemiş olur [22].

Wamp Server kullanılarak oluşturulan MySQL veri tabanına ara yüze ulaşan veriler kaydedilir [20]. Kaydedilen bu veriler istatistiksel proses kontrol yöntemi için giriş verisi olarak kullanılmaktadır. Veri tabanında oluşturulan tablolar giriş verilerinin kaydedilmesini sağladığı gibi analizler sonucunda elde edilen karar değerlerinin de kaydedilmesini sağlamaktadır. Böylece geçmişe yönelik analizler ve raporlamalar da kolaylıkla yapılabilmektedir [20].





## Deneysel Çalışmalar ve sonuçlar

Burada ŞKDMSM'nin online durum izlemesi ve rulman arızaları tespiti için deneyler yapılmıştır. Önerilen sisteminin metodolojisi; veri izleme ve toplama, sinyal işleme ve arıza tespiti aşamalarından oluşmaktadır. Önerilen SCADA tabanlı online durum izleme ve arıza tespit sistemine ait blog şema Şekil 6'te görülmektedir.



Şekil 6. Sistemin genel çalışma algoritması

Yukarıda görülen sistemin çalışması Arduino Mega'nın akım sensörlerini kullanarak akım sinyalini örneklemesi ve pozitif tepe noktalarını tespit etmesiyle başlamaktadır. Sinyal tepe noktaları mikro denetleyicide uygun paketler şeklinde düzenlenip seri iletişim yolu ile SCADA ara yüzüne aktarılmaktadır. Ara yüz kendisiyle uyumlu olarak çalışan veri tabanına bu değerleri kaydetmektedir. EWMA grafik analizini gerçekleştirmek için kayıtlı olan veriler kullanılmaktadır. Çalışmamızda EWMA analizi için 10 grup veri kullanılmış, her bir grupta ise 50 veri yer almıştır. Toplamda beş yüz veri kullanılmıştır. Kullanılan mikro denetleyiciye bağlı olarak örnekleme frekansının düşük olması zaman-frekans boyutlu analizlerde arıza teşhisinin yetersiz olmasına neden olmuştur. Kullanılan istatistiksel proses kontrol yöntemi çok küçük değişiklikleri tespit edebildiğinden dolayı rulman arızası başarılı bir şekilde tespit edilmiştir. Motorun normal çalışma durumunda elde edilen EWMA grafik sonuçları en büyük limit ve en küçük limit değerleri arasında salınım yaparken rulman arızası durumunda aşırı sapma yaparak arızayı net bir şekilde tespit edebilmiştir. Eğer talep edilirse donanım bölümünde yer alan röle modülü sayesinde motor kontaktör enerjisi kesilerek motor durdurulup daha fazla hasar görmesi engellenebilmektedir. Tüm bu işlemler gerçekleşirken SCADA ekranında online durum izleme yapılabilmektedir. Ayrıca tüm verilerin kaydedilmesine bağlı olarak geçmişe yönelik her türlü analiz ve raporlama yapılabilmektedir.

Motor arızalarının tespitinde en önemli hususlardan biri, motor verilerinin güvenilir bir şekilde izlenmesidir. Bu nedenle iyi bir deney düzeneği oluşturulmalıdır. Burada kullanılan deney düzeneği Şekil 7'de görülmektedir.



Şekil 7. Deney düzeneği

Deney düzeneği; motor ve yükten oluşmaktadır. Burada yük olarak DC makine kullanılmıştır. Motor ve yük arasındaki şaftın hizalanması için *Shaftalign Os3* lazer şaft hizalama sistemi kullanılmıştır. Böylece rulman arızası durumuna ait motor akım verisi şaft eksen kaçıklığı sorunu olmadan izlenebilmiştir. Çalışmada kullanılan motorun parametreleri Tablo 1'de görülmektedir.

Tablo 1. ŞKDMSM parametreleri

Güç	Frekans	Gerilim(∆/Y)	Akım(Δ/Y)	Hız	Cosø	Kutup Sayısı	% Verimi	Koruma Sınıfı
2,2kW	50Hz	230/400V	7,10/4,10A	1500d/d	0,85	4	91,2	IP 54

Yapılan uygulamalarda 4 farklı tipte rulman arızası üzerinde çalışılmıştır: dış bilezik (DB) arızası, iç bilezik (DB) arızası, kafes (KA) arızası ve hasarlı bilye (HB) arızası. Bu arızalar her bir rulmanda yapay olarak üretilmiştir. Şekil 8'de arızalı rulmanlara ait resimler görülmektedir [23].



Şekil 8. Arızalı rulmanlar

Motorun normal durumu için EWMA grafik analiz sonuçları Şekil 9'de görülmektedir. Sağlam durumda motor akım verisinden hesaplanan 10 adet veri noktasının tamamı MÇ üzerinde ve AKL ve ÜKL aralığındadır. Bu durum sürecin normal olduğunu göstermektedir.



Şekil 9. Normal durum ve arızalı durum için Ewma grafik analiz sonuçları

Motora ait Sağlam durum-HB durum grafiği Şekil 10'da sağlam durum-DB arızası durumu grafiği Şekil 11'de, sağlam durum-İB arızası durumu grafiği Şekil 12'de ve sağlam durum-KA arızası durumu grafiği Şekil 13'te görülmektedir. Bu grafiklerde ilk 10 veri noktası sürecin normal olduğunu 11-20 aralığındaki veri noktası arıza durumunu göstermektedir. HB, DB, İB ve KA durumlarında veri noktalarının tamamı ÜKL 'nin dışında çıkarak sürecin normalin dışında olduğu görülmektedir. Limit değerleri istatistiksel proses kontrol hesaplamaları sonucunda  $\pm 0.02$ şeklindedir.



Şekil 9. Hasarlı Bilye Arızası Ewma Grafiği



The International Conference

of Materials and Engineering Technology

UWE Bristol

Şekil 10. Dış Bilezik Arızası Ewma Grafiği



368



Şekil 12. Kafes Arızası EWMA Grafiği

## 4. Sonuçlar

Bu çalışmada kullandığımız mikro denetleyicinin düşük maliyetli olması kolayca yeniden programlamaya müsaade etmesi ve esnek yazılımı, sistemin erişilebilir olmasını sağlamaktadır. Ancak düşük örnekleme frekansına sahip olmasından dolayı frekans boyutundaki analizlerde yetersiz kalabilmektedir. Bu sorunun üstesinden gelebilmek için İPK yöntemi kullanılmıştır. Bu yöntem sinyaldeki çok küçük değişiklikleri dahi fark edebilmektedir. Böylece çok düşük seviyelerdeki örnekleme frekanslarında motor arızası tespit edilebilmekte ve gerekli önlemler alınabilmektedir.

Hazırlamış olduğumuz bu sistem düşük maliyetli mikro denetleyici ve ekipmanlar kullanarak motor arıza teşhisinin yapılabileceğini göstermektedir. Dezavantaj olarak görünen düşük örnekleme frekansı ise uygulanan İPK yöntemiyle ortadan kaldırılmıştır. Kullanılan mikro denetleyicinin açık kaynak olması gelecekte ihtiyaç duyulabilecek yeni uygulamalar için yeniden düzenleme imkânı tanımaktadır. SCADA ara yüzü ve veri tabanı da taleplere göre yeniden düzenlenebilir şekildedir. Talep edilen her türlü görüntüleme, analiz ve raporlama yazılım ile tekrardan düzenlenip oluşturulabilmektedir.

Hazırlanmış olan bu çalışmada mikro denetleyici olarak kullanılan Arduino Mega yeterli bir şekilde görevini yerine getirmiştir. Ancak daha kapsamlı analiz ve senaryolar için düşük maliyetli yüksek örnekleme frekansına sahip mikro denetleyiciler kullanılabilir. Bu durum sistemin daha karmaşık analizler yapabilmesini sağlarken düşük maliyetli özelliğini devam ettirebilmektedir.

Sonuç olarak elde edilen sonuçlar; tasarlanan SCADA tabanlı online durum izleme donanımlarıyla izlenen ŞKDMSM akım sinyallerinin, önerilen İPK tabanlı sinyal analizi yoluyla, rulman arızalarının başarılı bir şekilde tespiti edilebileceğini göstermiştir.

## Kaynaklar

- 1. Yu, M., Tao, X., Xin, D., and Xin, Z., Fault diagnosis and numerical simulation of broken rotor bars for small cage induction motors, in 2017 Chinese Automation Congress (CAC), pp. 5355-5359, 2017.
- 2. Kim, K., Kim, K., Kim, H. J., and Lee, J., Demagnetization Analysis of Permanent Magnets According to Rotor Types of Interior Permanent Magnet Synchronous Motor, IEEE Transactions on Magnetics, **45**, pp. 2799-2802, **2009**.



The International Conference of Materials and Engineering Technology

- Nandi, S., Toliyat, H. A., and Li, X., Condition Monitoring and Fault Diagnosis of Electrical Motors—A Review, IEEE Transactions on Energy Conversion, 20, pp. 719-729, 2005.
- **5.** Ferhat, Ç., Asenkron Motorlarda Gerçek Zamanlı Durum İzleme ve Arıza Tespiti, Gaziosmanpaşa Bilimsel Araştırma Dergisi, **7**, pp. 12-24, **2018**.
- 6. Proakis, J. G. and Manolakis, D. G., Digital Signal Processing: Principles, Algorithms and Applications, Pentice Hall, **2007**.
- 7. Boashash, B., "Time-Frequency Signal Analysis. Advances in Spectrum Estimation and Array Processing," S. Haykin, Ed., ed: Prentice- Hall, **1990**, pp. 418-517.
- 8. Eser, E., "Asenkron Motorlarda İstatistiksel Proses Kontrol Yöntemi İle Arıza Tespiti," Yüksek Lisan Tezi, Elektrik ve Elektronik Mühendisliği, Gaziosmanpaşa Üniversitesi, Tokat, Türkiye, **2018**.
- **9.** Bostan, H., "İstatistiksel proses kontrol tekniklerinin motor yenileştirme sürecinde kullanımı," Fen Bilimleri Enstitüsü, Balıkesir Üniversitesi **2010**.
- 10. Djmal, I. B., Mansouri, M., Nounou, M., Nounou, H., and Hamida, A. B., Fault detection using UKF-based optimized EWMA method in wastewater treatment plant, in 2018 4th International Conference on Advanced Technologies for Signal and Image Processing (ATSIP), pp. 1-6, 2018.
- **11.** Mirimani, S. M., Vahedi, A., Marignetti, F., and De Santis, E., Static eccentricity fault detection in single-stator–single-rotor axial-flux permanent-magnet machines, IEEE Transactions on Industry Applications, **48**, pp. 1838-1845, **2012**.
- Soreshjani, M. H. and Haghparast, M., Classical Direct Torque Control performance of Line Start PM Synchronous Motor for different conditions, International Transactions on Electrical Energy Systems, 25, 2014.
- 13. Stapenhurst, T., "Mastering statistical process control," ed Routledge, 2013.
- 14. Maraş, S. and Arslan, H., Düz Dişli Çark Sistemindeki Aşınma Hatasının İstatistiksel Proses Kontrol Metodu İle Belirlenmesi, Pamukkale Üniversitesi Mühendislik Bilimleri Dergisi, 20, pp. 9-14, 2014.
- **15.** Birgören, B., "İstatistiksel kalite kontrolü," ed Ankara: Nobel Akademik Yayıncılık, **2015**, pp. 95-111.
- **16.** Niezgoda, J., The use of statistical process control tools for analysing financial statements, Folia Oeconomica Stetinensia, **17**, pp. 129-137, **2017**.
- Şenol, Ş., "İstatistiksel Kalite Kontrol," ed: Nobel Akademik Yayıncılık, 2012, pp. 69-176.
- Fugate, M. L., Sohn, H., and Farrar, C. R., Vibration-based damage detection using statistical process control, Mechanical Systems and Signal Processing, 15, pp. 707-721, 2001.



- **19.** Zerbato, C., Furlani, C. E., Silva, R. P. d., Voltarelli, M. A., and Santos, A. F. D., Statistical control of processes aplied for peanut mechanical digging in soil textural classes, Engenharia Agrícola, **37**, pp. 315-322, **2017**.
- **20.** Gozuoglu, A. and Ozgonenel, O., Training Set Design for Smart Grids and Scada Co-Simulations, in 2019 7th International Istanbul Smart Grids and Cities Congress and Fair (ICSG), pp. 124-128, **2019**.
- **21.** Anonymous. (**2019, July 19**). Arduino Mega 2560 R3 Documentation [Online]. Available: <u>https://store.arduino.cc/usa/mega-2560-r3</u>
- **22.** Gözüoğlu, A., "Akıllı Şebekelerde Kullanılan Scada Sistemlerinin Yazılım ve Donanımsal Olarak Kulanılması," Yüksek Lisan Tezi, Elektrik ve Elektronik Mühendisliği, Ondokuz Mayıs Üniversitesi, Samsun, Türkiye, **2018**.
- **23.** Cruz, S. M. and Cardoso, A. M., Stator winding fault diagnosis in three-phase synchronous and asynchronous motors, by the extended Park's vector approach, IEEE Transactions on industry applications, **37**, pp. 1227-1233, **2001**.

# CONSISTENCY LIMITS OF CLAY WITH CRUSHED WASTE ASPHALT

The International Conference

of Materials and Engineering Technology

#### Ali Firat Cabalar

A need to recycle the increased amount of solid waste materials has recently led researchers to study their potential uses for various engineering purposes. Reuse of the reclaimed asphalt pieces is one of the best potential sources for geotechnical engineering. This study investigates the effects of crushed asphalt pieces produced from reclaimed asphalt aggregates on the consistency limits of low plastic (CL) type clay. The experimental program included the findings of liquid limit and plastic limit tests. The results showed that the liquid limits and plasticity index of natural clay decrease as the amount of crushed asphalt pieces increase.

Keywords: Clay, crushed asphalt pieces, consistency limits.

#### Introduction

The studies for clay stabilization using solid waste materials achieve three main advantages; (i) improving the geotechnical properties of clay, (i) decreasing environmental pollution, and (iii) economic benefits. The fact is that investigation of consistency limits (Liquid limit, Plastic limit, Plasticity index) is considered as an initial indicator of change in the geotechnical properties, when studying the use of any additive in a soil for various engineering purposes (Parsons et al., 2007; Okagbue, 2007; Cristelo et al., 2016). For example, Saltan et al. (2011) studied the possible use of pumice waste as a stabilizing additive to be used in road pavements. The consistency limits values they obtained showed that the pumice waste could be used as an alternative stabilizing agent in clays. Rifai et al. (2014) investigated the use of volcanic ash as a soil stabilization material. The tests including consistency limits verify the use of it to improve soft soils.

The present study aims to investigate the effects of crushed asphalt pieces produced from reclaimed asphalt aggregates on the consistency limits of a low plastic (CL) clay.

#### **Experimental Study**

The soil used during the experimental studies was quarried from the University of Gaziantep campus, Turkey. The liquid limit (LL), and plastic limit (PL) tests were carried out using BS 1377 and ASTM D4318-2000, respectively. The soil according to ASTM D2487-2000 and AASHTO was classified as CL and A-7-6, respectively. The crushed waste asphalt (CWA) used in the study was produced by destruction of old asphalt roads. The dragged asphalt aggregates are made of aggregate particles coated with bitumen (See the Figure 1).


Figure 1. Picture of the dragged asphalt aggregates used during the tests.

#### **Results and Discussion**

The Figure 2 displays the change in consistency limits with amount of crushed asphalt additions in the clay. It is clearly seen that there is a decrease in liquid limit values with an increase in the amount of crushed asphalt pieces in the mixtures. The values of the plasticity limit were found to increase up to 10% addition of asphalt pieces, and then decrease to a value lower than that of clean clay. Accordingly, change in the liquid limit and plasticity limit values resulted in to a significant decrease in the values of the clay-asphalt pieces mixtures. The author's interpretation is that the asphalt pieces addition in clay converts the behavior from cohesive to a cohesionless soil despite of the bitumen presence. Then, presence of the bitumen seems to be not very effective on the results. The present study also reveals that the classification of the clay changed from low plastic (CL) to silt clay (ML) by addition of crushed asphalt pieces in to the clay (Table 1).



Figure 2. Consistency limits of the clay with crushed asphalt pieces.



Specimens	Content (%)	Liquid limit (%)	Plastic limit (%)	Plasticity index (%)	Classification USCS	Classification AASHTO
Clean clay	0	42	25	17	CL	A-7-6
CWA	5	41	27	14	ML	A-7-6
CWA	10	40	27.5	12.5	ML	A-7-6
CWA	15	36	25	11	ML	A-6
CWA	20	30	22	8	ML	A-4

 Table 1. Consistency limits and classification of the specimens tested.

## Conclusions

This laboratory work investigates the effects of crushed asphalt pieces, which has been produced from the dragged asphalt aggregates, on the consistency limits (liquid limit, plastic limit) of a low plastic clay quarried from the University of Gaziantep campus. Following conclusions could be drawn at the end of this paper.

- The liquid limits and plastic limits of the mixtures decrease with the addition of crushed asphalt pieces.
- The crushed asphalt pieces changed the classification of soil from low plastic (CL) to silty clay (ML), possibly due to the decrease in cohesion.

### References

1. ASTM, American Society for Testing and Materials (2010). Standard test methods for liquid limit, plastic limit, and plasticity index of soils.D4318-2010 West Conshohocken, PA.

2.ASTM, American Society for Testing and Materials (2010). Standard test methods for laboratory determination of water (moisture) content of soil and rock by mass.D2216-2010West Conshohocken, PA.

3.Cristelo, N., Vieira, C.S. and De Lurdes Lopes, M. (2016). Geotechnical and geoenvironmental assessment of recycled construction and demolition waste for road embankments. *Procedia engineering*, 143, 51-58.

4.Okagbue, C. (2007). Stabilization of Clay Using Wood Ash. Journal of Materials in Civil Engineering: Vol. 19, Geotechnical aspects of Stabilized Materials, (pp.14-18).

5.Parsons, R. and Kneebone, E. (2004). Use of Cement Kiln Dust for the Stabilization of Soils." Geotechnical Engineering for TransportationProjects: (pp.1124-1131)

6. Saltan, M., Kavlak, Y., and Ertem, F. (2011). Utilization of Pumice Waste for Clayey Subgrade of Pavements. Journal of Materials in Civil Engineering.

7. Rifai and Yasufuku, N. (2014). Effect of Volcanic Ash Utilization as Substitution Material for Soil Stabilization in View Point of Geo-Environment."Ground Improvement and Geosynthetics: pp.138-147.

8. Yoshizawa S, Tanaka M, Shekdar AV (2004). Global Trends in WasteGeneration. In: Recycling, Waste Treatment and Clean Technology, TMS Mineral, Metals and Materials Publishers, Spain. pp. 1541-1552.

## THERMAL MODELING AND EXPERIMENTAL ANALYSIS OF FRICTION WELDED AA1100 ALUMINUM ALLOY WITH MILD STEEL

The International Conference of M

aterials and Engineering Technology

# FARES KHALFALLAH<sup>\*1,2</sup>, ZAKARIA BOUMERZOUG<sup>2</sup>, ELHADJ RAOUACHE<sup>3</sup>, SELVARAJAN RAJAKUMAR<sup>4</sup>

<sup>1</sup>University of M'sila, Faculty of Sciences, Department of Physics, M'sila, ALGERIA <sup>2</sup>University of Biskra, Faculty of Science and Technology, Department of Mechanical Engineering, Biskra, ALGERIA

<sup>3</sup> University of Bordj Bou Arreridj, Faculty of Sciences and Technology, Civil Engineering Department, Bordj Bou Arreridj, ALGERIA

<sup>4</sup> Annamalai University, Centre for Materials Joining & Research, Chidambaram-INDIA

#### Abstract

In this study, the effect of friction time on the properties of AA1100 aluminum alloy-mild steel jointso obtained by rotary friction welding was investigated. The welding process was developed by a continuous drive friction welding machine. A series of experimental tests, such as microhardness measurements, strength tests and temperature measurements were performed in conjunction with a thermal modeling to predict the temporal variation of temperature in the aluminum part. The numerical simulation was developed by Finite Element Analysis (COMSOL) software, and the numerical predictions were compared with thermocouple data recorded from real welds conducted under identical conditions. The results show that the properties of joints are affected by the friction time variations. Thermal analysis also helped to understand the relationship between the maximum temperatures reached and the mechanical properties of the joints as well as the quality of the weld. From the simulation results, the predicted and measured temperature values are in fair agreement.

**Keyword:** Rotary Friction Welding, Thermal modiling, Mild steel, AA1100 Aluminum alloy, Effect of friction time.

#### **1.Introduction**

In recent years, the joining of dissimilar materials such as aluminum and steel has received considerable attention in the automotive industry with the aim of reducing the vehicle weight, energy saving and environmental protection by exploiting the advantages of steel, such as good mechanical properties, and those of aluminum, such as low weight and good corrosion resistance [1,2]. However, due to the formation of brittle intermetallic compounds (IMC), porosity and other solidification issues, the joining of dissimilar materials by conventional welding processes is not efficient and thus advanced joining techniques are required [3]. The rotary friction welding (RFW) is one of the most suitable processes for joining industrially important dissimilar materials such as aluminum and steel. It is a solid state welding process requires frictional heat to weld the components and requires no melting of the base materials in the weld zone [4]. It can be applied in two ways; continuous drive friction welding and inertia friction welding [5]. In continuous drive

process, the welding heat was generated through mechanical friction between a rotating and a stationary work piece. A several welding parameters affect the quality of friction welds, such as friction time, forging time, friction pressure, forging pressure and rotational speed [6].

The International Conference of Materials and Engineering

While, the RFW process is becoming increasingly popular for joining similar and dissimilar materials, it has attracted the attention of many researchers in recent decades, and numerous studies have been conducted to develop a numerical model of continuous drive friction welding of various materials [7-12].

In the present study, an experimental work was made to join two dissimilar metals, AA1100 aluminum alloy and mild steel. The joints were developed by continuous drive friction welding process under various friction times. The scientific goals of this work are to understand the friction time effects on weld properties and to predict thermal behavior of joints during friction welding process.

#### 2. Materials and Methods

#### 2.1. Experimental welding Process

The materials used in the experimental work were aluminum AA1100 and mild steel. They were cylindrical rods of 70 mm in length and with 12 mm in diameter for aluminum pieces and 10 mm in diameter for mild steel pieces. Tables 1 and 2 present the chemical compositions and physical properties of these two dissimilar materials.

<b>Table 1.</b> Chemical composition (wt %) of base metals determined by XRF technique.										
Materials	С	Si	S	Р	Mn	Cu	Mg	Zn	Fe	Al
AA 1100		0.57		0.04		0.01	0.53	0.02	0.23	98.6
Mild Steel	0.39	0.28	0.03	0.03	0.9	0.14			98.2	0.03

Table 1. Chemical	composition	(wt %) of	base metals	determined by	/ XRF technique
-------------------	-------------	-----------	-------------	---------------	-----------------

Table 2. Physical properties of base metals.					
AA 1100	Mild Steel				
2700	7900				
640 - 660	1420 - 1460				
220	52				
900	470				
	0.6				
	AA 1100 2700 640 - 660 220 900				

The welding process was carried out using a continuous drive friction welding machine (Rexroth, R.V. Machine tools) as shown in Fig. 1. Before the welding process, the ends of samples were polished and cleaned to reduce the effect of contaminants, especially grease, which can affect the quality of joints. The welding parameters used in this work are listed in Table 3.

The temperature variations during the welding process were measured by K-type thermocouple attached to the stationary aluminum rod at distance of 20 mm from the interface (Fig. 1).

The successfully welded samples were machined and prepared for mechanical testing. The tensile strength tests were done using a 100 kN, servo controlled universal testing machine (Make: FIE-Bluestar, Model: UNITEK 94100) with a crosshead speed of 0.5 mm/min.

Technology



aterials and Engineering Technology

Figure 1. General view of a part of the RFW machine

Table 3. Experimental welding parameters					
Sample N°	01	02	3		
Rotational speed (rpm)	800	800	800		
Friction pressure (MPa)	31	31	31		
Friction time (s)	3	5	7		
Forging pressure (MPa)	192	192	192		
Forging time (s)	5	5	5		

For micro-hardness measurements, the welded specimens were sectioned and polished. The measurements were recorded using a micro-hardness tester (Make: Shimadzu, Model: HMV-2T) at 200 g load.

## 2.2. Thermal modeling of RFW

The aim of this numerical work is to simulate thermal behavior during welding cycle. The *heat transfer in solids* and *the mechanics structural* modules are used, in order to give better temperature profiles. A tow dimensional model consisting of two parts was created in COMSOL Multiphysics software.

#### **Boundary conditions**

The detailed boundary conditions are mentioned in fig. 2. The equation for thermal modeling of this problem is given by Fourier heat conduction phenomenon, which is written as [13]:







Where: *T* is the temperature, *Q* is the heat generation rate, *k* is the thermal conductivity, *t* is the time,  $C_p$  is the specific heat capacity and  $\rho$  is the materials density. The heat generation rate by friction is given by [14]:

 $Q = \mu.\omega.p.r$ 

(2)

The International Conference

Where: Q (W/m<sup>2</sup>) is the heat generation rate,  $\omega$  (rpm) is the angular velocity, r (m) is the work piece radius, and p (Pa) is the applied pressure.

## Geometry

A 2D axisymmetric geometry is used in the modeling study. The radius of base metals rods were fixed at 5 mm, and their lengths are 70 mm.

### 3. Results and Discussion

### 3.1. The effect of friction time on temperature variations

Figure 3 shows a comparison of thermal histories in Aluminum side, obtained from experiments and COMSOL simulation at various friction times. The large agreement between the experimentally recorded temperatures and the numerical values indicate that the COMSOL model can be used to predict temperature profiles in the aluminum rod during the friction welding process. At a distance of 20 mm from the interface of the joint, the maximum temperature in the aluminum side of samples 1, 2 and 3 are 208 °C, 242 °C and 347 °C. Both, experiment and simulation demonstrate that increasing the friction time result in an increase of the welding temperature.

## 3.2. The effect of friction time on tensile strength

Tensile tests were performed in order to investigate the mechanical properties of friction welded joints. Figure 4 shows the samples after tensile testing. It can be observed that s all the joints developed with various friction times exhibited a ductile rupture (necking shape), and fractured in the aluminum base metal.

Figure 5 shows the relation-ship between the tensile strength, the peak temperature and the friction time. The tensile strength is initially increased as the friction time increased. It reached a maximum value of 178 MPa with a friction time value of 5 s and then the joint strength was degraded by increasing the friction time above 5 s. However, the peak temperature values increases with the increase of the friction time as shown in Fig. 5.

This is means that the quantity of heat generated by the friction increased with increasing friction time. The decrease in tensile strength of joints welded under longer friction time is related to the formation of the brittle intermetallic layer at the weld interface. Although the formation of a thin intermetallic layer is necessary to obtain high joint strength, the formation of a large layer reduces the joint strength [15].

Technology



**Figure 3**. Thermal histories in aluminum rods, obtained from experiments and COMSOL simulation. (a) friction time = 3 s, (b) friction time = 5 s, (c) friction time = 7 s.



Figure 4. Photograph of samples after tensile test

UWE Bristol

Figure 5. Tensile strength and peak temperature variations versus friction time

379



## **3.3.** The effect of friction time on hardness

In order to study the variations in hardness along the weld zone, Vickers microhardness tests were carried out. Figure 6 shows the distribution of hardness around the weld zone of welded samples at different friction times. It is clear that the hardness of welded mild steel is higher near the interface when compared to the welding interface. This is due to the strain hardening effect during the friction process at the interface. However, the hardness of the welded aluminum alloy decreases a little because of the recrystallization effect resulting from friction heat and the deformation during the friction process. Near the weld interface, the width of the steel hardening region and the aluminum softening region increased with the increasing friction time because the amount of heat generated by the friction increased.



Figure 6. Distribution of microhardness across the weld zone of welded samples

## 4. Conclusions:

In the present work, mild steel and AA1100 aluminum alloy joints were developed successfully by rotary friction welding. In addition, a numerical simulation by COMSOL was performed to predict the temperature variations during the welding process. This study led to the following results:

- The predicted values of temperature are similar to those recorded by thermocouple during the welding process, and the shapes of the profiles are also similar.
- The peak temperature and the heat generation increase with increasing friction time, which indicates that the thermal behavior of welded joints is closely related to the friction welding parameters.
- The friction time has a great influence on the joints strength. The tensile strength increased and reached a maximum, then decreased again as the friction time increased. A longer friction time increased heat generation and caused excessive formation of the brittle intermetallic layer.
- Near the weld interface, the hardness of the steel increased and the hardness of the aluminum decreased. The width of the hardening zone and the softening zone increased with increasing friction time.

#### **References:**

1. Haghshenas, M., and Gerlich, A.P., Joining of Automotive Sheet Materials by Friction-Based Welding Methods: A Review. Eng. Sci. Technol. Int J., **2018**, 21:130-148.

The International Conference of Materials and Engineering Technology

- Fukumoto, S., Tsubakino, H., Okita, K., Aritoshi, M., and Tomita, T., Amorphization by Friction Welding between 5052 Aluminum Alloy and 304 Stainless Steel. Scr. Mater., 2000,42:807-812.
- 3. Sahin, M., Joining of Aluminium and Copper Materials with Friction Welding. *Int. J. Adv. Manuf. Tech.*, **2010**, 49:527-534.
- Selvamani, S.T., Palanikumar, K., Umanath, K., and Jayaperumal, D., Analysis of Friction Welding Parameters on the Mechanical Metallurgical and Chemical Properties of AISI 1035 Steel Joints. *Mater. Design.*, 2015, 65:652-66.
- 5. Li, W., Vairis, A., Preuss, M., and Ma, T., Linear and Rotary Friction Welding Review. *Int. Mater. Rev.*, **2016**, 61:71-100.
- Sahin, M., Erol Akata, H., and Ozel, K., An Experimental Study on Joining of Severe Plastic Deformed Aluminium Materials with Friction Welding Method. *Mater. Design.*, 2008, 29(1):265-274.
- 7. Emel, T., Jerry, E.G., and John, C.L., Dissimilar Friction Welding of 6061-T6 Aluminum and AISI 1018 Steel: Properties and Microstructural Characterization. *Mater. Design.*, **2009**, 31:2305-2311.
- Kimura, M., Kusaka, M., Kaizu, K., Nakata, K., and Nagatsuka, K., Friction Welding Technique and Joint Properties of Thin–Walled Pipe Friction–Welded Joint between Type 6063 Aluminum Alloy and AISI 304 Austenitic Stainless Steel. *Int. J. Adv. Manuf. Tech.*, 2016, 82:489-499.
- 9. Asif, M.M., Shrikrishana, K. A., and Sathiya, P., Finite Element Modeling and Characterization of Friction Welding on UNS S31803 Duplex Stainless Steel Joints. Eng. Sci. Technol. Int J., **2015**, 18(4):704-712.
- Dawood, A.B., Butt, S.I., Hussain, G., Siddiqui, M.A., Maqsood, A., and Zhang, F., Thermal Model of Rotary Friction Welding for Similar and Dissimilar Metals. Metals, 2017, 7:1-14.
- Seli, H., Ismail, A. I. M., Rachman, E., and Ahmad, Z. A., Mechanical Evaluation and Thermal Modeling of Friction Welding of Mild Steel and Aluminum. J. Mater. Process. Technol., 2010, 210(9):1209-1216.
- 12. Alves, E.P., Toledo, R.C., Piorino, N., Francisco, B., Fabio G., and Ying An, C., Experimental Thermal Analysis in Rotary Friction Welding of Dissimilar Materials. J. Aerosp. Technol. Manag, **2019**, 11:e4019.
- 13. Rajesh Jesudoss Hynes, N., Nagaraj, P., Palanichamy, R., Arumugham, C.A.K., and Angela Jennifa Sujana J., Numerical Simulation of Heat Flow in Friction Stud Welding of Dissimilar Metals, Arab. J. Sci. Eng., **2014**, 39:3217-3224,
- Seli, H., Awang, M., Ismail, A. I. M., Rachman, E., and Ahmad, Z. A., Evaluation of Properties and FEM Model of the Friction Welded Mild Steel-Al6061-Alumina. Mater. Res., 2012, 16(2):453-467.
- Fukumoto, S., Tsubakino, H., Okita, K., Aritoshi, M., and Tomita, T., Microstructure of Friction Weld Interface of 1050 Aluminium to Austenitic Stainless Steel. Mater. Sci. Tech., 1998, 14:333-338.

# ADSORPTION STUDIES OF CIBACRON BLUE ONTO BOTH UNTREATED AND CHEMICALLY TREATED PISTACHIO SHELL POWDER FROM AQUEOUS SOLUTIONS

The International Conference

aterials and Engineering Technology

## ABDULAZİZ KAYA\*1

<sup>1</sup>Gaziantep University, Engineering Faculty, Department of Metallurgical and Materials Engineering, Gaziantep, TURKEY.

#### Abstract

Pistachio shells, separated from locally harvested pistachios, were first cleaned and dried, and then crushed by using a hammer mill to produce particles less than 1.5 mm. Resulting pistachio shell powder (PSP) was treated with first aqueous HCl solution and then with aqueous NaOH solution to yield chemically treated pistachio shell powder (CTPSP). Batch adsorption of Cibacron blue (CB) dye onto both PSP and CTPSP surfaces in aqueous solutions were studied by UV Spectroscopy under different solution pH values. It was observed that there was no significant adsorption of CB dye onto PSP and CTPSP surfaces above pH=2. However, when the pH of the solution was lowered to pH=2, considerable adsorption of CB dye onto both PSP and CTPSP surfaces were observed. When CB dye adsorption onto two adsorbents was compared, CB dye adsorption onto CTPSP was significantly higher than that onto PSP surfaces.

Keyword: Pistachio shell powder, textile dye, adsorption.

#### **1. Introduction**

Textile, paper, carpet and printing industries generate industrial wastewater that contains a high concentration of coloured organic compounds. Due to presence of dyes, which have complex aromatic molecular structures of synthetic origin, it is not easy to degrade this effluent. Majority of the dye compounds are toxic and often carcinogenic. Water streams polluted with wastewater containing dye compounds into water streams may pose a serious threat to public health and the aquatic community. Recently, increased awareness of environmental aspects has forced many countries to impose stringent environmental laws and more attention has been directed towards development of treatment methodologies [1]. Hence, eliminating the dyes in the wastewater is important before the wastewater is discharged into the environment.

Synthetic dyes exhibit recalcitrance towards removal/biodegradation by conventional biological wastewater treatment methods [2]. Several methods exist for treating dyes-containing effluents including adsorption [3-5], membrane filtration [6], flocculation [7], ozonation [8], Fenton [9] and photocatalytic degradation [10]. Adsorption, one of many techniques used for dye removal, generally involves the adsorption of dye onto activated carbon. Activated carbon utilization is hindered by the costs involved, because activated carbon is expensive [11]. Therefore, several studies focused on investigation of the inexpensive and efficient alternative materials to activated carbon such as bagasse pith [12], wood [13], peat [14], soil [15] and rice husk [16].

Pistachio (Pistacia vera L.) is readily available in greater quantities in South East Anatolia Region of Turkey. As an agricultural residue of pistachio production, pistachio shells are generally discarded as a waste [17]. The main purpose of this study is to determine the adsorption abilities of the pistachio shell with or without chemical treatment for the elimination of Cibakron blue (CB) from aqueous solutions. The influence of pH on the CB adsorption onto pistachio shell under equilibrium conditions was investigated.

The International Conference of Materials and Engineering Technology

### 2. Materials and Methods

All the reagents used were of analytical grade. Cibakron blue was supplied by Ciba Specialty Chemicals, Ltd. Pistachio shells were obtained from a local pistachio processing factory (Gaziantep, Turkey). Then, they were washed thoroughly in deionized water in order to remove surface impurities and dried in ambient air for a period of nearly a week. Cleaned and dried pistachio shells were crushed, grounded, and sieved to obtain a particle size less than 1.5 mm. The resulting powder is pistachio shell powder (PSP) and this powder was used in experimental studies. For chemical treatment of PSP, a modified procedure of Devi et al.[18] was used. First, PSP was refluxed in 0.1 M HCl for three hours. After decanting the clear solution, the residue was refluxed again in 0.1 M NaOH for three hours. The treated powders were washed several times with deionized water until the filtrate has a neutral pH. The materials were soaked in deionized water for sufficient amount of time in order to protonate the surface which would make more sorption sites available and later dried at 40°C for 48 h. This procedure resulted in chemically treated pistachio shell powder (CTPSP). Both PSP and CTPSP have been employed for further experiments.

To evaluate the efficiency of prepared powders as an adsorbent, laboratory batch mode studies were conducted using a tube rotator at a constant speed of 50 rpm. The effect of initial pH values (pH=2, 6 and 10) on the adsorption process was investigated. The pH of each solution was adjusted to a predetermined value with 0.1 M HCl and 0.1 M NaOH aqueous solutions. For each adsorption experiment, a fixed mass of PSP and CTPSP (0.1 g) was added to a falcon tube and then 10 mL of 100 mg·L<sup>-1</sup> CB solution with different pH values (2, 6, and 10) was poured into the falcon tube and the suspension was put on the rotator for mixing for 12 h. After agitation, the dye solutions were separated from the adsorbent by centrifugation for 30 min at 4500 rpm. The residual dye concentrations in the supernatant solutions were determined by monitoring the absorbance changes at the wavelength of maximum absorbance using a UV-Visible spectrophotometer (Shimadzu model: UV 1800).

## 3. Results and Discussion

UV spectra for aqueous CB solutions at pH=2 before and after adsorption experiments were provided in Figure 1. It can be seen that after adsorption onto both PSP and CTPSP surfaces, absorbance values at maximum wavelength ( $\lambda$ =706 nm) shift to lower values. This indicates that CB dye molecules adsorbed onto both adsorbents. When compared, absorbance value of Cibakron blue after adsorption onto CTPSP is even lower than that onto PSP. This result points out that CB adsorption onto CTPSP occurs in higher amounts compared to the adsorption onto TSP.



**Figure 1.** UV spectra of aqueous solutions of CB at pH=2. Symbols correspond to (---) 100 mg·L<sup>-1</sup> CB in H<sub>2</sub>O, (- - -) 100 mg·L<sup>-1</sup> CB in H<sub>2</sub>O after mixing with PSP, and (-----) 100 mg·L<sup>-1</sup> CB in H<sub>2</sub>O after mixing with CTPSP.

UV spectra for aqueous CB solutions at pH=6 and 10 before and after adsorption experiments were provided in Figure 2 and 3, respectively. At both pH values, absorbance values at maximum wavelength ( $\lambda$ =615 nm) do not change significantly after adsorption onto PSP and CTPSP surfaces. This results indicates that at these pH values (pH=6 and 10), there is no noticeable adsorption onto PSP and CTPSP adsorbents.



**Figure 2.** UV spectra of aqueous solutions of CB at pH=6. Symbols correspond to (---) 100 mg·L<sup>-1</sup> CB in H<sub>2</sub>O, (- - -) 100 mg·L<sup>-1</sup> CB in H<sub>2</sub>O after mixing with PSP, and (.....) 100 mg·L<sup>-1</sup> CB in H<sub>2</sub>O after mixing with CTPSP.



**Figure 3.** UV spectra of aqueous solutions of CB at pH=10. Symbols correspond to (---) 100 mg·L<sup>-1</sup> CB in H<sub>2</sub>O, (- - -) 100 mg·L<sup>-1</sup> CB in H<sub>2</sub>O after mixing with PSP, and (-----) 100 mg·L<sup>-1</sup> CB in H<sub>2</sub>O after mixing with CTPSP.

**4. Conclusions:** In the present work, PSP and CTPSP, no-cost adsorbents, were investigated for the removal of CB from wastewater. The adsorption experimental investigation indicated that the pistachio shells can be used as alternative adsorbents for the removal of CB from aqueous environment at lower pH values. These investigations are quite useful in developing an appropriate technology for wastewater treatment. However, the adsorption characteristics largely depend on the type of dye and therefore these adsorbents should be tested for other dye types.

Acknowledgments: We thank Scientific Research Projects Governing Unit at Gaziantep University for funding the conference expenses.

#### **References:**

- 1. Amarasinghe, B.M.W.P.K., Jayasinghe, P.A. and Gunasekara, M.Y., Adsorption of Cibacron Blue Dye from Aqueous Solutions onto HCL Treated Waste Biomass. Engineer, **2007**, 40(3):7-15.
- 2. Velić, N., Stjepanović, M., Begović, L., Habuda-Stanić, M., Velić, D.V. and Jakovljević, T., Valorisation of Waste Wood Biomass as Biosorbent for the Removal of Synthetic Dye Methylene Blue from Aqueous Solutions. SEEFOR, **2018**, 9(2):115-122.
- 3. Dulman, V. and Cucu-Man, S.M., Sorption of some textile dyes by beech wood sawdust. Journal of Hazardous Materials, **2009**, 162:1457–1464.
- 4. Franca, A.S., Oliveira, L.S. and Ferreira, M.E., Kinetics and equilibrium studies of methylene blue adsorption by spent coffee grounds. Desalination, **2009**, 249:267–272.
- 5. Lataa, H., Gargb, V.K. and Gupta, R.K., Adsorptive removal of basic dye by chemically activated Parthenium biomass: equilibrium and kinetic modeling. Desalination, **2008**, 219:250–261.

6. Avlonitisa, S.A., Pouliosb, I., Sotirioua, D., Pappasa, M. and Moutesidisa, K., Simulated cotton dye effluents treatment and reuse by nanofiltration. Desalination, **2008**, 221:259–267.

The International Conference of Materials and Engineering Technology

- 7. Yue, Q.Y., Gao, B.Y., Wang, Y., Zhang, H., Sun, X., Wang, S.G. and Gu, R.R., Synthesis of polyamine flocculants and their potential use in treating dye wastewater. Journal of Hazardous Materials, **2008**, 152:221–227.
- 8. Yıldırım, A.Ö., Gül, Ş., Eren, O. and Kuşvuran, E., A Comparative Study of Ozonation, Homogeneous Catalytic Ozonation, and Photocatalytic Ozonation for C.I. Reactive Red 194 Azo Dye Degradation. Clean Soil, Air, Water, **2011**, 39(8):795–805.
- 9. Ramirez, J.H., Costa, C.A. and Madeira, L.M., Experimental design to optimize the degradation of the synthetic dye Orange II using Fenton's reagent. Catalysis Today, **2005**, 107–108:68–76.
- Singla, P., Sharma, M., Pandey, O.P. and Singh, K., Photocatalytic degradation of azo dyes using Zn-doped and undoped TiO2 nanoparticles. Applied Physics A Materials Science & Processing, 2014, 116:371–378.
- 11. Tezcan Un, U. and Ates, F., Low-cost adsorbent prepared from poplar sawdust for removal of disperse orange 30 dye from aqueous solutions. International Journal of Environmental Science and Technology, **2019**, 16:899–908.
- 12. Krishnan, K.A. and Anirudhan, T.S., A preliminary examination of the adsorption characteristics of Pb(II) ions using sulphurised activated carbon prepared from bagasse pith. Indian Journal of Chemical Technology **2002**, 9:32-40.
- 13. Ho, Y.S. and McKay, G., Kinetic Models For The Sorption Of Dye From Aqueous Solution By Wood. Process Safety and Environmental Protection, **1998**, 76(2):183-191.
- 14. Ho, Y.S. and McKay, G., Sorption of dye from aqueous solution by peat. Chemical Engineering Journal, **1998**, 70(2):115-124.
- 15. Ketelsen, H. and Meyer-Windel, S., Adsorption of brilliant blue FCF by soils. Geoderma, **1999**, 90:131–145.
- Han, R., Ding, D., Xu, Y., Zou, W., Wang, Y., Li, Y. and Zou, L., Use of rice husk for the adsorption of congo red from aqueous solution in column mode. Bioresource Technology, 2008, 99:2938–2946.
- 17. Şahin, Ö., Demirel, S. and Dilekoğlu, M.F., Removal of Pb(II) from aqueous solution by Antep Pistachio shells. Fresenius Environmental Bulletin, **2005**, 14(11):986-992.
- 18. Devi, N.S., Andal, N.M. and Vivithabharathi, K., Virtual Screening of Treated Pistachio vera Shell Powder as a Potential Sorbent in Sequestering Ubiquitous Divalent Metal Ions from Aqueous Matrices. Oriental Journal of Chemistry, **2018**, 34(1):352-361.

# SIMULATION OF BEHAVIOR OF CARBON/EPOXY AND GLASS/EPOXY COMPOSITES UNDER THE DROP WEIGHT IMPACT EVENT

The International Conference

aterials and Engineering Technology

## EYÜP YETER \*1, MEHMET HANİFİ DOĞRU <sup>2</sup>

<sup>1</sup>Gaziantep University, Aeronautics and Aerospace Faculty, Aircraft and Aerospace Eng. Department, Gaziantep, TURKEY. <sup>2</sup>Gaziantep University, Aeronautics and Aerospace Faculty, Pilotage Department, Gaziantep, TURKEY

#### Abstract

Mechanical behaviors of materials are determined by experimental tests. In cases where the test cannot be performed or a small number of samples can be tested, performing the numerical analysis is widely used for quick and reliable designs. This is particularly important for composite materials that are costly to design and manufacture but are widely used in many industries. Drop weight impact is a widely used test method to investigate the behavior of materials under the impact loads. Particularly in the areas where thin structures are frequently used, the behavior of materials under impact load is important. In this study, the behavior of Carbon/Epoxy and Glass/Epoxy composite plates under the drop weight impact load was simulated. Composite materials have been used to have symmetrical fiber orientations. The effects of different configurations of Carbon/Epoxy and Glass/Epoxy composite plate was increased by increasing the number of layers of the composite material and the effect of thickness increase on impact load was investigated. As a result, it is seen that the Carbon/Epoxy composite plate has higher impact resistance than the Glass/Epoxy composite plate. The maximum impact force increases with thickness increase for both materials.

Keyword: Composite materials, Drop weight, Impact.

#### **1. Introduction**

Materials have different behavior under the various loading conditions such as tension, compression, bending, twisting, buckling and impact. Especially, impact resistance of materials important for the structures that used in the thin forms. Composite materials are today's material chose for the industries that need high specific strength and modulus. Drop weight impact is a widely used test method to investigate the behavior of materials under the impact loads. In cases where the test cannot be performed or a small number of samples can be tested, performing the numerical analysis is widely used for quick and reliable designs. This is particularly important for composite materials that are costly to design and manufacture but are widely used in many industries.

Drop weight impact resistances of different materials have been researched by many researchers. Villavicencio and Guedes Soares [1] investigated drop weight impact characteristics of plates experimentally. From this study, it was seen that the plastic behavior of the samples is directly related to the restraint at the supports. Santiago et al. [2] investigated the effects of local impact loads on the fiber-metal laminates (FML) produced with the combinations of aluminum alloys and

polypropylene. Rawat et al. [3] studied failure characteristics of laminated composite plates using impactors having various shapes (hemispherical, spherical, oval shape, flat) under the impact loadings. The damage resistance of Steel and aluminum plates to a drop-weight impact event is researched numerically by Yeter and Doğru [4]. It was concluded that the damage resistance of the structural steel plate to a drop-weight impact event better than the aluminum plate used in this study. Yeter [5] investigated damage resistance of Armox 500T and Aluminum 7075-T6 plates subjected to drop-weight and ballistic impact loads. It was seen that the maximum impact loads of the Armox-500T target is higher than the Al7075-T6, and the deformation amount is less. n this study, the behavior of Carbon/Epoxy and Glass/Epoxy composite plates under the drop weight impact load was simulated. Composite materials have been used to have symmetrical fiber orientations. The effects of different configurations of Carbon/Epoxy and Glass/Epoxy composites on the drop weight impact loads considering 8 different inter-ply hybrid models are investigated. The thickness of the plate was increased by increasing the number of layers of the composite material and the effect of thickness increase on impact load was investigated.

The International Conference

#### 2. Materials and Methods

In this study, the behavior of Carbon/Epoxy and Glass/Epoxy composite plates under the drop weight impact load was simulated. Composite materials have been used to have symmetrical fiber orientations. Each lamina of composite material that has 0.25 mm thickness. The laminate is constructed by stacking several such laminae in the direction of the lamina thickness. The used configurations are listed in Table 1. Composite materials have been used to have symmetrical fiber orientations.

Configurations	thickness
$[0_{G}/90_{G}]_{s}$	1 mm
$[0_{\rm G}/90_{\rm G}/0_{\rm G}/90_{\rm G}]_{\rm s}$	2 mm
$[0_G/90_G/0_G/90_G / 0_G/90_G]_s$	3 mm
$[0_G/90_G/0_G/90_G/0_G/90_G/0_G/90_G]_s$	4 mm
$[0c/90_{\rm C}]_{\rm s}$	1 mm
$[0_{\rm C}/90_{\rm C}/0_{\rm C}/90_{\rm C}]_{\rm s}$	2 mm
$[0_{\rm C}/90_{\rm C}/0_{\rm C}/90_{\rm C}/90_{\rm C}]_{\rm s}$	3 mm
[0c/90c/0c/90c/0c/90c/0c/90c]s	4 mm

**Table 1** Composite and hybrid composite configurations

Also, the effects of different configurations of Carbon/Epoxy and Glass/Epoxy composites are considered. The used inter-ply hybrid models are listed in Table 2.

Technology



Configurations	Name
$[0_{C}/90_{G}/0_{G}/90_{G}]_{s}$	Model 1 (M1)
$[0_{\rm C}/90_{\rm C}/0_{\rm G}/90_{\rm G}]_{\rm s}$	Model 2 (M2)
$[0_{\rm C}/90_{\rm C}/0_{\rm C}/90_{\rm G}]_{\rm s}$	Model 3 (M3)
$[0_{\rm G}/90_{\rm C}/0_{\rm C}/90_{\rm C}]_{\rm s}$	Model 4 (M4)
$[0_{\rm G}/90_{\rm G}/0_{\rm C}/90_{\rm C}]_{\rm s}$	Model 5 (M5)
$[0_{\rm G}/90_{\rm G}/0_{\rm G}/90_{\rm C}]_{\rm s}$	Model 6 (M6)
$[0_{\rm C}/90_{\rm G}/0_{\rm C}/90_{\rm G}/0_{\rm C}/90_{\rm G}/0_{\rm C}/90_{\rm G}]$	Model 7 (M7)
$[0_{\rm G}/90_{\rm C}/0_{\rm G}/90_{\rm C}/0_{\rm G}/90_{\rm C}/0_{\rm G}/90_{\rm C}]$	Model 8 (M8)

## Table 2 Inter-ply hybrid composite configurations

The numerical models were developed using the transient finite element module of ANSYS. Simulation of the impact event is performed by the collision of two parts, namely, the impactor and the target plate.

A hemispherical impactor that has totally 5.5 kg mass and 8mm tip radius is used and as shown in figure 1 the distance between the impactor and target plate is 800 mm. In other words, the impactor is released from an 800 mm distance. The target plate dimensions are 150 mm in length and 100 mm in width. During the analyses, the target plate (as shown in figure 1) fixed from all edges.



Figure 1. Impactor and target plate properties



Carbon/Epoxy and Glass/Epoxy material properties of used Carbon/Epoxy and Glass/Epoxy are given in Table 3 and 4, respectively.

## Table 3. Material properties of Carbon/Epoxy

Property	Value	Unit
🔁 Density	1,518E-09	mm^-3 t
🔞 Orthotropic Secant Coefficient of Thermal Expansion		
🔁 Coefficient of Thermal Expansion		
Coefficient of Thermal Expansion X direction	-4,5E-07	C^-1
Coefficient of Thermal Expansion Y direction	3E-05	C^-1
Coefficient of Thermal Expansion Z direction	3E-05	C^-1
🔁 Orthotropic Elasticity		
Young's Modulus X direction	1,2334E+05	MPa
Young's Modulus Y direction	7780	MPa
Young's Modulus Z direction	7780	MPa
Poisson's Ratio XY	0,27	
Poisson's Ratio YZ	0,42	
Poisson's Ratio XZ	0,27	
Shear Modulus XY	5000	MPa
Shear Modulus YZ	3080	MPa
Shear Modulus XZ	5000	MPa
🔀 Orthotropic Stress Limits		
Tensile X direction	1632	MPa
Tensile Y direction	34	MPa
Tensile Z direction	34	MPa
Compressive X direction	-704	MPa
Compressive Y direction	-68	MPa
Compressive Z direction	-68	MPa
Shear XY	80	MPa
Shear YZ	55	MPa
Shear XZ	80	MPa

## Table 4. Material properties of Glass/Epoxy

Property	Value	Unit
🔁 Density	2E-09	mm^-3 t
📔 Orthotropic Elasticity		
Young's Modulus X direction	.50000	MPa
Young's Modulus Y direction	8000	MPa
Young's Modulus Z direction	8000	MPa
Poisson's Ratio XY	0,3	
Poisson's Ratio YZ	0,4	
Poisson's Ratio XZ	0,3	
Shear Modulus XY	5000	MPa
Shear Modulus YZ	3846,2	MPa
Shear Modulus XZ	.5000	MPa
2 Orthotropic Stress Limits		
Tensile X direction	1700	MPa
Tensile Y direction	35	MPa
Tensile 2 direction	35	MPa
Compressive X direction	-1000	MPa
Compressive Y direction	-120	MPa
Compressive 2 direction	-120	MPa
Shear XY	80	MPa
Shear YZ	46,154	MPa
Shear XZ	80	MPa



### **3.** Results and Discussion

Maximum impact forces and deformations are compared for the Carbon/Epoxy and Glass/Epoxy and their hybrid models. The impact forces for Carbon/Epoxy and Glass Epoxy for different thicknesses are given in Figure 2. As seen in the figure for all thickness, the impact forces of Carbon/Epoxy are higher than the impact forces of Glass/Epoxy. The maximum impact forces of Carbon/Epoxy for 1 mm plate is 1.6 times higher than the maximum impact force of Glass/Epoxy. This difference is 1.32, 1.38 and 1.45 for 2 mm, 3mm and 4 mm thickness, respectively.







(b)



Figure 2. Variation of Impact Force of Glass/Epoxy and Carbon Epoxy composites for different thickness. a) 1mm, b) 2 mm, c) 3mm d) 4 mm.

The effect of hybridization on the drop weight impact is investigated considering different hybrid models. In figure 3(a) comparisons of impact forces for the full Glass/Epoxy and hybrid models are given. These hybrid models are obtained adding 1, 2, and 3 layer carbon to the top and bottom symmetrically for full Glass/Epoxy composite. As seen in the figure, adding carbon layers increase impact loads and obviously impact resistance of laminate is increased. The maximum difference is between M3 and Glass/Epoxy. The impact force of M3 is nearly 1.33 times higher than the impact force of Glass/Epoxy.



In figure 3(b) comparison of impact forces for the full Carbon/Epoxy and hybrid models is given. These hybrid models are obtained adding 1, 2, and 3 layer glass to the top and bottom symmetrically for full Carbon/Epoxy composite. As seen in the figure, adding glass layers decrease the impact loads. The maximum reduction is with M6. The value of this reduction is nearly 30%



Figure 3. Variation of Impact Forces of a) Glass/Epoxy and Hybrid models b) Carbon/Epoxy and Hybrid models

Also, the hybrid configuration is obtained by adding one layer glass after one layer carbon until the getting 2mm thickness as in Model 7 and one layer carbon after one layer glass until they getting 2mm thickness as in Model 8. The comparison of these models is given in Figure 4(a). As seen in the figure, they have shown similar behavior under the drop weight impact load. In figure 4(b) comparison of one of these models with Full Glass/Epoxy and Carbon /Epoxy Laminates is



given. As seen in the figure, this model has the maximum impact loads between carbon/Epoxy laminate and Glass/Epoxy laminate. Also, it is observed in this figure, the impact forces of this model more close the full Glass/Epoxy laminate than full Carbon/Epoxy laminate.



(b)

**Figure 4.** Comparison of Impact Forces of Hybrid composite models a) M7 and M8 b) M7 and Glass/Epoxy and Carbon Epoxy.



Figure 5 gives deformation results for 2 mm Glass/Epoxy and Carbon Epoxy composite plates. The maximum deformation of Glass/Epoxy for 2 mm plate thickness is 1.22 times higher than the maximum deformation of the Carbon/Epoxy plate.



Figure 5. Deformation graph for 2 mm plate thickness a) Glass/Epoxy b) Carbon/Epoxy

In figure 6 comparison of deformation for the full glass/Epoxy and hybrid models is given. These hybrid models are obtained adding 1, 2, and 3-layer carbon to the top and bottom symmetrically for full Glass/Epoxy composite. As seen in the figure, adding carbon layers decrease the deformation and impact resistance of laminate is increased. The maximum difference is between M3 and Glass/Epoxy. The deformation of Glass/Epoxy is nearly 1.22 % times higher than the deformation of M3.



In figure 7 comparison of deformation for the full Carbon/Epoxy and hybrid models is given. These hybrid models are obtained adding 1, 2, and 3 layer glass to the top and bottom symmetrically for full Carbon/Epoxy composite. As seen in the figure, adding glass layers increase the deformation and the impact resistance of laminate is decreased. The maximum deformation increase is with M6. The value of this increase is nearly 20%.



Figure 6. Comparison of deformation graph of Glass/Epoxy and hybrid models



Figure 7. Comparison of deformation graph of Carbon/Epoxy and hybrid models

- **4.** Conclusions: From the present study, the conclusions can be summarized as:
  - The damage resistance of Carbon/Epoxy composite plates used in the study to a dropweight impact event better than the Glass/Epoxy composite plates used in this study.

E

The International Conference of Materials and Engineering Technology

- For the 1mm thickness, the maximum impact force of Carbon/Epoxy is 37.5 % higher than the maximum impact forces of the Glass/Epoxy.
- For the 2mm thickness, the maximum deformation of Glass/Epoxy is 18 % higher than the maximum deformation of the Carbon/Epoxy
- The effect of hybridization on the drop weight impact is investigated considering different hybrid models.
- These hybrid models are obtained adding 1, 2, and 3layer carbon to the top and bottom symmetrically for full Glass/Epoxy composite.
- Adding carbon layers increase impact loads and impact resistance of laminate is increased. The maximum difference is between M3 and Glass/Epoxy. The impact forces of M3 are nearly 1.33 times higher than the impact forces of Glass/Epoxy.
- Adding glass layers decrease the impact loads and impact resistance of laminate is decreased. The maximum reduction is with M6. The value of this reduction is nearly 30%.
- The hybrid configuration is obtained by adding one layer glass after one layer of carbon until the getting 2mm thickness as in Model 7 and one layer carbon after one layer glass until they getting 2mm thickness as in Model 8. they have shown similar behavior under the drop weight impact load.
- The maximum deformation of Glass/Epoxy for 2 mm plate thickness is 1.22 times higher than the maximum deformation of the Carbon/Epoxy plate.
- Adding carbon layers (to full Glass/Epoxy) decreases the deformation.
- Adding glass layers (to full Carbon/Epoxy) increases the deformation.

#### **References:**

- 1. Villavicencio, R., and C. Guedes Soares. Impact response of rectangular and square stiffened plates supported on two opposite edges. Thin-Walled Structures, **2013**, 68 : 164-182.
- 2. Santiago, Rafael, Wesley Cantwell, and Marcílio Alves. Impact on thermoplastic fibremetal laminates: Experimental observations. Composite Structures, **2017**, 159: 800-817.
- 3. Rawat, Prashant, K. K. Singh, and Nand Kishore Singh. "Numerical investigation of damage area due to different shape of impactors at low velocity impact of GFRP laminate." Materials Today: Proceedings, **2017**, 4(8): 8731-8738.
- 4. Yeter, E., and Doğru, M.H. A numerical investigation on damage resistance of materials to a drop weight impact event. **2018**, Gaziantep
- Yeter, E. Damage resistance investigation of Armox 500T and Aluminum 7075-T6 plates subjected to drop-weight and ballistic impact loads. Sakarya University Journal of Science. 2019, 23(6): 1095-1080.

# REMOVAL of DYE POLLUTION by MODIFIED HALLOYSITE CLAY as ECO-FRIENDLY ADSORBENT: KINETIC STUDIES

The International Conference

aterials and Engineering Technology

## DAVUT LACİN<sup>1</sup>, A.Z. AROGUZ<sup>2</sup>

<sup>1</sup>Istanbul University-Cerrahpasa, Faculty of Engineering, Department of Geological Engineering, Istanbul, Turkey <sup>2</sup>Istanbul University-Cerrahpasa, Faculty of Engineering, Department of Chemistry, 34850 Istanbul, Turkey

### Abstract

In this study, the adsorption capacity of modified halloysite was systematically investigated with respect to initial dye concentration and time in a batch process. The halloysite-mineral used in this work was an aqueous alumina silicate mineral including similar structural layers and it was gathered from the Biga Peninsula, Western Anatolia. The characteristic properties of the modified halloysite was analyzed by using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR). Cetyltrimethylammonium bromide (CTAB) aqueous solution was used for the modification process of halloysite. Methyl orange was used as model dye to find out the adsorptive capacity of modified halloysite. It was found that the adsorptive capacity of clay was increased by increasing the contact time and initial concentration. The pseudo-first order kinetic and pseudo-second-order kinetic models were applied to the data obtained at different concentrations of dye. The pseudo-second-order-model was found to be the best fit model for the adsorption kinetic. Langmuir and Freundlich isotherm models were used to find the suitable model for describing the adsorption equilibrium. Unmodified-clay was used to compare the adsorptive property of the modified-clay during the adsorption process. The modified halloysite performed better capacity as an adsorbent than untreated clay for the adsorption of methyl orange.

Keyword: Clay, Adsorption, Dye, Halloysite, Isotherm

#### **1. Introduction**

Many industries such as, textile, food, plastics, cosmetics, paper etc. produce serious contamination and they are often discharge in the form of colored waste to the environment. Dyes and pigments are carcinogenic, mutagenic and they are mostly toxic. Therefore they causes many environmental and detrimental problems [1]. Dyes in aqueous solution reduce the photosynthesis of the aquatic plants [2,3] Many conventional methods have been used for removal of dyes from wastewater. Adsorption is one of the technique which is preferred for the treatment wastewater because of simplicity of design, low cost investment and ease of operation [4]. Besides adsorption process there are mny other methods as precipitation, flocculation, coagulation, oxidation, aerobic and anaerobic degradation, filtration etc [5]. Adsorption is perfect techniques especially suitable and low cost natural adsorbents are used [6,7]. Clays are modified with different surfactants to improve their performance in adsorption process are defined as organoclays. Halloysite is silicate group clay mineral. It is formed within the melting cavities of the clayey limestones. These are created by the alteration of the magmatic depth, magmatic and metamorphic rocks. They include some minerals such as mica and feldspar.

Halloysite has large surface area It has hollow structure and shows excellent physical properties and chemical stability. Halloysite likes nanotubes therefore it is preferably used as adsorbents. It is a suitable mineral to be mixed polymers. Halloysite can be easily modified to further increase its adsorptive efficiency. The aim of the present study, was to assess the adsorption capacity of modified halloysite clay to remove methyl orange dye from waste water medium. After modification the adsorption capacity of the clay is increased by easy and simple operation The adsorption capacities of the modified and unmodified clay were also compared and it was observed that the modified halloysite performed high adsorption capacity than untreated halloysite during the adsorption process of methyl orange The characteristic properties of the prepared material was analyzed by using XRD and FTIR. The adsorption capacity of modified halloysite increases with respect to initial dye concentration and time in a batch process.

The International Conference of Materials and Engineering Technology

## Experimental

Halloysite clay was obtained from Biga, Çanakkale Province, in Turkey. Its cation-exchange capacity (CEC) is 5-10 meq/100g. The cetyltrimethylammonium bromide (CTAB, 99%) was supplied from Sigma Company. Methyl orange was purchased from Merck and used as received. *Preparation of Adsorbents:* The halloysite sample was first washed and cleaned from the dust and water soluble impuritiesThe wet clay was dried in an oven at 120 °C until unchange of its weight. They were first grounded and sieved through sieve (80 mesh), and stored in a desiccator for later use as an adsorbent (unmodified halloysite). To prepare the modified halloysite, the unmodified halloysite was dispersed in a cetyltrimethylammonium bromide (CTAB) aqueous solution with a dosage of 2 g/mmol halloysite/CTAB. The resultant solution was continuously mixed for at constant temperature (25°C) (almost 5h). Then the solution was left to settle down and then centrifugated to remove the solid particles. The product was then washed with distilled water.

#### Characterization of Materials

Fig1, Fig.2 show the XRD pattern, and FTIR imaging of the halloysite samples, respectively. *XRD measurements:* The halloysite sample was examined by X-ray powder diffraction (Philips diffractometer with a PW-1730 at 36 kV and 30 mA, using Ni-filtered CuK  $\alpha$ -radiation). Scanning speed was 1°/min. The clay samples were first saturated with ethylene-glycol vapour at 55 °C for 18 h and then X-rayed immediately (Fig.1).

The comparable FTIR recorded by the ThermoNicolet 380 FT model device. The graphs are shown in Fig 2.

#### Adsorption studies

Methyl orange solutions were prepared in distilled water with various concentrations (20, 40, 60 and 80mg/L. For adsorption studies adsorbent weight/solution volume are 0,5g/50mL. The solutions pH was adjusted to 6.5. by HCl or NaCl solutions. During the adsorption process, the concentration of methyl orange was measured by UV-vis spectrometer (T-80, PG Instruments) at  $\lambda_{max} = 464$  nm.

The adsorption capacity of halloysite was investigated using methyl orange dye and the through a batch adsorption experiment and the amount of adsorbed dye/unit mass  $(q_t)$  of the adsorbents was calculated by the equation given below

$$q_t = \frac{C_o - C}{W} \times V \tag{1}$$

Con

The International

terials and Engineering Technology

The percentage of uptake of dye was also estimated by Eq.2.

UWE Bristol

$$\% Uptake = \frac{C_o - C_t}{C_o} \times 100 \tag{2}$$

where  $C_o$  and  $C_t$  (in mg/L unit) are the concentration of dye at initial time and at time t, respectively. and  $C_e$  indicates the concentration of dye at equilibrium ( $C_e$ ).

#### **Results and discussion**

As seen from XRD results, the halloysite sample is composed mainly of halloysite and very small quartz minerals (Fig.1).



Figure 1: XRD result of Halloysite

Figure 2 shows comparative FTIR results of the adsorbents.



Figure 2: Comparative FTIR graphs of unmodified halloysite and modified halloysite clay

The maximum adsorption of dye ( for 80 mg/L solution) on both modified and unmodified clays was found 90% and 45 %, respectively. The percentages of adsorption are increase with increasing concentration (Fig.3)



**Figure 3:** The effect of initial concentration on the adsorption (%) of Methyl Orange on modified halloysite, at 25°C.



The pseudo second-order kinetic model for the MO adsorption in different initial concentrations on modified halloysite, at 25°C [8]. The kinetic studies indicate that the second order model is suitable for the adsorption of Methyl Orange on modified halloysite (Fig.4). The R<sup>2</sup> values are very high for second order kinetic model.



Figure 4: The pseudo second-order kinetic model for the MO adsorption in different initial concentrations on modified halloysite, at 25°C.

The Langmuir and Freundlich isotherm models were used to find the suitable model for describing the adsorption equilibrium [9]. The result indicates that the Freundlich model may be more suitable for predicting the adsorption of MO on modified halloysite. It also confirmed the heterogeneous nature of the surface of the modified halloysite.

Unmodified-clay was used to compare the adsorptive property of the modified-clay during the adsorption process. The modified halloysite performed better capacity as an adsorbent than untreated clay for the adsorption of methyl orange.



The uptake percentage of methyl orange adsorbed on unmodified halloysite increased from 38% to 46% as the concentration changes from 20 to 80 mg/L whereas the uptake value increased from 65% to 91% for the modified halloysite. Pseudo-second-order kinetics were more suitable for describing the adsorption of methyl orange on the modified halloysite. The adsorption of methyl orange on the modified halloysite. The adsorption of methyl orange on the diffusion into the pores. The results can be cofirmed by the intraparticle diffusion model. Adsorption isotherm of this process followed Freundlich model which shows multi-layer heterogenous adsorption. This process is economically useful for the adsorption of methyl orange dye and it was found that the modification process highly affects the adsorption capability of halloysite. Modified halloysite mineral used in this study can be well utilized for the adsorption of methyl orange from aqueous solutions.

The International Conference of Materials and Engineering Technology

### References

1- Zhai, L., Bai, Z., Zhu, Y., Wang, B., Luo, W. Chinese J. Chem Eng, **2018**, 26(3), 657-666.

2- TariqulIslam, Md., Saenz-Arana, R., Hernandez, C., Guinto, T., Ahsan, A. Md., Bragg, D.T., Wang, H., Alvarado-Tenorio, B., Noveron, J. C. J. Environ Chem Eng., **2018**, 6 (2), 3070-3082.

3- Reck, I.M., Paix, R.M., Bergamasco, R., Vieira, M.F., Vieira, A.M.S., J. Cleaner Production, **2018**, 171, 85-97.

4-Sciascia, L., Casella, S., Cavallaro, G., Lazzara, G., Milioto, S., Princivalle, F. and Parisi, F., *Ceramics International*, **2018**, 45 (2) 2751-2759.

5- Meenambal, T., Devi, D., J.Environ Sci Eng., 2006, 48(4), 247-252.

6- a) Sharma, A., Syed, Z., Brighu, U., Bhushan Gupta A. and Ram, C., *Journal of Cleaner Production*, 2019, 220, 23-32.

7- Chaari, I., Fakhfakh, E., Medhioub M., Jamoussi, F., *Journal of Molecular Structure*, **2019**, 1179, 672-677.

8- Ho, Y.S., McKay, G. Process Biochem, 1999, 34(5), 451-465.

9- Freundlich, H.M.F., 1906. Uber die adsorption in losungen. Z Phys Chem 57A, 385–470.

# VÜCUT SICAKLIĞI UYARISI VEREBİLEN AKILLI SPOR TEKSTİL ÜRÜNÜ TASARIMI

The International Conference of Materials and Engineering Technology

## Halil İbrahim ÇELİK\*, Dilek ALICI, Esile GÜLSEVGİ, Zeynep ŞENGÜL, Elif GÜLTEKİN

Gaziantep Üniversitesi, Tekstil Mühendisliği Bölümü, Gaziantep, TÜRKİYE.

## ÖZET

Teknoloji ve malzeme bilimindeki gelişmeler ile birlikte tekstil ürünlerinde yeni uygulamalar gündeme gelmiştir. Artık tekstil ürünlerinin sadece örtünme amacı ile kullanımlarının dışında daha fonksiyonel ve akıllı hale gelmeleri sağlanmaktadır. Akıllı tekstiller çevresindeki koşulları ve değişiklikleri algılayan ve buna yönelik tepki oluşturan ürünler olarak tanımlanmaktadır. Günümüzde özellikle spor giysilerinde akıllı tekstil ürünlerine olan yönelim artmıştır. Bu çalışmada, sporcunun vücut sıcaklığını ölçen ve uygun sıcaklık değerine ulaşıldığında yüksek tempolu hareketlere geçilebilmesi için uyarı veren prototip bir akıllı tekstil ürünü sunulmuştur. Böylece, spora başlamadan önce vücut ısısının dengesizliğinden kaynaklanan olumsuz sonuçların ortadan kaldırması ve sağlıklı bir şekilde spora başlanması amaçlamıştır. Çalışmada, sıcaklık sensörü, RGB led ve Lilypad Ardunio parçalarının iletken iplik kullanılarak oluşturulan devre ile örme bir tişört üzerine adapte edilmesi sağlanmıştır. Daha sonra, belirlenen sıcaklık değerinde uyarı ışığını aktif hale getirilebilesi amacı ile yazılım hazırlanmıştır. Sonuç olarak tasarlanan akıllı ürünün vücut sıcaklığındaki artışa göre farklı renklerde uyarı verildiği görülmüştür.

Anahtar Kelimeler: Akıllı tekstiller, spor giysisi, Lilypad arduino, vücut sıcaklığı

## 1. GİRİŞ

Gelişen teknoloji ile tekstil alanında yapılan çalışmaların önemli kısımlarından biri akıllı tekstillerdir. Akıllı tekstiller, tekstil ürünlerinin doğal atmosfer şartlarından koruma ve algılama gibi özellikleri ihtiva eden ve ilave olarak herhangi bir etkiye karşı (1şık, 1sı, basınç, elektromanyetik dalgalar, ses ve ses ötesi dalgalar, hareket vs.) bir tepki verme özelliğine sahip olan tekstil ürünleridir. Geniş kitlelere hitap etmesi ve ekonomik açıdan büyük paya sahip olması nedeniyle akıllı tekstil ürünlerinin ilgi alanlarından biri spordur. Sporcuların, çeşitli spor dallarındaki ihtiyaçları farklı koşullara göre değişmektedir. Normal koşullarda insanlar 37°C  $\pm$  0,5°C aralığında sabit bir sıcaklığa sahiptir. Vücut sıcaklığının 40,5°C 'nin üzerine çıkması veya 35°C 'nin altına düşmesi kalıcı hasarlara neden olmaktadır[1].

Günümüzde, küreselleşme iklim koşullarında değişikliğe sebep olsa da, sporcuların her durumda maksimum performansı göstermesi beklenir. Bu nedenle, son yıllarda spor dallarındaki ihtiyaçlara yönelik tasarlanan spor tekstili ürünlerine talep artmaktadır. Spor alanında akıllı tekstil ürünlerinden olan giyilebilir sensörler tercih edilmektedir. Giyilebilir sensörlerin temel çalışma prensibi, egzersiz sırasında kinematik analiz yaparak vücudun fizyolojik yanıtlarını izlemek olarak tanımlanmaktadır [2,3]. ). Çeşitli sensörlerin (sıcaklık sensörü, nabız sensörü, basınç sensörü vb.) ve elektronik devrelerin giysiye adapte edilebilmesi ile giyilebilir sensörler olarak adlandırılan akıllı tekstillerin en yaygın kullanım alanı vücut sıcaklığını ölçebilen ve buna bağlı olarak dışarıya uyarı verebilen özelliğe sahip tasarımlar olmasıdır[4,5]. Egzersiz sırasında vücutta meydana gelen

sıcaklık değişimlerini kayıt altına alan giyilebilir sensörler egzersiz performansının nasıl değiştiği hakkında sporcuya bilgi vermektedir. Bu alanda yapılan araştırmalar insan vücudu ile temas halinde olan kumaşların ısı iletkenliği ile sıcaklık değişimlerinin ölçülmesi prensibine dayanır [6]. Sporcu giysilerinde sıcaklık ölçümünün yanı sıra, nefes alma hızı ve sıklığı da spor esnasında önemli bir rol oynadığı için bu iki parametreyi ölçebilen akıllı tekstillerin üretimi mevcuttur. Farklı spor dalları için farklı nefes ritimleri söz konusudur. Yapılan çalışmalarda iletken polimer yapıların kullanıldığı giysi tasarımı ile göğüs kafesinin genişleme hacmi ve sıklığı ölçülebilmektedir [7]. Giyilebilir sensörlerin bir diğer alternatifi ise, basınç sensörü ile tasarlanan sporcu giysileridir. Basınç sensörü ile sporcunun temel hareketleri ve spor branşına bağlı olarak vücuda uygulan darbenin şiddeti basınç verileri ile kayıt altına alınabilmektedir.

The International Conference

sterials and Engineering Technology

Solunum hızı, kalp ritmi, nabız ve sıcaklık gibi birçok vücut parametrelerini ölçebilen akıllı giysi örnekleri günümüzde farklı firmalar tarafından ticari ürün olarak piyasaya sunulmaktadır (Şekil 1). Çeşitli spor dallarında kullanımı yaygınlaşan söz konusu bu ürünler ile sporcu sağlığının korunması amaçlanmıştır.



Şekil-1. Akıllı spor giysiler [8,9]

Spor aktivitesi sırasında vücut fonksiyonlarının anlık olarak ölçülmesi ve değerlendirilmesi akıllı tekstil ürünleri ile mümkün hale gelmiştir. Spor aktivitelerinde dikkat edilmesi gereken en önemli unsur yüksek tempolu hareketlere veya çalışmalara geçilmesinden önce vücudun ısınması ve vücudun spora hazır hale gelmesi gerekliliğidir. Aksi takdirde vücut ısınmadan spor yapmak olumsuz sonuçlara neden olabilmektedir. Bu çalışmada, sporcunun vücut sıcaklığını ölçen ve uygun sıcaklık değerine ulaşıldığında yüksek tempolu hareketlere geçilebilmesi için uyarı veren prototip bir akıllı tekstil ürünü sunulmuştur. Böylece, spora başlamadan önce vücut ısısının dengesizliğinden kaynaklanan olumsuz sonuçların ortadan kaldırılması ve sağlıklı bir şekilde spora başlanması amaçlamıştır.

## 2. MATERYAL VE METOT

#### 2.1. Material

Sunulan çalışma kapsamında arduino devre elemanları ve standart bir tişört kullanılarak akıllı bir sporcu giysisi tasarlanmıştır. Tasarlanan tişört lilypad arduino, sıcaklık sensörü, batarya yuvası, RGB led ve iletken iplikten oluşmaktadır (Şekil 2). Elektronik devre elemanları tişört üzerine uygun bir şeklide yerleştirilerek iletken iplik ile devre oluşturulmuştur. Tasarlanan giysi için ayrıca bir yazılım geliştirilmiştir. Bu yazılım ile belirlenen eşik sıcaklı değerlerinde RGB led üzerinde farklı ışık renklerinin yansıtılması sağlanmıştır.



LilyPad ürünler birden fazla bağlantı noktasına ve programlanabilme özelliğine sahip, giysi üzerine kolayca dikilebilen ve yıkanabilen arduino devre elemanıdır. Sıcaklık sensörü vücut sıcaklığını Celsius ve Frahrenheit cinsinden ölçüm imkanı sağlamaktadır. Kullanılan batarya yuvası (3 volt değerinde), el ile açıp/kapama imkanı olan veya otomatik açılmayı engellemeye yarayan bir anahtar içermektedir. Tercih edilen RGB led 4 farklı renk içermekte olup katot led türündedir. Ayrıca devre elemanlarını birbirine bağlamak için kullanılan iletken iplik bakır elyafından oluşmaktadır.



Şekil 2. Lilypad malzemeleri

## 2.2. Metot 2.2.1. Akıllı Tişört Tasarımı ve Prototip Üretimi

Öncelikle tasarlanan ürünü kullanım amacına uygun olarak devre elemanlarının tişört üzerine yerleştirilmesi planlanmıştır. Bu aşamada vücut sıcaklığının etkin bir şekilde ölçülmesi ve uyarı ışığının kullanıcı tarafından rahatlıkla görülmesi kriterleri göz önünde bulundurulmuştur. Diğer taraftan, akıllı giysi devre elemanları arasında iletim sağlanması için bu parçaların iletken iplik kullanılarak bağlanması gerekmektedir. Sıcaklık ölçümünde en sağlıklı sonucu alabilmek adına sıcaklık sensörü giyilebilir tişörtün koltuk altı kısmına yerleştirilmiştir. Böylece daha hızlı sonuç alınması sağlanmıştır. Ayrıca spor yapan kişinin vücudundaki sıcaklık değişimini rahat görebilmesi için RGB led, tişörtün karın bölgesine dikilmiştir.

Sporcunun tasarlanan ürünü rahat kullanabilmesini göz önünde bulundurarak batarya ve arduino giysinin sağ alt köşesine dikilmiştir. Tasarım sürecindeki en önemli parametrelerden biri, kullanılan malzemelerin bağlantılarının doğru bir şekilde belirlenip iletken iplik ile sporcu giysisi üzerine yerleştirilmesi olmuştur. Devre elemanları planlanan taslağa uygun olarak t-şort üzerine dikilmiştir. Tüm elemanların arasındaki bağlantı iletken iplik ile oluşturulan devre aracılığı ile sağlanmaktadır (Şekil 3).



Şekil-3. Akıllı giysi devre tasarımı

## 2.2.2. Yazılım Geliştirme

Tasarlanan akıllı sporcu giysi sisteminin çalışabilmesi için Ardunio programında yazılımının oluşturulması gerekmektedir. Tasarımda kullanınan RGB led 4 farklı renk (kırmızı, mavi, yeşil ve beyaz) seçeneklerini içermektedir. Hazırlanan yazılım ile normal vücut sıcaklık değeri (36,5 °C) temel alınarak her bir renk için farklı eşik sıcaklık değerleri belirlenmiştir. Sporcunun vücut sıcaklığı göre verilecek RGB led renkleri Tablo 1'deki gibi tanımlanmıştır.

<b>Table 1</b> . V deut Steakingina gole KOD ied Tenkien							
Led Uyarısı	Beyaz Işık	Mavi Işık	Yeşil Işık	Kırmızı Işık			
Vücut	35.5°C	36,5-37°C	37,2-39 °С	40°C			
Sicabhk							

Tablo 1. Vücut sıcaklığına göre RGB led renkleri

Tablo 1'de verilen eşik değerlerine göre hazırlanan algoritmaya (Şekil 4)'de verilmiştir. Bahsedilen hazırlanan yazılıma göre;

- Beyaz ışık yandığı zaman sporcunun mevcut durumdaki vücut ısısının normal değerden düşük olduğunun sinyali verilmektedir.
- Mavi ışık yandığında vücut ısısın normal değerde olduğu göstermektedir.
- Yeşil renk sinyali ile sporcunun vücudun ısınmış ve spora hazır olduğu gösterilmektedir.
- Son olarak kırmızı renk sinyalinin alınması sporcunun yaşamsal fonksiyonlarında bir problem olduğunu göstermektir.



Şekil-4. Geliştirilen yazılım algoritması

## 3. Tartışma Ve Bulgular

Bu çalışma kapsamında sunulan akıllı sporcu giysi tasarımının en önemli başarı kriteri sıcaklık ölçümünün hassas bir şekilde yapılmasıdır. Değerlendirme aralığında kullanılan sıcaklık değerleri literatürden elde edilen bilgiler doğrultusunda yazılımın hazırlanması aşamasında kullanılmıştır. Tasarımı ve üretimi tamamlana ürün bir kullanıcı tarafından giyilerek denenmiştir. Aktif spor koşulundaki kullanımı esnasında RGB led üzerindeki ışık değişimleri takip edilmiştir. Her bir ışık değişiminde vücut sıcaklığı termometre ile ölçülerek ürünün doğruluğu test edilmiştir. Elde edilen bulgulara göre prototip üretimi yapılan ürünün kabul edilebilir düzeyde doğru ölçüm yaptığı sonucuna ulaşılmıştır.

## 4. SONUÇ

21. yüzyılda hızlı bir şekilde artan teknolojik gelişmeler tekstil sektörünü de önemli ölçüde etkilemektedir. Farklı disiplinlerde elde edilen gelişmeler tekstil ürünlerine adapte edilerek akıllı giysiler üretmek mümkün hale gelmiştir. Bu tarz uygulamaların en iyi örneklerinden biride giyilebilir elektronik tekstil ürünleridir.

Çevresel koşullara duyarlı ve kullanıcının vücut özelliklerini ölçme yeteneğine sahip olan giyilebilir sensörler birçok farklı spor dalında kullanılmaktadır. Bu kapsamda geliştirilen farklı ürünler katma değerli ürünler olup yüksek ticarileşme potansiyeline sahiptir. Bu çalışmada, giyilebilir sensörlerle vücut ısısını ölçebilen ve buna bağlı olarak kullanıcıya uyarı sinyali veren akıllı bir tişört tasarlanmıştır. Kullanıcı üzerinde yapılan değerlendirme sonucunda hedeflenen renk uyarı sinyalleri doğru bir şekilde elde edilmiştir. İlerleyen çalışmalarda tasarlanan bu ürünün vücut ısınını ölçmekle kalmayıp aynı zamanda kalp atış hızı ve nabız ölçümü gerçekleştirebilen çok fonksiyonlu akıllı bir giysiye dönüştürülmesi hedeflenmektedir. Bu konudaki tüm parametreler tek tek incelenip gerekli ekipmanlar temin edilerek alternatif bir akıllı sistem ile desteklenmesi sağlanacaktır.
### 4. Referanslar

1. Akçalı, K, Farklı Spor Branşlarında Kullanılan Akıllı Tekstil Ürünlerinin İncelenmesi. International Journal of Science Culture and Sport, (2016), 4(Special Issue 3), 689-703.

The International Conference of Materials and Engineering Technology

- 2. Ermes M, Pärkkä J, Detection of daily activities and sports with wearable sensors in controlled and uncontrolled conditions. IEEE Transaction on Information Technology in Biomedicine, (2008), 12(1), 20-26.
- 3. Coyle S, Morris D, King-Tong L, Diamond D, Moyna N, Textile-based wearable sensors for assisting sports performance. Wearable and Implantable Body Sensor Networks, Sixth International Workshop, (2009), 307-311.
- 4. Kahraman, G, Şahin, Ö., Kayacan, O., and Yazgan, E. Akıllı Giysilerde Isıtma Kontrolü. Dokuz Eylül Üniversitesi Mühendislik Fakültesi Fen ve Mühendislik Dergisi, (2008), 10(3), 33-42.
- 5. Kayacan, O, and Bulgun, E. Y. Smart Textiles and Electrically Conductive Textile Based Structures, (2005), (Volume: 12), 58.
- 6. Coşkun, E., and Oğulata, T, Akıllı tekstiller ve genel özellikleri. Çukurova Üniversitesi Fen Bilimleri Enstitüsü, (2007),18(3).
- 7. Carpi F, De Rossi D, Electroactive polymer-based devices for e-textiles in biomedicine. IEEE Transaction on Information Technology in Biomedicine, (2005), 9, 295-318.
- 8. Global Smart Sports Clothing Market, [Online]. Available: https://onyourdesks.com/2019/09/04/global-smart-sports-clothing-market-2019-competitive-analysis-players-adidas-athos-works-atlas-wearables-basis-beddit-beurer/.
- 9. Global Smart Sports Clothing Market, [Online]. Available: https://www.ispo.com/en/trends/wearable-technology-swiss-technology-and-swedishperformance-unite-major-smart-textile

# A REVIEW STUDY ON SMART TEXTILE INNOVATIONS AND DEVELOPMENTS

The International Conference of N

aterials and Engineering Technology

# HALIL İBRAHIM ÇELİK<sup>1</sup>, ABDULKADİR AĞA<sup>2</sup>, ELİF GÜLTEKİN<sup>3</sup>, HATİCE KÜBRA KAYNAK<sup>4</sup>

<sup>1</sup>Gaziantep University, Textile Engineering Department, Gaziantep, TURKEY.

#### Abstract

Smart textiles are intelligent textile structures or fabrics that have the capability to sense and react to environmental stimuli, which may be mechanical, thermal, chemical, biological, magnetic, and energy production amongst others. Today's dynamic market segments such as digital, health, transportation, energy, or security have an important added value that may be captured by textiles as supports. Particularly in the field of energy conservation and saving, how to make an adaptable textile surface and smart garments enough to react to heat or moisture under hot conditions are becoming very popular. Various methods are used in this specifically studied field. Improvements are made by adding some chemicals in polymer structure or some coating is done with finishing processes. In this study, previous studies in literature on smart textiles for energy harvesting, saving and storing were investigated and the methods applied in these studies were examined in detail.

Keyword: smart textiles, intelligent textiles, energy harvesting, energy saving

#### 1. Introduction

Energy harvesting and energy saving are the main concern of the world, the earth cannot sustain the demand of energy that is already increasing, the sources that we used to take from are not sustainable. But what if we shift our attention from the outside to the inside, to the human body. The human body is in continuous movement, every individual is an excellent opportunity for harvesting energy. Textiles cover more than 90% of human body surface area most of his life. The critical question is that "How can we use this fact to harvest the human energy?".

Personal thermal management, piezoelectric and triboelectric harvesting methods, Nano technology integration, conductive carbon nanotubes etc. The breaking innovations that are being investigated in the recent years by many textile and material scientists. In this study, we will review the recent developments on this particular manner, energy saving or harvesting using wearable textile surfaces.

#### 2. Literature Research

Energy consumption is increasing with the rapid growth of electronic textile increases the demand for textile-based power sources, which should have comparable lightweight, flexibility, and comfort. Recently, wearable heaters have attracted interest for their efficiency in providing articular thermotherapy. Thermal management through personal feeling like heating and cooling is a process by which to keep ambient temperature under control to save energy.



The International Conference

aterials and Engineering Technology

In 2016 Hsu et al. developed a cooling textile fabric that is IR-Radiation emissive, allowing the human body heat to pass the cloth -which usually holds it up- using a nanoporous polyethylene. They investigated that the nanoporous polyethylene (nanoPE) is transparent to mid-infrared radiation coming off human body. Due to the pore size distribution, nanoPE is not transparent to visible light. For this scope, it was studied to enhance a wearable textile that has good properties like sufficient air permeability, water-wicking rate, and mechanical strength. A device was developed to simulate skin temperature and investigate the favorable material that is IR transparent as a human cloth and can be used as for personal thermal management. NanoPE is a good IRtransparent textile for human body cooling. NanoPE has interconnected pores that are 50 to 1000 nm in diameter (Figure 1). The pore sizes were in the size range comparable with the wavelength of visible light (400 to 700 nm), which scatter visible light strongly and make PE opaque to human eyes. The pore sizes were also much smaller than the IR wavelength, so the nanoPE film was still highly transparent to IR. The results experimentally demonstrated the cooling effect of nanoPE with a device that simulated the heat output of skin (Figure 2A). NanoPE increases the simulated skin temperature by 0.8°C, compared with 3.5°C for cotton and 2.9°C for the fibrous PE textile Tyvek that DuPont manufactures (Figure 2B). Because the difference between skin and ambient temperature (23.5°C) was small, this skin temperature difference can be approximated as the air conditioner set point difference. Also an H-shaped piece of metal showed the textile IR transmittance by use of thermal imaging (Figure 2C) [1].



Figure 1. Schematics of comparison between nanoPE, normal PE, and cotton. Only nanoPE satisfies IR transparency, visible light opacity, and air convection simultaneously [1].



Figure 2. Thermal measurement of nanoPE and various textile samples [2].

Later in 2017 Hsu et al. have also developed a dual mode textile that can be used for cooling and heating human body by flipping the textile surface. They demonstrated a dual-mode textile that can perform both passive radiative heating and cooling using the same piece of textile without any energy input. This dual-mode textile was composed of a bilayer emitter embedded inside an infrared-transparent nanoporous polyethylene (nanoPE) layer (Figure 3). They also clarified that the emissivity and the asymmetric properties of the nanoPE thickness may result in two different heat transfer coefficients, and the low emission layer may cause heating while facing out, and the high release layer may cause cooling by wearing of the textile while facing out. In the design, the bilayer thermal emitter, which has different emissivities on each side, was embedded inside the nanoPE textile, which also has asymmetric thicknesses on each side. Since the nanoPE is IRtransparent, the emitter inside it can freely radiate toward the ambience. The Emissivity and temperature could control by the bilayer emitter and the nanoPE thickness respectively from being closer or farther from the hot side (human skin). For the dual mode textile, when the ambient temperature is low, if the artificial skin temperature falls below almost 32 ° C, the dual mode textile enters heating mode and causes the artificial skin temperature to rise. When the ambient temperature becomes too high, the textile returns to cooling mode. As a result, dual-mode textile may maintain artificial skin temperature from 32 ° C to 36 ° C, while dynamic ambient temperature fluctuation of 9 ° C may be encountered [2].



**Figure 3.** Schematic of dual-mode textile. (A) Traditional textiles only have single emissivity, so the radiation heat transfer coefficient is fixed. (B) For a bilayer thermal emitter embedded in the IR-transparent nanoPE, when the high-emissivity layer faces outside and the nanoPE between the skin and the emitter is thin, the high emissivity and high emitter temperature results in large heat transfer coefficient, so the textile is in coolingmode. (C) The textile is flipped, and the low

emissivity and low emitter temperature cause the heat transfer coefficient to decrease. The textile now works in heating mode [2].

The International Conference

of Materials and Engineering Technology

A work of 2019 by Hazarika et al. have taken it a bit further and developed a self-powered heating textile, but it was mainly composed of woven Kevlar fibers, which are mainly used for armor clothes. The design of a personal thermal management device with a self-powering ability to generate heat through triboelectricity was reported. Composites were prepare with vertically aligned silver tipped nickel cobalt selenide (Ag@NixCo1-xSe) nanowire arrays synthesized on the surface of woven Kevlar fiber (WKF) sheets and reduced graphene oxide (rGO) dispersed in Polydimethylsiloxane (PDMS) (Figure 4.). Reduced graphene oxide (rGO) can provide a homogenous surface temperature when introduced into a polymer matrix for electric heating purposes, because it has a sufficiently high thermal conductivity of 3000-5000 W m<sup>-1</sup> K<sup>-1</sup>. It can also behave as a negative triboelectric material because the edges and basal planes of graphene nanosheets contain oxygen functional groups. The concept of a personal thermal management (PTM) device is significant interest because it can regulate body temperature to a thermally comfortable level by harvesting energy from the body, and can even raise the body temperature if necessary, imparting a localized heating effect. PTM of body armor would provide self-powered heating ability, flexibility, and reduced weight. These features would provide more comfort to wearers of traditional heavy and rigid body armor. It was stated that, Smart bulletproof armor offering thermal insulation and electrical heating that could be driven by a small battery, or even self-powered for electrical heating in the absence of a battery by harvesting energy from body motion, would be of particular interest in extreme climate conditions[3].



Figure 4. Schematic diagram of fabrication of the WKF/Ag@NixCo1-xSe/PDMS/rGO composite [3].

A self- powered and self-functional cotton sock was developed by Zhu et al. in 2019 using Piezoelectric and triboelectric hybrid mechanism to harvest energy from walking and jumping of human being in addition to sensing of various physiological signals, which can give important information on the human health condition. In this paper, they have developed a self-powered and self-functional sock (S2-sock) to realize diversified functions including energy harvesting and sensing various physiological signals, i.e., gait, contact force, sweat level, etc., by hybrid integrating poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS)-coated fabric triboelectric nanogenerator (TENG) and lead zirconate titanate (PZT) piezoelectric chips. The study showed that cotton socks worn daily can potentially be a power source for enabling self-sustained socks comprising wireless transmission modules and integrated circuits in the future (Figure 5). They also investigated the influences of environmental conditions such as; humidity,

413



The International Conference of

Materials and Engineering Technology



Figure 5. Schematic of PEDOT; PSS coated triboelectric S2-sock [4]

In another work of integrating health monitoring and smart textiles, Lin et al. in 2017 developed a sleep monitoring self-powered mattress that uses the pressure a sleeping individual acts on the mattress to generate electrical power and evaluate the quality of sleep. A bed sheet was reported which have a pressure-sensitive, large-scale, and washable smart textile for real-time and selfpowered sleep behavior monitoring based on triboelectric nanogenerator (TENG) array. This smart textile was fabricated by conductive fibers and elastomeric materials with a wave structure, the TENG units exhibit desirable features including high sensitivity, fast response time, durability, and water resistance, and are interconnected together, forming a pressure sensor array. On the other hand, highly integrated data acquisition, processing, and wireless transmission system was established and equipped with the sensor to realize real-time sleep behavior monitoring and sleep quality evaluation. The study also emphasized that this smart textile can also serve as self-powered warning system for emergency conditions, and to recognize and alert the medical staff in the case of clinically derived alarm (Figure 6). The smart textile product used in this study mainly consists of three layers: The top and bottom layers was made of the fully perpendicular column and row conductive fiber arrays as triboelectric layers. And a wavy-shaped PET film was sandwiched by the two layers of conductive fibers. When the external pressure was applied onto the smart textile, the structure change of the middle PET layer will induce a change of its contact area with the two conductive layers, resulting in an electrical potential difference between them, as shown in Figure 7.[5].



**Figure 6.** Demonstration of the smart textile with washableability as bedsheet for sleeping behavior monitoring and self-powered warning system in case of an aged sleeper falling down from bed. And an alarm lamp is activated as an emergency to inform others [5].



**Figure 7.** Electrical signal generation process of the smart textile under applied force. a) 3D illustration of the working mechanism of the smart textile. And the acquired electrical signal profiles b) open-circuit voltage and c) short-circuit current [5].

In 2019 Liu et al. focused on the high-energy asymmetric supercapacitor yarns for self-charging power textiles. Their novel work integrated a textile-based energy harvesting triboelectric nanogenerators (TENG) method, for the wide electrification fibers already used to textile production and for the tremendous surface area of microfibers. These materials that can be used for harvesting energy, with an energy-storing yarn-type asymmetric super capacitors method (Y-ASC) (Figure 8). The yarn supercapacitor consists of negative yarn electrode with hydrothermally self-assembled reduced graphene/carbon nanotubes coating while the positive electrode had Ni-Co bimetallic oxyhydroxide coating. The conductive yarns were coated with polydimethylsiloxane (PDMS) and woven into common fabrics. Surface fluoridation treatment was carried out to improve the polarity of PDMS. Highly conductive 1D yarns were obtained by electroless deposition (ELD) of Ni and subsequent electrodeposition of Cu coatings on common polyester yarns, which can be utilized as current collectors in all-solid-state Y-ASCs. Uniaxial tensile tests



The International Conference



Figure 8. The fabrication of fiber-shaped electrodes and asymmetric super capacitor (ASC) [6].

The mechanical energy of human motion was successfully converted into electricity by the Y-TENG to charge the Y-ASC. To demonstrate the application of the device in wearable electronics, two serially connected Y-ASCs were incorporated into common textiles, as shown in Figure 9. After being charged, this soft energy-storing fabric can power a red LED and a watch under bending state.



**Figure 9.** Optical image of two serial connected ASC devices woven into a fabric and a watch powered by this energy storage fabric [6].

In a recent study of 2019 Zhang et al. developed An IR-adaptive textile to directly regulate thermal radiation for personal thermal management using distance-dependent electromagnetic coupling between neighboring coated fibers in the textile yarns for the human body (Figure 10). In this way, the coupled temperature–sweating response of humans to thermal discomfort was directly used to allow adaptive gating of radiation through the textile [7].

Technology



**Figure 10**. The IR emissivity strongly depends on the electromagnetic coupling distance between the nanostructures [7].

Each textile yarn was composed of a bundle of metafibers that function via;

- (i) A meta-element (typically a conductive material) added in controlled quantities to the polymer textile fibers and
- (ii) An actuation mechanism that responds directly to changes in temperature and/or the relative humidity of skin.

When hot and wet, the yarn collapses into a tight bundle, bringing the neighboring metafibers into resonant electromagnetic coupling that shifts the IR emissivity to spectrally overlap with that of the skin. This effectively "opens" the cloth to promote radiative cooling of the human body (Figure 11.).



**Figure 11.** Bimorph fibers composed of hydrophobic triacetate and hydrophilic cellulose as the base polymer materials are used [7].

Owing to the competing hydrophobic and hydrophilic effects, the bimorph fibers can actuate to change interfiber spacing as a function of relative humidity, manifested as perspiration on human skin in response to the environment. Because the cellulose component of the fiber can absorb water molecules whereas the triacetate side cannot, this hydrophilic-hydrophobic side by-side structure leads to differential expansion in different relative humidities, causing subsequent mechanical actuation of the fibers within the knitted fabric to enable dynamically tunable IR gating in clothing. The meta coupling mechanism was not sensitive to the randomness and non-uniformity that typically exist in a textile. In-plane spatial randomness reduced emissivity by only ~10%, even for

large disorder of up to 30%. The coupling effect was also insensitive to axial randomness, causing only 2% variation in emissivity for 30% axial randomness .Also, these structural changes occur locally, preventing global distortion of the fabric, which would be undesirable for apparel [7].

The International Conference of Materials and Engineering Technology

# 3. Conclusion

After scientific efforts and development phases, nowadays smart textiles are an implanted customer interest and they are presented as the future of the textile industry. With the developing technology, the logging of smart textiles is increasing. A review of the past article on ordering from the literature, one of the most popular topics in recent times has been researched on body heat conservation and energy harvesting. Different applications and methods have been tried. In the previous studies, we have seen a method to allow a cloth fabric to transmit IR-radiation outside human body allowing it to cool itself better in comparison with conventional cloth fabric. Another method was related to both cooling and heating through one side of fabric that is IR emissive and another side non-emissive to IR radiation, and all you have to do is flip the cloth inside out to accommodate with the changing ambient temperature. A method was to generate electrical power from human body movement to use it in producing heat in the fabric, instead of depending on external power-consuming methods to heat the whole surrounding of an individual. However, it was mainly applicable to armor clothes such as Kevlar based suits. In relating smart textiles with smart health care smart evaluation methods, they have discussed two methods, one was a sock that generates electrical power using the strokes of the feet with the ground during walking or jumping, the generated power was used in sensing and evaluating certain walking patterns, which can tell if the person has health issues or problems. In a different study, the author also generating electrical power but from sleeping movements a person apply to a mattress, and by the pressure differences and the number of movements a person make during -say an 8 hours sleep- the sleep quality can be determined and health issues can be sensed using medical information and certain algorithms to alarm or tell if something is wrong, in addition, this mattress can sense if a patient falls down the bed while sleeping allowing the nurses to be alarmed about it. A novel and astonishing methods were about storing and harvesting energy with the same piece of textile fabric, with no use of bulky power storage devices this work promises big future capabilities. A lot of scientists are developing new solutions, ideas and concrete products with the emerging demand of smart textiles in various phases of life. In this study, previous studies in literature on smart textiles for energy harvesting and storing were investigated and the methods applied in these studies were examined in detail.

# 4. References

- 1. Hsu, P, C., Song, A. Y., Catrysse, P. B., Liu, C., Peng, Y., Xie, J., and Cui, Y. Radiative Human Body Cooling By Nanoporous Polyethylene Textile. Science, (2016), 353(6303), 1019-1023.
- Hsu, P. C., Liu, C., Song, A. Y., Zhang, Z., Peng, Y., Xie, J., and Zhai, S. A Dual-Mode Textile For Human Body Radiative Heating And Cooling. Science Advances, (2017), 3(11), e1700895.
- **3.** Hazarika, A., Deka, B. K., Jeong, C., Park, Y. B., and Park, H. W. Biomechanical Energy-Harvesting Wearable Textile-Based Personal Thermal Management Device Containing



Epitaxially Grown Aligned Ag-Tipped-NixCo1- xSe Nanowires/Reduced Graphene Oxide. Advanced Functional Materials, (2019), 1903144.

- **4.** Zhu, M., Shi, Q., He, T., Yi, Z., Ma, Y., Yang, B., and Lee, C. Self-powered and self-functional cotton sock using piezoelectric and triboelectric hybrid mechanism for healthcare and sports monitoring. ACSNANO, (2019), 13(2), 1940-1952.
- Lin, Z., Yang, J., Li, X., Wu, Y., Wei, W., Liu, J., and Yang, J. Large-Scale and Washable Smart Textiles Based on Triboelectric Nanogenerator Arrays for Self-Powered Sleeping Monitoring. Advanced Functional Materials, (2018), 28(1), 1704112.
- **6.** Liu, M., Cong, Z., Pu, X., Guo, W., Liu, T., Li, M., and Wang, Z. L. High-Energy Asymmetric Supercapacitor Yarns for Self-Charging Power Textiles. Advanced Functional Materials, (2019), 1806298.
- 7. Zhang, X. A., Yu, S., Xu, B., Li, M., Peng, Z., Wang, Y., and Wang, Y. Dynamic gating of infrared radiation in a textile. Science, (2019), 363(6427), 619-623.

# LOW VELOCITY IMPACT BEHAVIORS OF THERMOPLASTICS COMPOSITES WITH DELAMINATONS

The International Conference

aterials and Engineering Technology

# OĞUZHAN DURDU<sup>\*1</sup>, OKAN ÖZDEMİR<sup>2</sup>

<sup>1</sup> Dokuz Eylül Univ., The Graduate School of Natural and Applied Sciences, Dept. of Mechanical Engineering, Tinaztepe Campus, Izmir, TURKEY
<sup>2</sup> Dokuz Eylül Univ., Engineering Faculty, Dept. of Mechanical Engineering, Tinaztepe Campus, Izmir, TURKEY

#### Abstract

In various applications, thermoplastic materials are reinforced with glass fiber reinforcement and are widely used in areas requiring high mechanical strength. For this purpose, this study is aimed to reveal the effect of deficiencies that may occur during thermoplastic production affected the low velocity impact behavior of material. For the manufacturing of composite materials, long glass fiber reinforced homopolypropylene composite granules produced by pultrusion method and thermoplastic unidirectional laminates were used. Indefectible specimens and specimens with embedded delamination at two different diameters (i.e., 13 mm and 26 mm) were manufactured. Firstly, the materials were placed to a mold having thicknesses of 3 mm as the stacking sequence of [0/90/granule/90/0]. Embedded delamination was placed between upper unidirectional laminates. Subsequently, it was produced by hot pressing method under 100 kN and at a temperature of 180 °C. The produced specimens were cut to the required dimensions for the experiments. Finally, the low velocity impact test was applied to the samples at different energy levels (10J, 20J, 30J, 40J, and 50J). According to the data obtained from the experiments, the maximum contact force and maximum deflection values are not significantly changed depending on the delamination size for the same energy level. Besides, there is a significant reduction in the maximum contact force value of specimens with a delamination diameter of 13 mm at all energy levels after the perforation thresholds.

Keyword: Thermoplastic composite, Low Velocity Impact, Glass fiber, Delamination

#### **1. Introduction**

Thermoplastic materials are, in general, ductile and tougher than thermoset materials and are used for a wide variety of nonstructural applications without fillers and reinforcements. Thermoplastics can be melted by heating and solidified by cooling, which render them capable of repeated reshaping and reforming. Thermoplastic molecules do not cross-link and therefore they are flexible and reformable [1]. Polymer matrix composites are susceptible to internal damage caused by low velocity impact (LVI). In many instances, damage is not apparent on the surface [2]. Reyes et al. [3] the low velocity impact behavior of three layer thermoplastic laminates consisting of woven glass fiber and polypropylene has investigated for two different fiber volume configurations. Experimental testing showed a reduction in flexural strength of approximately 27% for the 50/50 and approximately 12% for 20/80 respectively after a 16 J impact. Özdemir et al. [4] the low velocity impact behaviors of glass fiber reinforced polypropylene thermoplastic composites were



The International Confer

In this research, effect of embedded delamination size on low velocity impact behavior of thermoplastic composites was investigated.

#### 2. Materials and Methods

In this study, two different types of thermoplastic composite samples were produced; nondelamination and embedded delamination. In the production of thermoplastic composite, 50% long glass fiber reinforced homopolypropylene composite granules produced by pultrusion method and glass fiber reinforced polypropylene composite prepregs were used. Release film was used to create embedded delamination. The materials used in the manufacturing are shown in figure 1.



**Figure 1. a)** Long glass fiber/polypropylene granule, **b)** Glass fiber reinforced prepreg, **c)** Realese film

Mechanical properties of long glass fiber reinforced granules used as core material are shown in Table 1 [8].

Technology



I I				
Mechanical Properties	Value	Unit	Test Method	
Density	1,33	g/cm <sup>3</sup>	ISO 1183	
Tensile Strength	121	MPa	ISO 527	
Tensile Module	10100	MPa	ISO 527	
Bending Strength	205	MPa	ISO 178	
Bending Module	9180	MPa	ISO 178	
IZOD Impact Resistance, Unnotched (11J)	48,5	kJ/m <sup>2</sup>	ISO 180	
IZOD Impact Resistance, Notched (11J)	17,5	kJ/m <sup>2</sup>	ISO 180	
HDT (0,45 MPa)	158	°C	ISO 75B	

Table 1. Mechanical properties of granules.

The orientation of the thermoplastic composite is set to [0/90/ Granule / 90/0]. In this orientation, the granules are considered to be the core material in the composite. The delamination material was placed between the prepreg laminates. Two different diameters(i.e., 13 mm and 26 mm) were used. Specimens were produced by hot pressing method. Specimens were first placed in the 3 mm thick mold and then produced in the Fontijne Presses - LabEcon60 Laboratory press machine. The specimens were processed in a hot pressing machine at 180 °C and under 100 kN for 6 minutes. After that allowed to cool to the room temperature. The specimens produced have dimensions of 350 x 350 mm. Specimens were cut to 100 x 100 mm size, making them suitable for the experiment. All manufacturing processes are given in Figure 2.



Figure 2. Manufacturing processes for embedded delamination composites

Composite materials may be subjected to various impacts depending on their application. Damage to the material caused by impact increases with increasing impact energy. In this study, low velocity impact test was applied to the specimens. Experiments were performed at energy levels of 10J, 20J, 30J, 40J and 50J. İmpactor used in the experiments have hemisperical shape and noses



at 12.7 mm diameter. The equipment was used in the experiment of this research are given in Figure 3.



Figure 3. a) Fractovis Plus low velocity impact testing machine, b) Impactor, c) Impactor noses

# **3. Results and Discussion**

During the impact tests, the energy is gradually raised from impact energy level of 10 up to 50J. In order to investigate the effects of embedded delamination on the impact behavior of thermoplastic composite, contact force– deformation diagrams of the specimens was investigated in detail in Figure 4. It can be said that the contact force is increased by increasing the impact energy level. In 10J energy, there is clearly no difference. In 20J energy, the maximum deformation decreased in the specimens with embedded delamination. All specimes were perforated at an energy level of 30J. In 40J energy, the maximum contact force decreased in the specimens with embedded delamination. In 50J energy, the maximum contact force is increased in the large diameter delamination specimen, while the others are about the same level.

# TICMET'19 The International Conference of Materials and Engineering Technology



Figure 4. Contact force-deformation for delamination diameters of 0,13,26 mm a) 10J energy level, b) 20J energy level, c) 30J energy level, d) 40J energy level, e) 50J energy level

Energy-time diagrams for delamination diameters of 0,13,26 mm are given in Figure 5. In 10J and 20J energy levels, rebounding situation was observed for all specimens. After the penetration energy, the absorbed energy values for diameter of 0 mm are approximate but the absorbed energy values for diameter of 13 mm continue to increase. While the absorbed energy values for diameters of 26 mm are approximate in 30J and 40J, the value increased in 50J energy level .



**Figure 5.** Energy-time for delamination diameters of 0,13,26 mm **a**) 10J energy level, **b**) 20J energy level, **c**) 30J energy level, **d**) 40J energy level, **e**) 50J energy level

#### 4. Conclusions

This study presents the influence of size of embedded delamination on impact behavior of thermoplastic composite, experimentally. According to the data obtained, the results can be summarized as follows:

• The maximum contact force and maximum deflection values are not significantly changed depending on the delamination size for the same energy level.

The International Conference of Materials and Engineering Technology

- The maximum contact force value of specimens with a delamination diameter of 13 mm decreased at all energy levels after the perforation thresholds.
- In 10J and 20J energy levels, rebounding situation was observed for all specimens.
- The maximum energy absorbed value is observed with diameters of 26 mm.

#### References

- 1. Mazumdar, S., Composites manufacturing: materials, product, and process engineering: Crc press, **2002**, (51-52).
- Abrate, S., Impact Engineering of Composite Structures, New York: Springer Wien, 2010, (98-99).
- 3. Reyes, G., and Sharma, U. Modeling and Damage Repair of Woven Thermoplastic Composites Subjected to Low Velocity Impact. Composite Structures 92, **2010**, 523–531.
- Özdemir, O., and Kandaş, H. Thickness and Temperature Effects on the Impact Behavior of Glass Fiber Reinforced Polypropylene Composites. Tekstil ve Mühendis, 2018, 25: 110, 103-112.
- Doğan, A., and Arıkan, V. Low Velocity Impact Response of E-glass Reinforced Thermoset and Thermoplastic Based Sandwich Composites. Composites Part B 127, 2017, 63-69.
- 6. Russo, P., Langella, A., Papa, I., Simeoli, G., and Lopresto, V. Thermoplastic Polyurethane/Glass Fabric Composite Laminates: Low Velocity Impact Behavior Under Extreme Temperature Conditions. Composite Structures 166, **2017**, 146–152.
- Sabancı, E., and Karakuzu, R. Effect of Embedded Delaminations Impact on Behavior of Laminated Composites. Pamukkale Üniversitesi Mühendislik Bilimleri Dergisi, 2013, Cilt 19, Sayı 7, 303-309.
- 8. http://www.nuhkompozit.com.tr/duramax-LFT50-PP.html

# ÇOK YÖNLÜ TEKERLERE SAHİP BİR ÇATALLI YÜKLEYİCİNİN HAREKET KONTROLÜNÜN DENEYSEL İNCELENMESİ

The International Conference

aterials and Engineering Technology

# MAHMUT CIMEN<sup>\*1</sup>, METE KALYONCU<sup>2</sup>

<sup>1</sup> Sekizli Makina ve Vinç San. Tic. A. Ş., 42050 Konya, TÜRKİYE
<sup>2</sup> Konya Teknik Ü. Mühendislik ve Doğa Bil. Fak. Makina Mühendisliği Bölümü, Konya, TÜRKİYE

#### Özet

Bu çalışmada, çok yönlü tekerlere sahip bir çatallı yükleyicinin hareket kontrolü deneysel olarak incelenmiştir. Çok yönlü tekerler, kullanıldığı aracın ileri-geri, çaprazlama-yanlama, olduğu yerde dönme ve her yöne gidebilme hareketlerini sağlayan 45 derece açılarla özel bir şekilde yerleştirilen ve özel bir geometriye sahip polimer kaplı özel periferik makaralardan olusmaktadır. Cok yönlü tekerler teorik olarak kullanıldığı aracın serbestlik derecesini artırmaktadır. İleri-geri hareketi esnasında tüm makaralar, dişli kutusu çıkış mili ekseninde dönmekte ve ötelenmektedir. Çapraz hareket sırasında ise bir ön, bir de arkadan olmak üzere yalnızca iki teker üzerinde bulunan makaralar dönme ve ötelenme hareketi yapmakta, diğer teker üzerinde yer düzlemiyle temas halinde olan makaralar ise kendi mil eksenleri etrafında dönerek ötelenmektedir. Yanlamasına hareketi esnasında ise arka ve ön tekerler birbirlerine göre ters yönde dönmektedir. Dönme hareketinin, yanal bir kuvvete dönüşmesiyle makaralar kendi mil eksenlerinde dönmekte ve oluşan yanal kuvvetin etkisiyle kaymaktadır. Günlük hayatta istifleme ve taşıma amacıyla kullanılan çatallı yükleyiciler; fonksiyonellik, basit kullanılabilirlik, kıvraklık, güvenlik ve manevra kabiliyeti açısından yetersiz düzeydedir. Özellikle dar alanlarda fazla manevra ihtiyacı böyle bir araçta hem fazla enerji ve zaman sarfiyatı, hem de sürücü açısından daha fazla dikkat gerektirmektedir. Çok yönlü tekerlere sahip çatallı yükleyiciler, hassas kullanım ve yüksek manevra kabiliyetleri sebebiyle günümüz endüstrisinde otomotivden havacılığa birçok alanda son derece kullanışlıdır. Çok yönlü tekerli çatallı yükleyicilerin optimum çalışma performansının sağlanması amacıyla, çatallı yükleyici prototipi üzerinde bir takım deneysel çalışmalar yapılmış ve bu çalışmalar sonucu elde edilen değerler grafikler üzerinde gösterilmiştir. Değerler prototip aracın elektrik motorlarını kontrol eden sürücülerden alınmış ve elde edilen deneysel değerler ile hesaplamalar sonucu elde edilen teorik değerler karşılaştırılmıştır. Beklenildiği üzere teorik sonuçlar ile deneysel sonuçlar arasında farklar oluşmuş, oluşan bu farklar grafikler üzerinden yorumlanmış, enerji tüketimi ve performans ile alakalı bazı sonuçlar elde edilmiştir.

Anahtar kelimeler: Çatallı yükleyici, Çok yönlü teker, Kinematik analiz, Mekatronik tasarım, Sistem dinamiği, Hareket kontrolü, Deney tasarımı.



#### 1. Giriş

Çatallı yükleyicinin yürüyüş sistemi bileşenlerinden biri olan çok yönlü tekerlekler, hareket sahanlığı oldukça dar alanlar için tasarlanmış başlıca omurga elemanlardandır. Çok yönlü tekerlekler aracın yönünün değişmesini sağlarken aracın oryantasyonu ise değişmeden kalır. [12] Bu tekerleğe herhangi bir yönlendirme yapmaya gerek duymadan ön, arka ve çapraz yönlerde aracın kayarak üzerinde hareket etmesine imkan veren ve her yönde hareketi kolaylıkla sağlayan bir tekerledir. Günümüzde karşımıza çıkan bu tekerlek, genellikle Ilon'un ileri sürdüğü tasarım esaslarına bağlı olarak dizayn edilmektedir. Ilon'un öne sürdüğü çok yönlü tekerlek, jant ile bu jant çevresinde 45 derece açı ile konumlanmış belli sayıda makaralardan oluşmaktadır. Tekerlek yönünde normal bir kuvvet için, açılı dizilmiş makaralar kuvvetin bir bölümünü tekerleğin dönme yönünde çevirmektedir. Her bir bağımsız tekerleğin yönü ve hızına bağlı olarak, sonuçta elde edilmiş tüm kuvvetler kombinasyonu istenilen yönde bir toplam kuvvet oluşturmaktadır. Makara yüzeyinin sadece küçük bir bölümü yere temas etmektedir.[11]

aterials and Engineering Technology



# Şekil 1.1. Çok yönlü tekerleklerin hareket kabiliyetlerinin basit gösterimi

Çok yönlü tekerlek kontrol sistemleri daha yüksek manevra kabiliyeti, yüksek hız kontrolü ,düşük hızlarda dar alanlarda dönebilme ve kolay park edebilme imkanı sağlamaktadır. İleri ve geri yürüyüş hareketi sırasında tüm polimer kaplı makaralar, redüktör çıkış mili ekseninde hem dönmekte, hem de ötelenmektedir. Çapraz hareketi esnasında ise bir ön, bir de arkadan olmak üzere yalnız iki tekerlek üzerinde bulunan periferik makaralar dönme ve ötelenme hareketi yapmakta, diğer tekerlek üzerinde yer düzlemine temas halinde olan makaralar ise kendi eksenleri etrafında dönerek ötelenmektedir. Yanlamasına hareketi sırasındaysa arka ve ön tekerlekler birbirlerine göre tam ters yönde dönmektedir. Tekerlekler ve zemin arasındaki sürtünmenin etkisi ile yanal bir kuvvet oluşmakta ,makaralar kendi eksenlerinde dönmekte ve oluşan bu yanal kuvvetin etkisi ile sistem kaymaktadır.

Çok yönlü tekerleklerin hareketleri sırasında tekerlek yönlendirme yoktur.Bu sebeple her bir tekerleğin farklı yönlerde ve farklı miktarlarda dönüşleri planlanan yönde hareket etmesini sağlar. Mobil aracın yön ve hız kontrolünde zemin sürtünme katsayısı doğrudan etkili bir parametredir. Bu parametre sistemin matematiksel modeline de dahil edilmiştir. Çok yönlü hareket için gerekli parametreler denkleme eklendikten sonra araç modelinin ivmesi, hızı ve konum değişimi Matlab programından elde edilmiştir.

Daha önce yapılmış olan teorik çalışmalar sonucu elde edilen verilerin doğruluğunu sağlamak ve sonuçları yakınsamak amacıyla Şekil 1.2 de görülen prototip üzerinde bir takım deneysel

çalışmalar yapılmış, ve bu çalışmalar sonucu elde edilen değerler grafikler üzerinde gösterilerek sonuçlar karşılaştırılmıştır.

he International

als and Engineering Technology



Şekil 1.2. Deneysel çalışmaların yapılabilmesi için üretilmiş test aracının üretim basamaklarından bazıları

# 1.1. Motor Sürücülerinden Verilerin Toplanması ve Analizi

Anlık hız, anlık güç ve anlık akım değerleri gibi veriler motorları control eden sürücülerden alınmıştır. Çok yönlü tekerlek sisteminde aynı anda 4 tekerlek sürülmektedir fakat sürücülerden veri çekme işlemini aynı anda sadece bir sürücü üzerinden yapabilmekteyiz. Bu sebeple deneysel çalışmalarımız da veriler sol ön tekeri kontrol eden (E1) sürücüsünden çekildi. Deneysel çalışmalar için 'Tester' moduna alınan bu yürüyüş sürücüsü ile araç düz zeminde ileri yönde 30 [s] boyunca sürüldü. Daha sonra ise sürücü üzerinde bulunan RS232 portundan seri olarak bağlanılarak Akım [Ah] ve Motor hızı [rpm] verileri çekildi. Bu veriler ve giriş parametre değerleri kullanılarak hesaplanmış Araç hızı [m/s] ve anlık güç tüketimi [watt] değerlerinin zaman içerisindeki değişimleri grafiklerde gösterilerek diğer hareketlerle karşılaştırmaları yapıldı. Bu çalışma sonucu elde edilen çıktılar aracın hassas kontrolü için kontrolcü arabiriminde mutlaka değerlendirilmelidir. Karşılaştırılmış 4 durumda aracın ileri gitme durumu referans alınmış olup, tüm artış, azalış ve karşılaştırma çalışmaları 1.2. İleri-Geri Düz Hareket Durumuna göre yorumlanmıştır.



Şekil 1.3. Test aracında bazı ölçümler yapılırken ve veriler toplanıp kayıt altına alınırken

# 1.2. İleri-Geri Düz Hareket

İleri-Geri düz hareket sırasında makaralar kendi eksenlerinde dönmemektedir. Bu durumda araç 4 tekerden tahrikli normal bir araç gibi çalışmaktadır. Aracın düz gidebilmesi için tüm tekerleklerin aynı yönde ve aynı hızla dönmesi gerekmektedir. Aksi durumda makaralar eksenlerine paralel doğrultuda sürtünmeye zorlanacak, düz giderken aracın yörüngesi bozularak hedeflendiği yere sapmayla varacaktır. Bu farkların oluşmasının bir sebebi motor hızlarının farklı olması,düzgün yataklama yapılmaması,makara kaplamasındaki bozulmalar yada yoldaki fiziksel bozukluklar olabilir. Özellikle insansız araçlarda çok önemli olan bu hata kaynakları ölçülebilir olmalı ve sistem geri beslemeleriyle yok edilmelidir. Aksi takdirde sistem doğru düzgün çalışmayacak hatta kazalara sebebiyet verebilecektir. Tekerin dönme hızları lazer destekli takometre yardımıyla araç gövdesine sabitlenmiş mekanik bir takometre kullanılarak hareketli araç üzerinde tekerlek tur sayısı verisine ulaşılabilinmiştir.



Şekil 1.4. Motor devri ile verimlilik arasındaki bağıntıyı gösteren grafik Yukarıdaki grafikten görüldüğü üzere AC motorların verimi düşük devirlerde çok düşük olup, verim değeri, teorik hesaplamalarda ortalama 1500 [rpm] değerindeki karşılığı olan % 85 olarak alınmıştır.



Şekil 1.5. Motor devri, akım ve tork arasındaki bağıntıyı gösteren grafik

Düz İleri-Geri hareket testinde aracın kalkış anında aşırı akım çekmesi beklenen bir durumdur. Motorun 1500 [rpm] değerine ulaşana kadar verimsiz çalışmasından kaynaklanan bu durum literatürde 'Demeraj' akımı diye geçer. Sürücünün PC arayüzünden girilen 'Acceleration Time' parametresi 2000 [ms] olduğu için araç maksimum hıza 2 saniye içinde çıkabilmekte ve 2. saniyeden itibaren motorların çektiği anlık akım ortalama 100 [ah]'e düşmektedir. Nominal anlık güç tüketimi 4,3 [kw] iken bu değer ilk 2 saniyede 8,3 [kw] değerini bulmaktadır.

The International Conference

aterials and Engineering Technology

**Çizelge 1.1.** İleri düz hareket sırasında E1 (Sol Ön) Sürücüsünden alınmış giriş ve çıkış parametreleri

Giriş para	ametre	eleri ve sabit	girdiler	Üretici verilerine göre	e Ölçülen çıkış değerleri				
T-Zaman [s]	п	Teker Çapı [m]	Çevrim Oranı (i)	T-Moment [Nm]	[D]-Tekerlek Hızı [rpm]	[I]-Akım [Ah]	[P]-Anlık Güç [Kw*100]	[V]-Araç Hızı [m/s*100]	N-Motor Hızı [rpm]
0	3,14	0,458	26,8	0	0,0	0	0,0	0,0	0
0,5	3,14	0,458	26,8	42	27,5	269	324,0	65,9	737
1	3,14	0,458	26.8	40	58,6	248	657,3	140,4	1570
1,5	3,14	0,458	26,8	38	78,4	221	835,6	187,9	2101
2	3,14	0,458	26,8	24	112,3	185	755,9	269,1	3009
2,5	3,14	0,458	26,8	16	129,3	145	580,3	309,9	3465
3	3,14	0,458	26,8	12	138,3	112	465,5	331,4	3706
3,5	3,14	0,458	26,8	11	140,2	102	432,7	336,1	3758
4	3,14	0,458	26,8	11	140,1	102	432,4	335,9	3756
4,5	3,14	0,458	26,8	11	138,8	96	428,3	332,7	3720
5	3,14	0,458	26,8	11	139,4	100	430,0	334,0	3735
5,5	3,14	0,458	26,8	11	139,6	100	430,6	334,5	3740
6	3,14	0,458	26,8	- 11	139,4	94	430,1	334,1	3736
6,5	3,14	0,458	26,8	11	139,4	100	430.3	334,2	3737
7	3,14	0,458	26,8	11	139,6	96	430,6	334,5	3740





**Şekil 1.6.** İleri düz hareket sırasında E1 (Sol Ön) Sürücüsünden alınmış giriş ve çıkış parametrelerinin grafiksel gösterimi

# 1.3. Sağ-Sol Yanal Hareket

Aracın saga-sola kayabilmesi için tekerlekler üzerinde bulunan polimer kaplı makaralar mil eksenlerinde dönmeye zorlanmaktadır. Araç yan gitmeye başladığı anda yere temas eden makara eksenine paralel sürtünme meydana gelir. Yer düzleminde oluşan 45°'lik vektörlerin bileşkesi aracı yan gitmeye zorlamaktadır. Bir yandan tekerlek dönmekte olduğundan sürekli bir sonraki makara gelir ve hareket boyunca bu böyle devam eder. Bu hareketin oluşabilmesi için sağ ve sol

taraftaki motorlar birbirlerine ters yönde dönmesi gerekmektedir. Bu yönler tekerleklerin "X" veya "O" düzeninde oluşlarına göre değişkenlik göstermektedir. Ayrıca tekerleklerin yerleşim düzeninin kare şeklinden uzaklaştıkça bu hareket esnasında oluşacak sürtünme kuvvetinin artacağı beklenmiştir.

The International Conference

aterials and Engineering Technology

Giriş parametreleri ve sabit girdiler				Üretici verilerine göre	Ölçülen çıkış değerleri					
T-Zaman [s]	п	Teker Çapı [m]	Çevrim Oranı (i)	T-Moment [Nm]	[D]-Tekerlek Hızı [rpm]	[I]-Akım [Ah]	[P]-Anlık Güç [Kw*100]	[V]-Araç Hızı [m/s*100]	N-Motor Hızı [rpm]	
0	3,14	0,458	26,8	0	0,0	0	0,0	0,0	0	
0,5	3,14	0,458	26,8	42	23,8	208	280,5	57,1	638	
1	3,14	0,458	26,8	41	54,7	196	628,7	131,0	1465	
1,5	3,14	0,458	26,8	30	78,1	172	656,9	187,1	2092	
2	3,14	0,458	26,8	27	112,1	154	849,2	268,8	3005	
2,5	3,14	0,458	26,8	24	136,5	132	918,9	327,2	3658	
3	3,14	0,458	26,8	18	136,0	126	686,7	326,0	3645	
3,5	3,14	0,458	26,8	14	136,2	120	534,8	326,4	3650	
4	3,14	0,458	26,8	12	136,5	116	459,6	327,2	3659	
4,5	3,14	0,458	26,8	12	136,2	112	458,4	326,4	3650	
5	3,14	0,458	26,8	12	136,0	115	457,8	326,0	3645	
5,5	3,14	0,458	26,8	12	135,4	115	455,9	324,7	3630	
6	3,14	0,458	26,8	12	135,6	118	456,6	325,1	3635	
6,5	3,14	0,458	26,8	12	135,8	116	457,2	325,5	3640	
7	3.14	0,458	26,8	12	136,0	118	457,8	326,0	3645	
			GÜC	[P] = MOMENT	[T] x DEVIR	INI x 2 x	II x 1/60			

Çizelge 1.2. Sağa yan hareke	t sırasında E1 (Sol Ön)	Sürücüsünden alınan giriş ve çıkış
	parametreleri	

Çizelge 1.2'den görüldüğü gibi bir makaradan diğerine geçiş anında meydana gelen sürtünme kuvvetleri, ileri gitme durumuna göre artmış, bunun sonucunda ise güç tüketimi yaklaşık %7 artmıştır. Aracın hızında da %2 azalma meydana gelmiştir. Oluşan kayıpların etkisinden dolayı systemin verimli tüketime ulaşma süresinde yaklaşık 650 [ms] gecikme yaşanmıştır. Güç grafiği incelendiğinde ise araç maksimum hıza ulaştığı anda ortalama 120 [ah] akım çekmeye başlamıştır.



Şekil 1.7. Sağa yan hareket sırasında ölçülen giriş ve çıkış parametrelerinin grafiksel gösterimi



# 1.4. Çapraz Hareket

Çapraz hareket sırasında tüm yük sadece 2 motorun üstündedir. Yapılacak seçimde bu göz önünde bulundurulması gereken kritik bir noktadır. Çizelge 5.3'de verilmiş deneysel çalışma sonuçlarından da görüldüğü üzere harcanan güç 1,7, çekilen akım ise nerdeyse 2 katına çıkmıştır. Şüphesiz ki aracın maksimum güç tüketimi çapraz hareketler esnasında gerçekleşmekte.

Giriş parametreleri ve sabit girdiler				Üretici verilerine göre	Ölçülen çıkış değerleri				
T-Zaman [s]	п	Teker Çapı [m]	Çevrim Oranı (i)	T-Moment [Nm]	[D]-Tekerlek Hızı [rpm]	[I]-Akım [Ah]	[P]-Anlık Güç [Kw*100]	[V]-Araç Hızı [m/s*100]	N-Motor Hızı [rpm]
0	3,14	0,458	26,8	0	0,0	0	0,0	0,0	0
0,5	3,14	0,458	26,8	60	23,1	275	389,4	27,7	620
1	3,14	0,458	26,8	66	58,6	325	1084,6	70,2	1570
1,5	3,14	0,458	26,8	58	91,8	312	1492,8	110,0	2459
2	3,14	0,458	26,8	46	100,9	301	1302,4	121,0	2705
2,5	3,14	0,458	26,8	39	111,0	283	1214,0	133,0	2974
3	3,14	0,458	26,8	30	138,5	210	1165,6	166,0	3712
3,5	3,14	0,458	26,8	22	137,6	200	849,5	165,0	3689
4	3,14	0,458	26,8	19	138,4	200	737,8	165,9	3710
4,5	3,14	0,458	26,8	19	138,8	194	739,8	166,3	3720
5	3,14	0,458	26,8	19	138,2	198	736,8	165,7	3705
5,5	3,14	0,458	26,8	19	138,1	198	736,0	165,5	3701
6	3,14	0,458	26,8	19	138,1	192	736,2	165,5	3702
6,5	3,14	0,458	26,8	19	138,2	198	736,8	165,7	3705
7	3,14	0,458	26,8	19	138,1	194	735,8	165,5	3700

**Çizelge 1.3.** Çapraz hareket sırasında E1 (Sol Ön) Sürücüsünden alınmış giriş ve çıkış parametreleri

Tam çapraz hareketler yerine 'Curved' adı verilen parabolik rampalar izletilerek bu çok yüksek tüketime sahip düşük verimli durum daha az enerji tüketebilir hale getirilebilir. Bunun için sisteme aynı anda 2 giriş parametresi girilmesi gerekmektedir. Hem x veya y ekseninde ilerleme, hem de dönme hareketi verilmelidir.





# 1.5. Olduğu Yerde Dönme Hareketi

Bu hareket sırasında hem tüm tekerlekler hem de bağımsız makaralar sırasıyla dönmektedir. Tekerleklerin birbirlerine olan mesafeleri eş olmadığı için oluşan sürtünme kuvvetleri fazladır. İstenmeyen sürtünme durumları hem güç tüketimi açısından hem de makaraların ömrü açısından irdelenmesi ve azaltılması gereken bir değerdir. Yapılan deneysel çalışmalar sonucu elde edilen ve Çizelge 5.4'de görüldüğü üzere, ileri düz hareket durumuna nazaran harcanan güç %18 artmıştır.

The International Conference

Giriş par	ametro	eleri ve sabit	girdiler	Üretici verilerine göre	e Ölçülen çıkış değerleri				
T-Zaman [s]	п	Teker Çapı [m]	Çevrim Oranı (i)	T-Moment [Nm]	[D]-Tekerlek Hızı [rpm]	[I]-Akım [Ah]	[P]-Anlık Güç [Kw*100]	[V]-Araç Hızı [rad/s*100]	N-Motor Hızı [rpm]
0	3,14	0,458	26,8	0	0,0	0	0,0	0,0	0
0,5	3,14	0,458	26,8	56	16,2	245	255,0	18,5	435
1	3,14	0,458	26,8	48	36,8	234	494,9	41,8	985
1,5	3,14	0,458	26,8	46	49,4	226	637,9	56,3	1325
2	3,14	0,458	26,8	40	87,8	204	985,5	100,0	2354
2,5	3,14	0,458	26,8	28	131,7	182	1034,5	150,0	3530
3	3,14	0,458	26.8	26	136,1	145	992,7	155,0	3648
3,5	3,14	0,458	26,8	19	135,3	135	720,9	154,0	3625
4	3,14	0,458	26,8	14	136,5	132	536,0	155,4	3658
4,5	3,14	0,458	26,8	14	136,0	127	534,1	154,8	3645
5	3,14	0,458	26,8	14	136,3	125	535,1	155,1	3652
5,5	3,14	0,458	26,8	14	136,5	128	535,9	155,4	3657
6	3,14	0,458	26,8	14	136,3	135	535,1	155,1	3652
6,5	3,14	0,458	26,8	14	136,1	132	534,6	155,0	3648
7	3,14	0,458	26,8	14	135,8	135	533,4	154,6	3640

**Çizelge 1.4.** Saat yönünde olduğu yerde dönme hareketi esnasında E1 (Sol Ön) Sürücüsünden alınmış giriş ve çıkış parametreleri

Bu durum bize makaraların ömrü açısından da bir fikir vermektedir. Bu güç tüketimi tekerleklerin birbirine olan uzaklıkları ile de doğrudan etkilidir. Aracın en fazla sürtünme kuvvetine maruz kaldığı hareket durumudur.



Şekil 1.9. Saat yönünde olduğu yerde dönme hareketi esnasında ölçülen giriş ve çıkış parametrelerinin grafiksel gösterimi

#### 2. Sistemin hareket denkleminin elde edilmesi ve Matlab üzerinde modellenmesi

Bu çalışmada sistem cevabını karakterize etme, kontrol sistemleri tasarlama ve sistem davranışını tahmin etmek için Matlab/Simulink'de mobil aracın matematiksel modeli oluşturulmuş ve Matlab/Simmechanics'de benzetim modeli oluşturulmuştur. Simulink, çok disiplinli sistemlerin davranışalarını modellemek ve simule etmek için dahası bu sistemleri geliştirmek için gerekli

Technology



The International

Mobil aracın benzetim modeli Matlab-Simulink ve Simmechanics blokları kullanılarak oluşturulmuştur. Mobil araç üç boyutlu bilgisayar-destekli tasarım (Solidworks) ortamında şasi ve buna bağlı olan dört çok yönlü tekerlek olarak modellenmiş ve oluşturulan bu model Simulink ortamına aktarılmıştır.

SimMechanics, mekanik sistemlerin modellenmesini sağlayan bir simülasyon paketidir. Simulink ortamında çalışan bu paket, geniş kapsamlı fonksiyon kitaplığıyla Simulink ortamını çoklu gövdeli mekanik modelleme olanağıyla genişletmektedir. Yaratılan modellerin otomatik olarak 3 boyutlu şekli görülebilmekte ve benzetim sırasında hareketler canlı olarak izlenebilmektedir.

#### 2.1.Benzetim Model ile Yön Kontrolü

Yerel koordinat düzleminde {R} mobil robota monte edilmiş bir çok yönlü teker için eksenler  $X_R Y_R Z_R$  olmak üzere; şekil 2 de de görüldüğü gibi A noktası merkez, diğer geometrik parametreler ise:

 $\alpha$ , *GA* vektörünün açısı, G robotun geometrik merkezinin orijin noktası olmak üzere, A  $X_R$  eksenine bağlı olarak geometrik merkez,  $\beta$  ise merkez tekerlek ekseni ve *GA* vektörü arasındaki açıdır.



Şekil 2.1. Çok yönlü tekerin koordinat düzleminde gösterimi [13]

Robotun ilk ötelenme hızının {R} cinsinden ifadesi ise  $[\vec{X}_R \quad \dot{Y}_R]$ ;  $Z_R$  ye bağlı dönme hızı $\dot{\theta}$  dır. Tekerlek merkezinin hızı öteleme hızlarının  $\vec{x}_R, \vec{y}_R$  toplamıyla hesaplanır ve dönme hızına bağlı hız  $l\dot{\theta}$  şekil 1 de görüldüğü gibidir. Böylece, tekerlek merkezi A'nın makara temas ekseni boyunca hız bileşeni aşağıdaki gibi ifade edilir:

$$\dot{x}_{R} \cos\left[\frac{\pi}{2} - \left(\frac{\pi}{2} - (\alpha + \beta)\right) - \left(\frac{\pi}{2} - \gamma\right)\right] + \dot{y}_{R} \cos\left[\left(\frac{\pi}{2} - (\alpha + \beta)\right) + \left(\frac{\pi}{2} - \gamma\right)\right] + l\dot{\theta} \cos\left[\alpha + \left(\frac{\pi}{2} - (\alpha + \beta)\right) + \left(\frac{\pi}{2} - \gamma\right)\right] \\ = \dot{x}_{R} \cos\left[\left(\alpha + \beta + \gamma\right) - \left(\frac{\pi}{2}\right)\right] + \dot{y}_{R} \cos\left[\pi - (\alpha + \beta + \gamma)\right] + l\dot{\theta} \cos\left[\pi - (\beta + \gamma)\right] \\ = \dot{x}_{R} \sin\left(\alpha + \beta + \gamma\right) - \dot{y}_{R} \cos\left(\alpha + \beta + \gamma\right) - \dot{\theta} l\cos\left(\beta + \gamma\right) \\ = \left[\sin\left(\alpha + \beta + \gamma\right) - \cos\left(\alpha + \beta + \gamma\right) - l\cos\left(\beta + \gamma\right)\right] \cdot \left[\dot{x}_{R} \quad \dot{y}_{R} \quad \dot{\theta}\right]^{T} \\ \dots (1)$$

MET

Eğer makaranın kontak noktası boyunca hiç kayma olmuyorsa, aynı tekerlek dönüş hızı olan  $\dot{\varphi}$ . ile aynıdır. Çok yönlü teker için sabit denklem:

$$\begin{bmatrix} \sin(\alpha + \beta + \gamma) & -\cos(\alpha + \beta + \gamma) & -l\cos(\beta + \gamma) \end{bmatrix} \begin{bmatrix} \dot{x}_{R} & \dot{y}_{R} & \dot{\theta} \end{bmatrix}^{\mathrm{T}} = r\dot{\phi}\cos\gamma$$
... (2)

Robotun şasi ataleti {I}'nin robot şasisi {R}' ye göre rotasyon matrisi ise aşağıdaki gibidir:

 ${}^{R}\boldsymbol{R}_{I}(\theta) = \begin{bmatrix} \cos\theta & \sin\theta & 0\\ -\sin\theta & \cos\theta & 0\\ 0 & 0 & 1 \end{bmatrix}$ 

UWE Bristol

 $\theta$ ,  $X_R$  ve  $Y_R$  arasındaki açıdır. Robotun şasisi {R} için hız vektörü ise;

$$\dot{\boldsymbol{\xi}}_{R} = \begin{bmatrix} \dot{\boldsymbol{x}}_{R} & \dot{\boldsymbol{y}}_{R} & \dot{\boldsymbol{\theta}} \end{bmatrix}^{\mathrm{T}}$$
$$\dot{\boldsymbol{\xi}}_{R} = {}^{R}\boldsymbol{R}_{I}(\boldsymbol{\theta})\dot{\boldsymbol{\xi}}_{I}$$

Denklem (2) aşağıdakine dönüştürülür:

$$\begin{bmatrix} \sin(\alpha + \beta + \gamma) & -\cos(\alpha + \beta + \gamma) & -l\cos(\beta + \gamma) \end{bmatrix} \cdot {}^{R}R_{I}(\theta)\dot{\xi}_{I} = r\dot{\phi}\cos\gamma$$
...(3)

Makaranın temas noktasına dikey olan yönde hareket kısıtlı değildir çünkü pasif temas makarası serbest dönmektedir. Bunun sonucunda aşağıdaki hız bağıntısına ulaşılır:

$$\dot{x}_{R}\sin\left[\frac{\pi}{2}-\left(\frac{\pi}{2}-(\alpha+\beta)\right)-\left(\frac{\pi}{2}-\gamma\right)\right]-\dot{y}_{R}\sin\left[\left(\frac{\pi}{2}-(\alpha+\beta)\right)+\left(\frac{\pi}{2}-\gamma\right)\right]-l\dot{\theta}\sin\left[\alpha+\left(\frac{\pi}{2}-(\alpha+\beta)\right)+\left(\frac{\pi}{2}-\gamma\right)\right]$$
$$=r\dot{\phi}\sin\gamma+r_{sw}\dot{\phi}_{sw}$$
$$\therefore\left[\cos\left(\alpha+\beta+\gamma\right)\quad\sin\left(\alpha+\beta+\gamma\right)\quad l\sin\left(\beta+\gamma\right)\right]\cdot\left[\dot{x}_{R}\ \dot{y}_{R}\ \dot{\theta}\right]^{\mathrm{T}}+r\dot{\phi}\sin\gamma+r_{sw}\dot{\phi}_{sw}=0$$

The International Conference of Materials and Engineering Technology

Yukarıdaki denklem aşağıdakine dönüştürüldüğünde:

$$\begin{bmatrix} \cos(\alpha + \beta + \gamma) & \sin(\alpha + \beta + \gamma) & l\sin(\beta + \gamma) \end{bmatrix}^{R} R_{I}(\theta) \dot{\xi}_{I} + r \dot{\phi} \sin \gamma + r_{sw} \dot{\phi}_{sw} = 0$$
...(4)

Şekil 2.1 deki gibi çok yönlü tekerlekli bir mobil robot olduğu farz edilirse;

 $\alpha_i$ ,  $\beta_i$  ve  $\gamma_i$  açıları, 4 tekerlek monte edildiğinde, i=1,2,3,4 için çizelge 1 deki gibi olur. Denklem 3 ten yola çıkıldığında;

$$\begin{bmatrix} \sin(\alpha_{1}+\beta_{1}+\gamma_{1}) & -\cos(\alpha_{1}+\beta_{1}+\gamma_{1}) & -l_{1}\cos(\beta_{1}+\gamma_{1}) \\ \sin(\alpha_{2}+\beta_{2}+\gamma_{2}) & -\cos(\alpha_{2}+\beta_{2}+\gamma_{2}) & -l_{2}\cos(\beta_{2}+\gamma_{2}) \\ \sin(\alpha_{3}+\beta_{3}+\gamma_{3}) & -\cos(\alpha_{3}+\beta_{3}+\gamma_{3}) & -l_{3}\cos(\beta_{3}+\gamma_{3}) \\ \sin(\alpha_{4}+\beta_{4}+\gamma_{4}) & -\cos(\alpha_{4}+\beta_{4}+\gamma_{4}) & -l_{4}\cos(\beta_{4}+\gamma_{4}) \end{bmatrix} \cdot {}^{R}R_{I}(\theta)\dot{\xi}_{I} = \begin{bmatrix} r_{1}\dot{\phi}_{1}\cos\gamma_{1} \\ r_{2}\dot{\phi}_{2}\cos\gamma_{2} \\ r_{3}\dot{\phi}_{3}\cos\gamma_{3} \\ r_{4}\dot{\phi}_{4}\cos\gamma_{4} \end{bmatrix}$$
...(5)

Elde edilir.

Her çok yönlü tekerin eşit ve bağlantı uzaklıkları eşit alındığında,  $r_i = r, l_i = 1, 2, 3, 4$  ve çizelge 1 deki parametreler denklem 5 te yerine koyulduğunda aşağıdaki ters kinematik denklemi elde edilir:

$$\begin{bmatrix} \dot{\varphi}_{1} \\ \dot{\varphi}_{2} \\ \dot{\varphi}_{3} \\ \dot{\varphi}_{4} \end{bmatrix} = -\left(\sqrt{2}/r\right) \begin{bmatrix} \sqrt{2}/2 & \sqrt{2}/2 & l\sin(\pi/4 - \alpha) \\ \sqrt{2}/2 & -\sqrt{2}/2 & l\sin(\pi/4 - \alpha) \\ -\sqrt{2}/2 & -\sqrt{2}/2 & l\sin(\pi/4 - \alpha) \\ -\sqrt{2}/2 & \sqrt{2}/2 & l\sin(\pi/4 - \alpha) \\ -\sqrt{2}/2 & \sqrt{2}/2 & l\sin(\pi/4 - \alpha) \end{bmatrix}$$
$$\cdot \begin{bmatrix} \cos\theta & \sin\theta & 0 \\ -\sin\theta & \cos\theta & 0 \\ 0 & 0 & 1 \end{bmatrix} \begin{bmatrix} \dot{x}_{1} \\ \dot{y}_{1} \\ \dot{\theta} \end{bmatrix}$$



The International Con

terials and Engineering Technology

UWE Bristol

Çizelge 2.1. Çok yönlü tekerin parametreleri



Şekil 2.2. 4 Tekerlekli çok yönlü robot [13]

Ţx,	1	$\dot{\varphi}_1$
ý,	$=-(\sqrt{2}/2)rJ^{+}$	$\varphi_2$
Ù.		ψ <sub>3</sub>

Çok yönlü tekerli aracın ileri kinematik denklemi ters kinematik denklemin jakobian'ı alınarak aşağıdaki gibi elde edilir [13];



UWE dithe Bristol Engla

$$J^+ = (J^T J)^{-1} J^T$$

Burada  $J^+$  J 'nin pseydoinversidir.



Şekil 2.3. 4 Tekerlekli çok yönlü robotun serbest cisim diyagramı [13] Şekil 2.3 teki gibi bir çok yönlü araç ele alındığında, pozisyon vektörü  $r_{G=}|x_l \quad y_l|$  ile G geometrik merkez olmak üzere, bağıl pozisyon vektörü  $r_{G'/G} = [-d_1 \quad -d_2]^T \{R\}$ 'ye bağlıdır. Hız  ${}^{R}\boldsymbol{v}_{G}$ aşağıdaki gibi ifade edilir;

$${}^{R}\boldsymbol{v}_{G} = \begin{bmatrix} \cos\theta & \sin\theta \\ -\sin\theta & \cos\theta \end{bmatrix} \begin{bmatrix} \dot{x}_{I} \\ \dot{y}_{I} \end{bmatrix} = \begin{bmatrix} \dot{x}_{I}\cos\theta + \dot{y}_{I}\sin\theta & -\dot{x}_{I}\sin\theta + \dot{y}_{I}\cos\theta \end{bmatrix}^{\mathrm{T}}$$

...(9)

aterials and Engineering Technology

Conference

The International



The International Conference

of Materials and Engineering Technology

$${}^{R}\boldsymbol{v}_{G'} = {}^{R}\boldsymbol{v}_{G} + \dot{\boldsymbol{\theta}}\boldsymbol{k}_{R} \times {}^{R}\boldsymbol{r}_{G'/G}$$
$$= \left(\dot{x}_{I}\cos\theta + \dot{y}_{I}\sin\theta + \dot{\boldsymbol{\theta}}d_{2}\right)\boldsymbol{i}_{R} + \left(-\dot{x}_{I}\sin\theta + \dot{y}_{I}\cos\theta - \dot{\boldsymbol{\theta}}d_{1}\right)\boldsymbol{j}_{R}$$
...(10)

Toplam kinetik enerji aşağıdaki gibi ifade edilir:

$$T = \frac{1}{2} \left[ m_b \mathbf{v}_{G'}^{\mathrm{T}} \mathbf{v}_{G'} + I_b \dot{\theta}^2 + \sum_{i=1}^4 m_{wi} \left( r \dot{\phi}_i \right)^2 + \sum_{i=1}^4 I_i \dot{\phi}_i^2 \right]$$
...(11)

 $m_b$  Platformun ağırlığı,  $m_{wi}$  i nolu tekerleğin ağırlığı, i=1,2,3,4;  $\iota_b z_R$  ye bağlı ataletsel moment,  $\iota_i$  i nolu tekerleğin ataletsel momenti,  $\dot{\theta}$  platformun açısal hızı ve  $\dot{\phi}_i$  tekerleğin açısal hızı ve r her tekerin yarıçapıdır. Toplam potansiyel enerji V = 0 olduğundan; Lagrange denklemi L=T-V=T aşağıdaki gibi elde edilir;

$$\begin{split} L &= \frac{1}{2} I_b \dot{\theta}^2 + \frac{1}{2} m_b \Big[ \Big( \dot{x}_I \cos \theta + \dot{y}_I \sin \theta + \dot{\theta} d_2 \Big)^2 + \Big( -\dot{x}_I \sin \theta + \dot{y}_I \cos \theta - \dot{\theta} d_1 \Big)^2 \Big] \\ &+ \frac{1}{2} m_w \Big\{ \Big[ -\dot{x}_I \big( \cos \theta - \sin \theta \big) - \dot{y}_I \big( \sin \theta + \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \\ &+ \Big[ -\dot{x}_I \big( \cos \theta + \sin \theta \big) - \dot{y}_I \big( \sin \theta - \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \\ &+ \Big[ -\dot{x}_I \big( -\cos \theta + \sin \theta \big) + \dot{y}_I \big( \sin \theta + \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \\ &+ \Big[ \dot{x}_I \big( \cos \theta + \sin \theta \big) - \dot{y}_I \big( -\sin \theta + \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \\ &+ \frac{1}{2} \frac{1}{r^2} I_1 \Big[ -\dot{x}_I \big( \cos \theta - \sin \theta \big) - \dot{y}_I \big( \sin \theta + \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \\ &+ \frac{1}{2} \frac{1}{r^2} I_2 \Big[ -\dot{x}_I \big( \cos \theta + \sin \theta \big) - \dot{y}_I \big( \sin \theta - \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \\ &+ \frac{1}{2} \frac{1}{r^2} I_3 \Big[ -\dot{x}_I \big( -\cos \theta + \sin \theta \big) + \dot{y}_I \big( \sin \theta + \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \\ &+ \frac{1}{2} \frac{1}{r^2} I_3 \Big[ -\dot{x}_I \big( \cos \theta + \sin \theta \big) - \dot{y}_I \big( \sin \theta + \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \\ &+ \frac{1}{2} \frac{1}{r^2} I_4 \Big[ \dot{x}_I \big( \cos \theta + \sin \theta \big) - \dot{y}_I \big( -\sin \theta + \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \\ &+ \frac{1}{2} \frac{1}{r^2} I_4 \Big[ \dot{x}_I \big( \cos \theta + \sin \theta \big) - \dot{y}_I \big( -\sin \theta + \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \\ &+ \frac{1}{2} \frac{1}{r^2} I_4 \Big[ \dot{x}_I \big( \cos \theta + \sin \theta \big) - \dot{y}_I \big( -\sin \theta + \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \\ &+ \frac{1}{2} \frac{1}{r^2} I_4 \Big[ \dot{x}_I \big( \cos \theta + \sin \theta \big) - \dot{y}_I \big( -\sin \theta + \cos \theta \big) - \sqrt{2} \dot{\theta} l \sin \big( \pi/4 - \alpha \big) \Big]^2 \end{split}$$

441

Dinamik model ise;

$$\frac{\mathrm{d}}{\mathrm{d}t}\left(\frac{\partial L}{\partial \dot{q}_i}\right) - \frac{\partial L}{\partial q_i} = F_i, \quad i = 1, 2, 3$$

JWE

...(13)

 $q_i$ i nolu genelleştirilmiş koordinat ve  $F_i$ i nolu genelleştirilmiş kuvvet / tork'tur. Genelleştirilmiş koordinat vektörü;

of Materials and Engineering Technology

The International Conference

$$\boldsymbol{q} = \begin{bmatrix} q_1 & q_2 & q_3 \end{bmatrix}^{\mathrm{T}} = \begin{bmatrix} x_1 & y_1 & \theta \end{bmatrix}^{\mathrm{T}}$$

.  $f_i$  Tekerleğin temas yüzeyinin sürtünme kuvveti olmak üzere;

$$F_{1} = \sum_{i=1}^{4} \left(\tau_{i} - r \operatorname{sgn}\left(\dot{\phi}_{i}\right) f_{i}\right) \frac{\partial \varphi_{i}}{\partial x_{I}} = \sum_{i=1}^{4} \left(\tau_{i} - r \operatorname{sgn}\left(\dot{\varphi}_{i}\right) f_{i}\right) \frac{\partial \dot{\varphi}_{i}}{\partial \dot{x}_{I}}$$
...(14)

Denklem (6) da alınırsa;

$$\frac{\partial \dot{\varphi_1}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_2}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta + \sin \theta); \\ \frac{\partial \dot{\varphi_3}}{\partial \dot{x}_I} = \frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = \frac{1}{r} (\cos \theta + \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta + \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta + \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{r} (\cos \theta - \sin \theta); \\ \frac{\partial \dot{\varphi_4}}{\partial \dot{x}_I} = -\frac{1}{$$

Böylece;

$$F_{1} = \left[\tau_{1} - r\operatorname{sgn}(\dot{\phi}_{1})f_{1}\right] \left[-\frac{1}{r}\left(\cos\theta - \sin\theta\right)\right] + \left[\tau_{2} - r\operatorname{sgn}(\dot{\phi}_{2})f_{2}\right] \left[-\frac{1}{r}\left(\cos\theta + \sin\theta\right)\right] + \left[\tau_{3} - r\operatorname{sgn}(\dot{\phi}_{3})f_{3}\right] \left[\frac{1}{r}\left(\cos\theta - \sin\theta\right)\right] + \left[\tau_{4} - r\operatorname{sgn}(\dot{\phi}_{4})f_{4}\right] \left[\frac{1}{r}\left(\cos\theta + \sin\theta\right)\right]$$
(15)

...(15) Aynı Şekilde;

$$F_{2} = \sum_{i=1}^{4} \left(\tau_{i} - r \operatorname{sgn}(\dot{\phi}_{i})f_{i}\right) \frac{\partial \dot{\phi}_{i}}{\partial \dot{y}_{i}} = \left[\tau_{1} - r \operatorname{sgn}(\dot{\phi}_{1})f_{1}\right] \left[-\frac{1}{r} (\sin \theta + \cos \theta)\right] \\ + \left[\tau_{2} - r \operatorname{sgn}(\dot{\phi}_{2})f_{2}\right] \left[-\frac{1}{r} (\sin \theta - \cos \theta)\right] + \left[\tau_{3} - r \operatorname{sgn}(\dot{\phi}_{3})f_{3}\right] \left[\frac{1}{r} (\sin \theta + \cos \theta)\right] \\ + \left[\tau_{4} - r \operatorname{sgn}(\dot{\phi}_{4})f_{4}\right] \left[\frac{1}{r} (\sin \theta - \cos \theta)\right] \\ \dots (16) \\ F_{3} = \sum_{i=1}^{4} \left(\tau_{i} - r \operatorname{sgn}(\dot{\phi}_{i})f_{i}\right) \frac{\partial \dot{\phi}_{i}}{\partial \dot{\theta}} = \left(\tau_{1} + \tau_{2} + \tau_{3} + \tau_{4}\right) \left[-\frac{\sqrt{2}}{r} l \sin(\pi/4 - \alpha)\right]$$

+ 
$$\left[\operatorname{sgn}(\dot{\varphi}_1)f_1 + \operatorname{sgn}(\dot{\varphi}_2)f_2 + \operatorname{sgn}(\dot{\varphi}_3)f_3 + \operatorname{sgn}(\dot{\varphi}_4)f_4\right] \left[\sqrt{2l}\sin(\pi/4 - \alpha)\right]$$
...(17)

Denklemin düzenlenmesi sonucu genel hareket denklemi;

UWE Bristol

$$M(q) \ddot{q} + C(q, \dot{q}) \dot{q} + B^{T} S f = \frac{1}{r} B^{T} \tau$$

$$\ddot{q} = M^{-1} [\frac{1}{r} B^{T} \tau - C \dot{q} - B^{T} S f]$$
Olduğu yerde,
$$q = [x_{1}y_{1} \dot{\theta}]^{T}$$

$$\tau = [\tau_{1} \tau_{2} \tau_{3} \tau_{4}]^{T}$$

$$f = [f_{1} f_{2} f_{3} f_{4}]^{T}$$
S = diag[ sgn( $\dot{q}_{1}$ ) sgn( $\dot{q}_{2}$ ) sgn( $\dot{q}_{3}$ ) sgn( $\dot{q}_{4}$ )]<sup>T</sup>

$$M = [m_{ij}]_{3x3}, m_{12} = m_{21} = 0$$

$$m_{11} = m_{22} = m_{b} + 4(m_{w} + \frac{l_{w}}{r^{2}})$$

$$m_{13} = m_{31} = m_{b}(d_{1} \sin\theta + d_{2} \cos\theta)$$

$$m_{23} = m_{b}(d_{1}^{2} + d_{2}^{2}) + l_{b} + 8(m_{w} + \frac{1}{r^{2}}) l^{2} \sin^{2}(\pi/4 + \alpha)$$

$$B = \begin{bmatrix} \cos\theta + \sin\theta & \sin\theta - \cos\theta & -l\sqrt{2}\sin(\pi/4 + \alpha) \\ \cos\theta - \sin\theta & \sin\theta + \cos\theta & -l\sqrt{2}\sin(\pi/4 + \alpha) \\ -\cos\theta - \sin\theta & -\sin\theta + \cos\theta & -l\sqrt{2}\sin(\pi/4 + \alpha) \\ -\cos\theta + \sin\theta & -\sin\theta - \cos\theta & -l\sqrt{2}\sin(\pi/4 + \alpha) \end{bmatrix}$$

$$C = \begin{bmatrix} 0 & 0 & m_{b} \dot{\theta} (d_{1} \cos\theta - d_{2} \sin\theta) \\ 0 & 0 & m_{b} \dot{\theta} (d_{1} \sin\theta + d_{2} \cos\theta) \end{bmatrix}$$

The International Conference of Materials and Engineering Technology

Şekil 2.4. Sistemin Simulink Modeli

443



UWE Bristol

ليفة

Şekil 2.5. Simulasyon ortamında hareketleri gözlemlenen çok yönlü araç


Şekil 2.6. Sistemin dinamiğinin matematiksel modelinin simulink blok diyagramı - 1



يفة

Şekil 2.7. Sistemin dinamiğinin matematiksel modelinin simulink blok diyagramı - 2



Şekil 2.8. Sistemin kinematik modelinin simulink blok diyagramı



Şekil 2.9. Solidworks'te çizilen CAD modelin simmechanics'teki blok diyagram hali



The International Conference of Materials and Engineering Technology

Şekil 2.10. Sistemin tekerlek dönüş yönleri ve hareket yönü ile alakalı gösterim. [14]

Sisteme girilen parametreler ile sistem 10 farklı hareketi simulasyon ortamında tamamlamıştır. Mobil aracın benzetim modeli MATLAB/ Simulink ve Simmechanics blokları kullanılarak oluşturulmuştur. Mobil araç üç boyutlu bilgisayar-destekli tasarım (SOLIDWORKS) ortamında şasi ve buna bağlı olan dört çok yönlü tekerlek olarak modellenmiş ve oluşturulan bu model Simulink ortamına aktarılmıştır.

SimMechanics, mekanik sistemlerin modellenmesini sağlayan bir simülasyon paketidir. Simulink ortamında çalışan bu paket, geniş kapsamlı fonksiyon kitaplığıyla Simulink ortamını çoklu gövdeli mekanik modelleme olanağıyla genişletmektedir. Yaratılan modellerin otomatik olarak 3 boyutlu şekli görülebilmekte ve benzetim sırasında hareketler canlı olarak izlenebilmektedir.

#### Sonuçlar

UWE

Yapılan deneysel ve teorik çalışmalar sonucunda çok ciddi sonuçlar elde edilmiştir. Bu çalışmalar sonucunda çok yönlü tekerlekli mobil araç yön kontrolünde makara yüzeyindeki polimerin özellikleri, tekerleğin ve makaraların tasarımı, çalıştığı zeminin özellikleri, makara üzerindeki toplam kuvvetler ve tekerlek tork/devir değerlerinin önemli rol aldığı tespit edilmiştir. Aracın XY düzlemindeki 10 farklı temel hareketi tekerleklere farklı yön ve yükseklikte hızlar verilerek incelenmiştir. Yüksek manevra kabiliyetinin olabilmesi için dönüş hareketleri anında tekerlek ile zemin arasında meydana gelen sürtünmelerden uygun olarak etkilenmesi istenilmiştir. Yapılan tüm deneysel çalışmaların sonuçları kayıt edilmiş, sistemin hataları ve bu hatalar karşısında cevapları yorumlanmıştır. Aracın verimli çalıştığı durumlar ve sürtünme kuvvetinin fazla olduğu durumlar saptanmıştır. En fazla güç sarfiyatı olduğu yerde dönerken yaklaşık 4,5 [kW/h] ve bunu sırasıyla 4 [kW/h] ile ileri-geri hareketi, 3,7 [kW/h] ile yan kayma hareketi ve 3,1 [kW/h] ile çapraz hareketlerin takip ettiği görülmüştür. Deneyde kullanılan araç ileri-düz ve yan kayma hareket halinde maksimum 12 [km/h] hıza ulaşabilmiş, çapraz hareketler esnasında ise bu hız 6 [km/h] 'a düşmüştür. Ayrıca olduğu yerde dönme hareketini ise toplam 4 saniyede tamamlayarak 360° dönüşü sağlamıştır.



# Teşekkür

Bu çalışmayı Teydeb 1507 Programı kapsamında destekleyen Bilim, Sanayi ve Teknoloji Bakanlığına ve proje ortağı olan Sekizli Makina ve Vinç Sanayi Ticaret. A.Ş.'ye teşekkür ederiz. Ayrıca bu çalışmanın gerçekleştirilmesinde değerli yardımlarını esirgemeyen Okta Lift Makine ve Mühendislik çalışanları Mehmet Emin ÖRS, Adem DİLBAZ, Serhat BENLİ ve Burak YEŞİLGAZEL'e teşekkürü bir borç biliriz. Bu makale Mahmut ÇİMEN'in yüksek lisans tezinden türetilmiştir.

The International Conference of Materials and Engineering Technology

### Kaynaklar:

- 1. Shimada, A, Yajima, S, Viboonchaicheep, P, Samura, K, Mecanum-wheel vehicle systems based on position corrective control. Icon Proceedings, pp.2077-2082, **2005**.
- 2. Wang Yizhi and Chang Degong, "Motion Performance Analysis and Layout Selection for Motion System with Four Mecanum Wheels", JOURNAL OF MECHANICAL ENGINEERING, May. **2009**, vol.45(5), pp. 307-310, 316.
- Plumpton, J.J., Hayes, M.J.D., Langlois, R.G., Burlton, B.V.: Atlas Motion Platform Mecanum Wheel Jacobian in the Velocity and Static Force Domains. No. 13-CSME-192, E.I.C. Accession 3650 (September 2013)
- 4. M. de Villiers and Prof. G. Bright,"DevelopmentOf A Control Model For A Four Wheel Mecanum Vehicle" 25th International Conference of CADICAM, Robotics & Factories of the Future Conference, July **2010**, Pretoria, South Africa
- 5. Lih-Chang Lin and Hao-Yin Shih, ''Modeling and Adaptive Control of an Omni-Mecanum-Wheeled Robot'' Intelligent Control and Automation, **2013**, 4, 166-179
- 6. S. Bruins, "Mecanum Wheel," https://grabcad.com/library/mecanum-wheel, accessed **2014**-12-13.
- 7. Lippit, T.C., Jones, W.C., (1998), "OmniBot Mobile Base", KSC Research and Technology Report, NASA, USA, **1998**.
- 8. Olaf Diegel, Aparna Badve, Glen Bright "Improved Mecanum wheel design for Omnidirectional Robots" Australian Conference on Robotics and Automation Auckland, 27-29 November **2002**
- 9. Purwin O. ve D'Andrea R., Trajectory Generation and Control for Four Wheeled Omnidirectional Vehicles, Robotics and Autonomous Systems 54, 13–22, **2006**.
- Han K.-L., Choi O.-K., Kim J., Kim H. ve Lee J. S., Design and Control of Mobile Robot with Mecanum Wheel, ICROS-SICE International Joint Conference, Fukuoka International Congress Center, Japan, 2932-2937, 2009.
- 11. Badve AA. All Terrain Omni-Directional Autonomous Mobile Robot. Master's Thesis, Massey University, Auckland, New Zealand **2003**
- 12. Tlale, Nkgatho, and Mark de Villiers. "Kinematics and dynamics modelling of a mecanum wheeled mobile platform." *Mechatronics and Machine Vision in Practice, 2008. M2VIP 2008. 15th International Conference on.* IEEE, **2008**.
- 13. Lin, Lih-Chang, and Hao-Yin Shih. "Modeling and adaptive control of an omni-mecanum-wheeled robot." *Intelligent Control and Automation* 4.02 (**2013**): 166.
- Doroftei, I., Grosu, V., Spinu, V., Omnidirectional Mobile Robot Design and Implementation", Bioinspiration and Robotics: Walking and climbing Robots, Book edited by: Maki K. Habib, ISBN 978-3- 902613-15-8, pp. 544, I-Tech, Vienna, Austria, EU, September 2007.

# INVESTIGATION OF YARN PROPERTIES PRODUCED FROM 100% RECYCLED POLYESTER (r-PET) BY DIFFERENT SPINNING SYSTEMS

The International Conference

aterials and Engineering Technology

# ESİN SARIOĞLU<sup>1</sup>, DENİZ VURUŞKAN<sup>1</sup>, OSMAN YAYLA<sup>2</sup>, EYÜP ALİ SATIL<sup>2</sup>, EBRU ÇELİKTEN<sup>2</sup>

<sup>1</sup>Gaziantep University, Faculty of Fine Arts, Department of Fashion and Textile Design, Gaziantep, TURKEY. <sup>2</sup>Selçuk İplik Industry and Trade Co. Inc. Research and Development Center, Gaziantep, TURKEY.

#### Abstract

In recent years, the amount of waste is increasing with the increase of production and consumption in many areas of the world (textile, plastic, automotive, health, etc.). Reducing the amount of waste can be achieved by adding waste materials to the product cycle by re-evaluating them. Recycling of PET bottle wastes provides energy and cost-saving, protection of natural resources and diminish the amount of waste. Worldwide, researches are being carried out in many different fields within the scope of the recycling. In textile industry, using recycled polyester (r-PET) fiber obtained from recycled PET bottle wastes is one of the best examples. In light of the research about textiles with r-PET fiber, sustainable and eco-friendly products can be produced. On the other hand, the spinning of the r-PET fiber is difficult because of the variable fiber quality. So, the recycling procedure of PET bottle should be precise to obtain optimum fiber quality. In spite of the variable fiber properties, the spread of the use of r-PET fiber in the textile industry will make great contributions to waste management. In this study, the spin ability of r-PET fibers with different spinning systems to produce 100% r-PET yarns was investigated. Therefore, 100% r-PET yarns were produced with four different spinning systems (ring, compact, rotor (open-end) and vortex) at their own optimum spinning parameters. In addition, 100% virgin polyester (v-PET) yarns were also produced to compare the quality difference of r-PET and v-PET varns. Yarn properties such as tenacity, elongation, unevenness (CVm %) and hairiness were determined and results were evaluated statistically.

Key Words: Recycled polyester, PET bottle, yarn quality, yarn spinning systems.

#### **1. Introduction**

Worldwide, the increase in the population and consumption also cause an increase in production. Therefore a circle of production and consumption that is difficult to control has been formed in the world. In textile industry, consumption of fibers has reached to 100 million tons in the last decade. Polyester (PET) is one of the most used synthetic fiber in textile.

Polyester polymer is mostly used material for packaging products (e.g. food, soft drinks, cosmetics, oil etc.) because of its strength, thermal stability, transparency and lightweight. In average, it takes 35-45 years and even more for PET bottles to degrade in soil. Instead of storing or burning, these PET bottles can be recycled to raw materials (r-PET) which can also be used in textile industry [1,2]. In recent years, recycling in textile industry began to gain more importance especially polyester polymers [3]. Worldwide, r-PET that are produced from recycling materials (generally from plastic bottles) have been expanded [4].

There are two main recycling process types that can be applied to PET bottles, respectively; mechanical and chemical recycling processes. Mechanical recycling process consists of contamination, sorting, washing, drying and melt-processing steps [5]. Chemical recycling process is made according to hydrolysis, methanolysis, glycolysis, ammonolysis, and aminolysis processes [5].

The International Conference of Materials and Engineering Technology

Within the scope of this study, 100% r-PET yarns were produced by different spinning systems (ring, compact, open-end and vortex) with at their own optimum spinning parameters. In addition, 100% virgin polyester (v-PET) yarns were also produced to compare the quality difference of r-PET and v-PET yarns. Yarn properties such as tensile strength, elongation, unevenness (CVm %) and hairiness were determined and results were evaluated statistically.

#### 2. Materials and Methods

In this study, all yarn samples were produced with Ne 30/1 yarn number. Fiber properties are shown in Table 1. Yarn production parameters are given Table 2.

Table 1. Physical properties of fibers.						
Fiber Type	Fineness (dtex)	Length (mm)	Elongation (%)	Strength (cN/tex)	Density (g/cm <sup>3</sup> )	
Virgin Polyester (v- PET)	1.2-1.3	32	32	50.3	1.33 - 1.38	
Recycled Polyester (r-PET)	1.2	38	22	60	1.38 - 1.41	

Table 2. Production parameter of yarns.					
	Ring	Compact	<b>Open-End</b>	Vortex	
Delivery Speed (m/min)	20	23.3	117	350-400	
Total Draft	45	33.3	240	174	
Sliver linear density (ktex)	3.68	3.80	3.59	3.47	
Spindle Speed (rpm)	16.000	18.000	-	-	
Ring Diameter (mm)	40	40	-	-	
Twist (T/m)	780	760	780	-	
Rotor Speed (rpm)	-	-	92.000	-	
Rotor Diameter (mm)	-	-	33	-	
Opener Speed (rpm)	-	-	9000	-	
Air Pressure (MPa)	-	-	-	0.55	

All samples were conditioned at  $20\pm2^{\circ}$ C and  $65\pm4$  relative humidity. List of the applied tests and the related standards are given in Table 3.

Table 3. List of the applied tests and the related standards				
Test	Standard	Tester		
Breaking Strength and Elongation	TS EN ISO 2062	Uster Tensojet		
Unevenness and Imperfections	TS 2394 ISO 2649	Uster Tester 5		
Hairiness	TS 12863	Uster Tester 5		

C .1

For statistical analysis, ANOVA (analysis of variance) was performed at 0.05 significance level in order to compare if there was statistically significant effects of the parameters as yarn spinning types (ring, compact, rotor, and vortex) and raw materials (v-PET, r-PET) on yarn properties (tensile strength, elongation, unevenness, IPI and hairiness). Student Newman Keuls multivariate

451



The International

#### 3. Results and Discussion

#### 3.1. Tensile Strength and Elongation

In Figure 1, it is obviously seen that 100% compact v-PET yarns have the highest tensile strength values. The probable reason of this, eliminating the protruding fibers end form the yarn body as an advantage of compact spinning system. Lower tensile strength values were obtained in openend spinning system because of internal twist and less fiber parallelism. When the fiber type is regarded, it is seen that the strength of r-PET yarn is lower than that of v-PET yarn.



Figure 1. Tensile Strength test result of yarn samples

When elongation results are examined v-PET samples have lower yarn elongation values than r-PET samples (Figure 2).

Technology



The International

ials and Engineering Technology

Figure 2. Elongation test result of yarn samples

# **3.2.** Unevenness

When spinning systems are compared, lower CVm values were obtained by the vortex spinning system among all spinning systems (Figure 3). It should be considered that the yarn produced from r-PET fiber have slightly higher CVm values than that of v-PET fibers. This situation can be explained by the deterioration of structure of r-PET fibers due to recycling process. On the other hand, it should be considered that vortex spinning system can compensate this failure.



Figure 3. CVm% test result of yarn samples

# **3.3. Imperfection Index**

Imperfection index refers to the cumulative sum of the number of thin places (-50%/km), thick places (+50%/km) and neps (+200%/km) of yarn. As shown in Figure 4, IPI values of v-PET and



r-PET yarns have higher in the open-end spinning system than the other systems. The reason of this, open-end spinning system elements cause increase of IPI values especially v-PET yarns.



Figure 4. IPI test result of yarn samples

# 3.4. Hairiness

As seen from figure 5, both 100% v-PET and 100% r-PET yarns have lower hairiness values in vortex spinning system (Figure 5). Since, in vortex spinning system there is a high number of wrapping fibers around the yarn. This situation provide lower number of protruding fiber ends from yarn body. In contrary, among the spinning systems used in this study, ring spinning system is more prone to produce hairy yarns than the other systems owing to spinning principle.



Figure 5. Hairiness test result of yarn samples

Table 4 illustrates the result of variance analysis. Generally, statistical analysis indicates that, yarn production method have statistically influenced on all yarn properties investigated such as tensile strength, elongation, unevenness, IPI and hairiness (p<0.05). On the other hand, fiber type has statistically significant effect on tensile strength, elongation, IPI and hairiness except unevenness property. It can be said that the interaction of yarn production method and fiber type variables (A\*B) have also statistically significant effect on all response variables except unevenness property. According to the multivariate test results for tensile strength variable, it can be said that the highest tensile strength was obtained from both vortex and compact spun yarns and there is no difference between these two yarn types in terms of tensile strength (Table 5).

The International Conference of Materials and Engineering Technology

Source/ Dependent Variable	Yarn Production Method (A)	Fiber Type (B)	A*B
Tenacity (kgF*Nm)	0.000*	0.000*	0.000*
Elongation (%)	0.004*	0.000*	0.006*
Unevenness (CVm %)	0.000*	0.314	0.141
Imperfection Index (IPI)	0.000*	0.002*	0.000*
Hairiness (Uster H)	0.000*	0.000*	0.001*

Table 4. Analysis of variance (ANOVA) result
--

\*: statistically significant at 0.05 level.

Vom	SNK Ranges							
Production Method	Tensile Strength (kgf*Nm)	Elongation (%)	Unevenness (CVm%)	Imperfection Index (IPI)	Hairiness (Uster H)			
Open-end	17.4233 <b>a</b>	10.9333 <b>a</b>	14.3550 <b>c</b>	332.0833 <b>c</b>	5.3517 <b>c</b>			
Ring	22.9388 <b>b</b>	11.5200 <b>a</b>	15.8463 <b>d</b>	168.1250 <b>b</b>	6.3900 <b>d</b>			
Vortex	26.4517 <b>c</b>	10.5200 <b>b</b>	12.3217 <b>a</b>	15.4167 <b>a</b>	4.0433 <b>a</b>			
Compact	28.1800 <b>c</b>	12.5400 <b>b</b>	12.9912 <b>b</b>	24.6875 <b>a</b>	5.1363 <b>b</b>			

 Table 5. SNK analysis test results

In addition, it was found that there is no difference between open-end and ring yarn type when the multivariate analysis for elongation is taken into consideration. Similar result was obtained for elongation values of vortex and compact yarn types. When the unevenness properties of yarn types are compared, we can put in order from lowest to highest unevenness values at vortex<compact<ol>
 we can put in order from lowest to highest unevenness values at open-end and vortex yarn types, respectively. Also, it was found that there is no difference between IPI values of the vortex and compact yarn type. It is seen that hairiness values in terms of yarn types vary on vortex<compact<ol>
 open-end<ring.</li>

#### 4. Conclusions

In this study, v-PET and r-PET yarn properties such as tensile strength, elongation, IPI and hairiness in different spinning systems were investigated. According to the test results,

- It was found that produced yarns in vortex spinning systems have lower unevenness and hairiness values than the other spinning systems.
- Yarn produced on compact spinning system has higher tensile strength and elongation than the other spinning systems.

Yarn produced on open-end spinning has higher IPI values than the other spinning systems.

The International Conference of Materials and Engineering Technology

- If the fiber type is examined, v-PET yarns have higher tensile strength and lower hairiness values in comparison to r-PET yarns. On the other hand, r-PET yarns have higher elongation and lower IPI values than v-PET yarns.

#### Acknowledgement

The authors would like thank to Selçuk İplik Industry and Trade Co. Inc. Research and Development Center contribution to yarn samples productions and performing yarn quality tests.

#### References

- 1. Komly, CE, Azzaro-Pantel, C, Hubert, A, Pibouleau, L and Archambault, V, Multiobjective waste management optimization strategy coupling life cycle assessment and genetic algorithms: Application to PET bottles, Resources, Conservation and Recycling. 2012, 69:66-81.
- 2. Telli, A and Özdil, N, Properties of the yarns produced from r-pet fibers and their blends, Tekstil ve Konfeksiyon. 2013, 23(1):3-10.
- 3. Necef, ÖK, Seventekin, N and Pamuk, M, A study on recycling the fabric scraps in apparel manufacturing industry, Tekstil ve Konfeksiyon. 2013, 23(3):286-289.
- 4. Dutt, K and Soni, RKA, Review on synthesis of value added products from polyethylene terephthalate (PET) waste, Polymer Science. 2013, 55(7-8):430-452.
- 5. Khoonkari, M, Haghighi, HA, Sefidbakht, Y, Shekoohi, K, and Ghaderian, A, Chemical recycling of PET wastes with different catalysts, International Journal of Polymer Science. 2015, *ID*:124524, 11 pages.

# NADİR TOPRAK METAL HEKZABORÜRLERLE KOMPOZİT OLUŞUMU

The International Conference

aterials and Engineering Technology

# CENGİZ BOZADA<sup>\*1</sup>, MİKAİL ASLAN<sup>2</sup>, HALİL İBRAHİM KURT<sup>2</sup>, ZİHNİ ÖZTÜRK<sup>1</sup>

<sup>1</sup> Gaziantep Üniversitesi, Mühendislik Fakültesi, Fizik Mühendisliği, Gaziantep, TÜRKİYE
<sup>2</sup> Gaziantep Üniversitesi, Mühendislik Fakültesi, Metalürji ve Malzeme Mühendisliği, Gaziantep, TÜRKİYE

# ÖZET

Nadir Toprak Metal hekzaborürler (ReB<sub>6</sub>) ile X<sup>IV</sup>B<sub>2</sub> (X<sup>IV</sup>–Ti, Zr, Rf ve Hf), SiC, MgO-Karbon Nanotüp, Alümina vb seramiklerle kompositlenmesiyle yüksek sıcaklıkta güçlü mukavemet ve sürtünme direnci göstermesi onları yapısal malzemeler olarak çekici kılmıştır. Kompozitlerin sertliği, yüzey morfolojisi, optik, deşarj özellikleri, kırılma dayanaklığı, yoğunlaşma davranışı, mikroyapı ve mekanik özellikleri incelenmiştir. Bu özelliklerin incelenmesi için TEM, XRD, SEM, Raman, XPS analizleri yapılmıştır. Bu kompozit yapıları üretmek için de yönlendirilmiş katılaşma işlemi ve yüzer bölge yöntemi, vakum sıcak press sinterleme yöntemleri genellikle tercih edilmektedir. LaB<sub>6</sub>-TiB<sub>2</sub> kompozitinin yüksek sıcaklıkta TiB<sub>2</sub> gerilme sertleşmesini ortaya çıkardığını buldular. SiC seramiği çok yüksek sıcaklıklarda oluşan çatlak çökmesi ve dallanması kompozit malzemenin sertleşmesinde önemli rol oynadığı ortaya çıkardılar. ReB<sub>6</sub> kaplı CNT kompozitlerde elektrikli alanın azaldığını, akım yoğunluğunun düştüğünü ve eşik alanında artma olduğunu gözlediler. LaB<sub>6</sub> katkılı çift koruyucu MgO katmanın geçirgenliğinin, LaB<sub>6</sub> katkılama konsantrasyonundaki artışla yavaş yavaş azaldığı açıkça gösterdiler. Biz bu çalışmada, REB<sub>6</sub>ile nano veya mikro seramik malzemelerin kompozit oluşturmasıyla elde edilen özellikler ile ilgili güncel bilgiler üzerine odaklandık.

Anahtar Kelimeler: Nadir Toprak Hekzaborürler, Seramik Kompozitler, İleri Malzemeler, Nanomalzemeler, Nanoparçacıklar

#### 1. Giriş

Nano veya mikro seramik malzemeler önemli özellikler gösterdiğinden nadir toprak metal hekzaborürlerle (REB<sub>6</sub>) kompozit oluşturması bu malzemelerin istenilen özeliklerin oluşturulmasında önemli rol oynamıştır. Çoğunlukla REB<sub>6</sub>'lar X<sup>IV</sup>B<sub>2</sub> (X<sup>IV</sup>–Ti, Zr, Rf ve Hf), SiC, MgO, CNT ve Al<sub>2</sub>O<sub>3</sub> gibi malzemelerle kompozit oluşturmaktadır. X<sup>IV</sup>B<sub>2</sub> (X<sup>IV</sup>–Ti, Zr, Rf ve Hf) seramikler malzemenin sertliğini, bükülme mukavemetini, eğilme dayanımını, yüksek sıcaklıkta gerilme sertleşmesini, SiC seramikleri yoğunlaşma davranışını, korozyon direncini, yüksek termal şok dayanımını, sertlik özelliklerini, MgO seramikleri ateşe dayanıklık, yüksek termal iletkenlik, düşük elektriksel iletkenlik özelliklerini, CNT optiksel ve mekaniksel özelliklerini ve Al<sub>2</sub>O<sub>3</sub> seramikleri yüksek aşınma ve korozyon direnci, düşük yoğunluk gibi özeliklerinin iyileşmesinde katkıları bulunmaktadır.

Bu kompozitleri üretilmesi için birçok yöntem vardır. Örneğin, LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitlerini vakum sıcak press sinterleme tekniği ile üretilmektedir ve tozların pota serbest bölge erimesine dayalı yüzer bölge metodu ile hazırlanmaktadır. LaB<sub>6</sub>-TiB<sub>2</sub> kompozitlerini pota serbest bölge erimesine

dayalı yüzer bölge metodu ile üretilmekte ve elektrik arkındaki argon akışında eriterek elde edilmektedir.CeB<sub>6</sub>-TiSi<sub>2</sub> kompozitleri reaktif spark (kıvılcım) plazma (SPS) ile sinterleyerek üretilmektedir.LaB<sub>6</sub>-SiC kompozitleri SPS tekniği ile elde edilmektedir. LaB<sub>6</sub>-MgO kompozitleri ekran baskısı tekniği ve magnetron püskürtme yöntemini ile üretilmektedir. LaB<sub>6</sub>-CNT ile CeB<sub>6</sub>-CNT kompozitleri fiziksel (PVD) ve kimyasal buhar biriktirme (CVD) ile üretilmektedir. LaB<sub>6</sub>-Al<sub>2</sub>O<sub>3</sub> kompozitleri yüksek enerjili bilyeli öğütme, tavlama ve liç işlemlerinden oluşan bir yöntem ile sentezlenmektedir.

The International Conference of Materials and Engineering Technology

Biz bu çalışmada, nadir toprak metal hekzaborürleriyle nano ve mikro seramik malzemelerden oluşan kompozitlerin faz stabilitesi, mikroyapısı, mekanik performansı ve emisyon özellikleri üzerinde durduk.

# 2. Tartışma ve Sonuç

#### 2.1 REB<sub>6</sub>-X<sup>IV</sup>B<sub>2</sub>Kompozitler

Sert ve kırılgan malzemeler olan REB<sub>6</sub>-X<sup>IV</sup>B<sub>2</sub> kompozitler, düşük tokluğa sahiptirler, termal şoklara karşı dayanıksızdırlar ve çok düşük süneklik gösterirler[1].REB<sub>6</sub>-X<sup>IV</sup>B<sub>2</sub> kompozit malzemelerin özelliklerini belirleyen dört temel faktör ardır. Bunlar: Matris malzemesinin özellikleri, takviye malzemesinin özellikler, ara yüzey özellikleri ve mikro yapı özellikleridir [2].Bu kompozitlerden yüksek mukavemet artışı sürekli fiber (elyaf) kullanımı ile sağlanmakta ve elde edilen kompozit çok üstün özelliklere sahip olmaktadır. Fiber miktarı arttıkça kompozit malzemenin mukavemeti de artmaktadır[3,4].

Yüksek erime noktasından dolayı, ReB<sub>6</sub>-X<sup>IV</sup>B<sub>2</sub> yüksek sıcaklıklarda iyi bir yapısal malzeme olarak da kullanılabilir. Mevcut oksit yönlü katılaşmış kompozitler, bu alanda çok dikkat çekmiştir, çünkü yüksek sıcaklıkta (>1200°C) mükemmel mukavemet ve sürtünme direnci göstermesi, onları yapısal malzemeler olarak çekici kılmıştır[5,6]. Ancak bu malzeme sınıfında kırılma dayanıklılığı düşüktür, çünkü iki faz arasındaki arayüzler tipik olarak güçlü bağlamayı destekleyen ve arayüzün yapışmasını önleyen yönsel katılaşma prosesi sırasında düşük enerjili bağlanma yönelimi ilişkilerini benimsemektedir[7].REB<sub>6</sub>-X<sup>IV</sup>B<sub>2</sub>'nin (X<sup>IV</sup>-Ti, Zr,Rf ve Hf) yönsel olarak kristalize olmuş kompozitleri bu alanda en çok araştırılan seramik malzemelerdir[8,9,10]. Yapılan çalışmalar, en iyi mekanik özelliklerLaB<sub>6</sub>– ZrB<sub>2</sub> yönsel olarak katılaşmış kompozitlerden elde edildi. Aynı zamanda, refrakter boritler eriyikteki çoğu metal ile etkilesime girdiğinden, böylece pota kalıp malzemelerinin kirlenme olmadan eriyik içermesi uygun bulunmadığından dolayı, yüzer bölge yönteminin bu kompozitlerin sentezi için bir avantaja sahip olduğu görülmektedir[11,12].Yüksek sıcaklık eğilme dayanımı, yapısal malzemelerin mekanik özelliklerini değerlendirmek için kullanılan ana parametrelerden biridir. Yapılan çalışmalarda, yönsel olarak katılaşan LaB6-TiB2yüksek sıcaklık eğilme dayanımının, LaB6 matris fazının kristallografik oryantasyonuna, matris ve fiberlerin plastikliğine bağlı olduğunu göstermiştir. Özellikle, 1000–1600 °C sıcaklık aralığında, LaB<sub>6</sub> matris fazı (100) yönelimi ile yetiştirilen bileşik için en yüksek kuvvet (470 MPa) ve (110) yönelimi ile yetiştirilen bileşik için en düşük kuvvet (350 MPa) gözlenmiştir[13,14].

# 2.1.1 LaB6-ZrB2Kompozitler

 $ZrB_2$  gri renkte olup, çok yüksek bir erime sıcaklığına (3245°C) ve hekzagonal kristal yapıya sahiptir.  $ZrB_2$ ; karbon, zirkonyum ve bor oksitin elektrik ark fırınında reaksiyona sokulması ile üretilebilmektedir.  $ZrB_2$ ,oksidasyon direnci, yüksek sertlik ve termal şok direncine sahiptir. Eriyik metal kabı, yarı iletkenlerde difüzyon bariyeri, nükleer reaktör çekirdeklerinde ise tutuşma emici olarak kullanılmaktadır[15]. LaB<sub>6</sub> ve  $ZrB_2$  yüksek erime sıcaklığı ve iyi elektrik iletkenliği gösterir. Buna ek olarak, borürler, düşük iş fonksiyonlarına sahiptir. Mikroskoplarda kullanılan elektron ışının termo-iyonik yayıcısı olarak kullanılırlar. Ancak zayıf termal şok direnci ve oksijen zehirlenmeleri uygulamaları büyük ölçüde sınırlar.LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitlerinin, ısı şokuna ve zehirlenmeye karşı direnci saf boratlardan daha belirgin şekilde arttar. Özellikle, LaB<sub>6</sub>-ZrB<sub>2</sub> alaşımının bileşenleri, saf LaB<sub>6</sub>'dan 4-5 kat daha yüksek bir akım yoğunluğu ve minimum çalışma fonksiyonu gösterir[16]. Paderno ve diğerleri LaB<sub>6</sub>'daki bor-boron (BB) mesafesinin ZrB<sub>2</sub> eklenerek modifiye edilebildiğini bildirdi. Hekzaborürdeki B-B mesafesi, di-borür içindeki mesafeye yakınsa, yarı uyumlu arayüz, LaB<sub>6</sub>-ZrB<sub>2</sub> kompozit arasında oluşturulabileceğini öne sürdü[9].

The International Conference

aterials and Engineering Technology

Farklı bir çalışmada Chen ve diğerleri,LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitlerini LaB<sub>6</sub> ve ZrB<sub>2</sub> tozlarından hazırladılar, daha sonra çubuk örnekleri oluşturmak için elektrik ark ile preslediler ve erittiler. Yönlü katılaşma işlemini, elektron ışın ve yüzer bölge eritme fırını ile birlikte 2900 °C'de ısıtılan bir vakumda gerçekleştirdiler[10].Ayrı bir çalışmada ise Wang ve diğerleri,LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitlerinin mikroyapılarını enerji ayrımlı X-ışını spektrometresi(EDS) ile donatılmış transmisyon elektron mikroskopları (TEM, 100CXII veya HRTEM, TECNAI F-30, Philips, Holland) ile karakterize ettiler.LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitler, bir yön boyunca büyütülerek iyi yönlendirilmiş ZrB<sub>2</sub>fiberi ve yönlü katılaşmadan sonra iyi dağılmış LaB<sub>6</sub> matrisi göstermeyi başardılar.LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitinin enine kesitinin ve boyuna kesitinin TEM mikrografileri Şekil I'de gösterilmiştir. Enine kesit, ZrB<sub>2</sub> fiberlerinin tesir kesitte neredeyse yuvarlak olduğu ve LaB<sub>6</sub> matrisine kıyasla daha koyu kontrast gösterdiği görülür[17].



**1.**(a) Yönsel katılaşmış LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitinin boyuna kesit ve (b) enlemesine kesit TEM mikrografileri Adopted from[17]Copyright© 2019 by Trans Tech Publications Ltd.

Farklı bir çalışmada ise Min ve diğerleri,farklı  $ZrB_2$  içeriğine sahip polikristalize  $LaB_6$ - $ZrB_2$  kompozitleri vakum sıcak press sinterleme tekniği ile ürettiler. $ZrB_2$  içeriğinin mikroyapı ve mekanik özellikler üzerindeki etkilerini incelediler. $LaB_6$ - $ZrB_2$  kompozit için sertlik, eğilme dayanımı ve kırılma tokluğu sırasıyla 93.0 (HRA), 330.0MPa ve  $3.70MPa \cdot m^{1/2}$  olarak buldular. Elde edilen sonuçlara göre,  $LaB_6$ - $ZrB_2$  polikristalinin sertliği ve bükülme mukavemetinin  $ZrB_2$  içeriğinin artmasıyla arttığını göstermiştir; ancak kırılma tokluğu ilk önce ağırlıkça %21  $ZrB_2$  içeriğine karşılık gelen tepe değerine ulaşmaktadır. Mikroyapı gözleminde  $ZrB_2$  ilavesi nedeniyle bir yoğunlaşma tespit edildi. Kırılma morfolojisi,  $ZrB_2$  içeriğinin arttırılmasıyla tanecikler arasında kırılma eğiliminin ortaya çıktığını göstermiştir. $LaB_6$  matrisinde  $ZrB_2$  kullanıldığında, çatlaklar iki şekilde uzamıştır:  $LaB_6$  matrisinde transgranüler kırık ve  $ZrB_2$  alanında intergranüler ayrıntılı morfolojiyi göstermiştir [18].



**Şekil 2.** (a) LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitinin ve (b)LaB<sub>6</sub>'nıntek kenarı çentikli çekme ve eğme testleri (SENB) kırık morfolojisi Adopted from[18]© 2019 by Trans Tech Publications Ltd.

Buna benzer bir çalışmada ise Bogomol ve diğerleri, yönlendirilmiş bir LaB<sub>6</sub>-ZrB<sub>2</sub>, sıkıştırılmış tozların pota serbest bölge erimesine dayalı yüzer bölge metodu ile hazırlamıştır. İlk malzemeler olarak ZrB<sub>2</sub> ve LaB<sub>6</sub> tozları kullanıldı. Kompozitin eğilme mukavemeti 25–1600°C sıcaklık aralığında değerlendirilmiş ve 1600 °C'de 950 MPa' la ulaşmıştır. Kırılma dayanıklılığı, SEM ve TEM kullanılarak, farklı koşullar altında sertleştirme mekanizmaları incelenmiştir. LaB<sub>6</sub>-ZrB<sub>2</sub> 25–1200 °C'deki gücünün temelde çatlak sapması, köprü sertleştirme mekanizmaları, ZrB<sub>2</sub> fazının artan plastikliği, 1200–1600 °C'de, matris ve fiberlerin artan plastikliği ile ilişkili olduğunu tahmin ettiler. Fiberlerin çıkık yapısı analiz edilerek, tek kristalli ZrB<sub>2</sub>'de yüksek sıcaklık deformasyonu sırasında zorlanma oluşumunu ortaya çıkardılar. Yön olarak katılaşmış LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitin cilalanmış bir yüzeyinin SEM-EDS analizi, ZrB<sub>2</sub> fiberiyle güçlendirilmiş bir LaB<sub>6</sub> matrisinden oluştuğunu ortaya çıkardı (Şekil 3 (a)).Temsili bir EDS spektrumu (Şekil 3 (b)), yüksek enerjilerde B piklerini ve Zr ve La' dan çıkan güçlü bir sinyali gösterir[19].



**Şekil 3.**Yönlendirilmiş LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitinin (a) Haritalaması ve (b)EDS spektrumları Adopted from[19]Copyright © 2011 Elsevier B.V.

Gao ve diğerleri,LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitlerin oksidasyon davranışını 600-1300 °C sıcaklık aralığında incelediler. LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitleri sıcak presleme ile sinterlediler. Malzeme ağırlığının oksidasyon sıcaklığı arttıkça ve bekleme süresinin uzadıkça arttığını göstermişlerdir.LaB<sub>6</sub>-ZrB<sub>2</sub> kompozitlerinin oksidasyon direncinin, monolitik LaB<sub>6</sub>'dan daha yüksek olduğunu bulmuştur[20].



TiB<sub>2</sub>, yüksek erime sıcaklığına, yüksek aşınma dayanımına, yüksek sertliğe, yüksek mukavemete ve kimyasallara karşı yüksek dirence sahiptir. Sinterlemeye karşı dirençlidir ve çoğunlukla izostatik presleme veya sıcak press ile yoğunlaştırılır.TiB<sub>2</sub>'nin basınçsız olarak sinterlenmesi ile yüksek saflıkta elde edilebilmesi mümkün ancak sıvı formda karbon, krom ve demir gibi yardımcılara ihtiyaç vardır[21]. Yüksek sertliğe sahip olmasından ötürü çoğu endüstri alanında kullanılmasına rağmen şekil vermekteki zorlukları işlenebilirliğini zorlaştırdığı için TiB<sub>2</sub>'yi tek başına kullanılmasından ziyade kompozit olarak kullanılmaya itmektedir[22].

The International Conference

aterials and Engineering Technology

Bogomol ve diğerleri, yönlendirilmiş katılaşmış bir LaB<sub>6</sub>-TiB<sub>2</sub> kompoziti, sıkıştırılmış tozların pota serbest bölge erimesine dayalı yüzer bölge metodu ile hazırladılar. İlk malzemeler olarak TiB<sub>2</sub> ve LaB<sub>6</sub> tozları kullanıldı. Erimiş LaB<sub>6</sub>-TiB<sub>2</sub> kompozitinin eğilme dayanımı, 1000–1600 °C sıcaklık aralığında değerlendirildi ve 1400 °C'de 470 MPa'ya ulaştı. Yüksek sıcaklıklarda yönsel olarak güçlendirilmiş LaB<sub>6</sub>-TiB<sub>2</sub> kompozitinin eğilme mukavemetinin esas olarak TiB<sub>2</sub>fiberlerin ve LaB<sub>6</sub> matrisinin plastikliğine bağlı olduğunu tahmin ettiler. Yer değiştirme yapısını incelediklerinde, yüksek sıcaklık deformasyonu sırasında monokristal TiB<sub>2</sub> gerilme sertleşmesini ortaya çıkardılar[13].Şekil 4(a)'da, gerilen fiberler, farklı yön değiştirme hatları bulunan bölgelere ayrılır. Böylece, yüksek sıcaklıkta deformasyon sırasında monokristal titanyum diborürün yer değiştirmeşi gözlendi. I.bölgede matris fazına yakın bir bölgede, TiB<sub>2</sub> hekzagonal (altıgen) (P6/mmm) kristallografik yapısının (0001) kayma düzlemine tekabül eden fiber eksenine yönelmiş yer değiştirme çizgileri gözlenir. III. bölgede Fiber kırığının yakınında yer değiştirme yoğunluğu 10<sup>9</sup> cm<sup>-2</sup> 'ye yükselir ve dağılmış hücresel alt yapı gözlenir. Şekil 4(b)'de, doğrudan güçlendirilmiş LaB<sub>6</sub>-TiB<sub>2</sub>kompozitindegerilmesiz TiB<sub>2</sub> lifleri görülmektedir.



**Şekil 4.**(a) Gerilmemiş TiB<sub>2</sub> fiberlerin1400 ° C'de ve (b) Gerilmiş TiB<sub>2</sub> liflerinin doğrudan takviyeli LaB<sub>6</sub>-TiB<sub>2</sub>kompozit içindeki TEM görüntüleri Adopted from[13]Copyright © 2010 Elsevier B.V.

Ayrı bir çalışmada ise Soloviova ve diğerleri, polikristal (PC) ve LaB<sub>6</sub>-TiB<sub>2</sub>, PC ve (111) tekli kristalin (MC) tohumlarında kullanılan tozları yüzen bölge eritme metoduna dayanarak elde ettiler. Yaptıkları bu çalışmanın amacı, 2370/2408 °C bir sıcaklıkta faz bileşimindeki ısıl tavlamaları, kalıntı gerilmeleri ve polikristalik LaB<sub>6</sub>-TiB<sub>2</sub> seramik kompozitlerin mekanik özelliklerini belirlemekti[23].Farklı bir çalışmada ise Nesmelov ve diğerleri, elektrik arkla eritilmiş LaB<sub>6</sub>-TiB<sub>2</sub> kompozitin mikroyapısını, sertliğini ve kırılma dayanaklığını incelediler. Yüzen bölge yönteminin aksine, ark erimenin makro ölçekte tek yönlü katılaşan alaşım elde edilmesine izin vermediğini belirlediler. Ark eritilmiş alaşımların yapısı blokların farklı yönelimi ile (kristalleşme sırasındaki farklı sıcaklık derecelerine göre) oluşabilir. Yön olarak katılaşan LaB<sub>6</sub>-TiB<sub>2</sub> alaşımını, elektrik arkındaki argon akışında eriterek elde ettiler. Kompozitlerin yapısı, LaB<sub>6</sub> matrisindeki yönsel olarak kristalize olan TiB<sub>2</sub> liflerinden (çubuklardan) oluşur[24].

The Internationa

als and Engineering Technology

## 2.1.3 CeB6-TiSi2Kompozitler

Titanyum disilisyumlar; düşük dirençlilikleri, yüksek sıcaklıklarda kararlılığı ve kimyasal uyumluluğu nedeniyle yarı iletken endüstrisinde yaygın şekilde kullanılmaktadır[25]. TiSi<sub>2</sub> yüksek frekanslı, yüksek akımla çalışan cihazlardaki uygulamalar için perspektif bir malzemedir ve Si bazlı nanoyapılar için potansiyel bir adaydır[26]. Zhou ve diğerlerinin yaptığı bir çalışmada 1500 °C'de CeH<sub>2</sub> ve bor tozu karışımını reaktif kıvılcım plazma ile sinterleyerek CeB<sub>6</sub>'yı hazırladılar[27]. Sonber ve diğerlerinin yaptığı ayrı bir çalışmada ise CeB<sub>6</sub> tozu, 1600 <sup>0</sup>C'de vakumda CeO<sub>2</sub>'nin bor karbür indirgenmesi ile sentezlediler. CeB<sub>6</sub> sinterlenmesi sırasında gelişmiş yoğunluğu ve daha düşük erime noktasından dolayı TiSi<sub>2</sub> sinter katkı maddesi olarak kullandılar. Bu çalışma, SPS ile konsolidasyon ve mekanik özelliklerin araştırılması sonuçlarını içermektedir[28].Şekil-5, monolitik CeB<sub>6</sub> ve CeB<sub>6</sub>+ %5 TiSi<sub>2</sub> numunesinin kırılma yüzeylerini göstermektedir. Kırılma modu, her iki örnekte tane sınırlarında kırılgan fazların olmadığına işaret eden baskın transgranüler olduğu görülmektedir.



**Şekil 5.** (a) monolitik CeB<sub>6</sub>'nın (b) CeB<sub>6</sub> + % 5 TiSi<sub>2</sub>'nin çatlaklı yüzeyleri Adopted from [28] Copyright © 2015 Elsevier Ltd and Techna GroupS.r.l.

#### 2.2 LaB<sub>6</sub>- SiC Kompozitler

SiC seramiklerinin sahip oldukları yüksek young modulü (441- 475 GPa), yüksek ergime sıcaklığı (2545 °C), yüksek sertlik (24,5-28,2 GPa), yüksek oksidasyon, düşük teorik yoğunluk (3,21 g/cm<sup>3</sup>), korozyon direnci, yüksek termal şok dayanımı, düşük termal genleşme katsayısı, düşük termal iletkenlik gibi özellikleri, böyle malzemelere uzay ve havacılık endüstrisi, parlatma işlemleri ve aşındırıcı takım malzemeleri, ileri teknoloji seramikleri ve zırh malzemeleri gibi pek

çok uygulama alanları bulmalarına olanak sağlamaktadır[29].SiC, monolitik MeB2'den daha yüksek mukavemet, kırılma dayanıklılığı ve oksidasyon direncine sahip olduğundan, SiC-LaB<sub>6</sub> kompoziti, SiC'nin mükemmel mekanik performansı ile LaB6'nın yüksek elektron emisyon verimliliğini bir araya getirmesi nedeniyle çekicidir[30,31]. Diğer yandan, SiC-LaB<sub>6</sub> kompozitlerin, yüksek erime sıcaklığı, güçlü kovalent bağ ve düşük öz-difüzyon katsayıları nedeniyle, hiçbir katkı maddesi olmadan geleneksel sinterleme kullanılarak yüksek yoğunluklu bileşik hazırlamak zordur[32,33]. Yang ve diğerlerinin yaptığı çalışmada SPS tekniği ile bir SiC-LaB6kompozit hazırladılar ve yoğunlaşma davranışı, mikro yapı özellikleri ve mekanik özellikleri sistematik olarak incelediler. SPS tekniği ile 1600-1880 °C sıcaklıkta 300 saniye boyunca 40 MPa'lık bir basıncında bir SiC-LaB<sub>6</sub>kompozit hazırlandı ve yoğunlaşma davranışı, mikroyapı özellikleri ve mekanik özelliklerini araştırdılar. 1840 °C' de sinterlenmiş SiC-LaB<sub>6</sub> maksimum % 98.6'lik oranda kısmi yoğunluğunu buldular. 1840 °C'de sinterlenmiş SiC-LaB6 kompozit için elde edilen maksimum Vickers sertliği ve kırılma tokluğu sırasıyla 26.5 GPa ve 7.15 MPa m $^{1/2}$  idi. Çatlak çökmesi (sapması) ve çatlak dallanması, SiC-LaB<sub>6</sub> kompozitinin sertleşmesinde önemli rol oynadığı sonucuna vardılar. Şekil-6, öğütme işleminden sonra karışım tozlarının XRD analizlerini ve 40 MPa basınçta 3h boyunca 1600-1880 °C'de sinterlenmiş SiC-LaB<sub>6</sub> kompozitini göstermektedir. SiC veya LaB<sub>6</sub> piklerinde kaymalar tespit edilemedi, bu iki bileşen arasında 1600-1880 °C'de kimyasal bir reaksiyonun meydana gelmediğini gösterdiler[34].

The International Conference of Materials and Engineering Technology



**Şekil 6.** 1600-1880 °C'de sinterlenmiş SiC-LaB<sub>6</sub> kompozitinin XRD desenleri ve 3 saat boyunca bilyalı öğütmeden sonra toz karışımları Adopted from [34]Copyright© 2017 Elsevier B.V.

# 2.3 LaB<sub>6</sub>-MgO Kompozitler

Magnezyum oksit (MgO), kristal ve beyaz renkli şeklinde bulunur[35]. MgO'nun önemli özelliği şunlardır: yüksek termal iletkenlik, düşük elektriksel iletkenlik ve ateşe dayanıklı olmasıdır. MgO malzemesi, yüksek dayanıklılığı ve iyon bombardımanına karşı iyi koruma özellikleri nedeniyle koruyucu bir tabaka olarak kullanılır. Alternatif akım plazma ekran panelinde(AC PDP), LaB<sub>6</sub>-MgO kompositleri kullanılmaktadır[36]. Deng ve diğerleri,yeni bir AC PDP koruyucu tabakası oluşturmak için ekran baskısı tekniği ile MgO tabakası üzerinde LaB<sub>6</sub> nanofilm kompositlerini hazırladılar. Saf MgO film ile karşılaştırıldığında, baskılı LaB<sub>6</sub>/MgO koruyucu katmana sahip test paneli saf MgO nanokompozit filmden daha iyi deşarj performansları göstermiştir[37]. Deng ve diğerlerinin yaptığı başka bir çalışmada ise, LaB<sub>6</sub> katkılı MgO koruyucu tabakaları üretmek için AC PDP 'nin üretim işlemleriyle uyumlu magnetron püskürtme yöntemini kullandılar ve deşarj performanslarını AC PDP test panellerinde incelediler. Yüzey morfolojisini, optik özellikleri ve deşarj özelliklerini değişken LaB<sub>6</sub> doping konsantrasyonuyla incelediler. Bu nanokompozit filminin, AC PDP'nin ateşleme voltajını azaltmada etkili olduğu gösterilmiştir. Özellikle LaB<sub>6</sub>

463



The International Conference

terials and Engineering Technology



Şekil 7.LaB<sub>6</sub> katkılı MgO / MgO koruyucu tabakaların geçirgenliği Adopted from[38]Copyright © 2014 Elsevier B.V.

#### 2.4 REB<sub>6</sub>-Karbon Nanotüpler

Karbon nanotüpler (CNT), yaklaşık 1 mm'ye varan uzunlukları ve 4~30 nm dış çapı olan ve bal peteği şekline sahip karbon yapılardır[39]. CNT'ler, çok önemli mekanik ve elektronik özelliklere sahip eşsiz nano-yapılardır. Nano boyutlu elektronik uygulamalarının yanında çeşitli uygulamaları, potansiyellerine olan ilgiyi artırmıştır[40]. CNT'ler sahip olduğu küçük boyutlarından dolayı incelenmesi zordur, bu yüzden genellikle kompozit malzemeler oluşturmak için kullanılırlar. Bu nedenle CNT'lerin işlevselliği, polimer kompozitlerinde işlenmesi ve potansiyel uygulamalarında son derece önemlidir. Genel olarak, kimyasal olarak değiştirilmiş nanotüplere dayanan kompozitler, en iyi mekanik sonuçları gösterir çünkü işlevselleştirme, hem dağılım (dispersiyon) hem de gerilme transferinde önemli bir gelişme sağlar[41].

#### 2.4.1 LaB6- CNT

LaB<sub>6</sub> 'nın düşük çalışma fonksiyonu, CNT'lerin yüksek boyut oranıyla uyarlanabilir, daha sonra gelişmiş alan emisyonuna katkıda bulunması beklenir[42]. Wei ve diğerleri, CNT-yayıcı dizilerden ziyade LaB<sub>6</sub>ile çok duvarlı tek CNT emitör ucunu değiştirmiştir. Elektrikli alanın azaldığını ve akım yoğunluğunun düştüğünü bildirdiler[43]. Kumari ve diğerleri tarafından yapılan çalışmada CNT'lerin alan emisyonu, LaB<sub>6</sub>nano parçacıklar (NP'ler) ile dikkate değer şekilde geliştirilmiştir. CNT'ler silisyum substrat üzerinde kimyasal buhar biriktirme ile büyütüldü. Fiziksel, morfolojik, elementer ve grafitik doğa değişimleri SEM, TEM, EDS ve Raman analizi ile tespit edildi.LaB<sub>6</sub>-NP'lerin dekore edilmiş CNT'lerin, elektrik alanını 3,0'tan 2,1 V / lm'ye düşürdüğünü gösterdiler[44].

LaB<sub>6</sub> kaplı çok duvarlı karbon nanotüp (MWCNT) filmleri, bozulmamış LaB<sub>6</sub> filmleri ve bozulmamış MWCNT filmlerinin saha emisyon çalışmalarından elde edilen ayrıntılı sonuçlar

464



The International Conference

terials and Engineering Technology



Şekil 8.(a) LaB<sub>6</sub> kaplı CNT filmi ve (b) bozulmamış bir MWCNT filminin SEM mikrografı Adopted from [45] Copyright © 2014 AIP Publishing LLC.

#### 2.4.2 CeB6- CNT

CeB<sub>6</sub>; yüksek erime noktası, yüksek sertlik, mükemmel termal stabilite, düşük buhar basıncı, yüksek elektriksel ve termal iletkenlik ve düşük termal genleşme katsayısı ile karakterize olan istisnai bir seramiktir[46]. Mikrodalga plazma ile geliştirilmiş kimyasal buhar biriktirme (MPECVD) ve hidrotermal sentez rotaları birleştirilerek yetiştirilen CeB<sub>6</sub> kaplı CNT filmleri üzerinde detaylı bir FE analizi Jha ve diğerleri tarafından gerçekleştirildi.CeB6 kaplı CNT filmdeki FE akım yoğunluğu, stabilite ve güçlendirme faktöründe belirgin bir azalma ve bozulmamış CeB<sub>6</sub> ile kıyaslandığında eşik alanında önemli bir artış gözlediler[47].Farklı bir çalışmada ise Si substrat üzerindeki CeB<sub>6</sub> kaplı CNT'lerden kaynaklanan yoğunlaştırılmış alan emisyonunun ve yeni kompozit materyalin gelecek nesil elektron kaynakları için potansiyel bir aday olarak önerilmesi gerektiği Patra ve diğerleri tarafından bildirildi. Filmi, kimyasal ve fiziksel biriktirme işlemlerinin bir kombinasyonu ile sentezlediler.CeB<sub>6</sub> kaplamalı CNT filminde, saf akım CeB<sub>6</sub> filmine kıyasla maksimum akım yoğunluğunda ve alan geliştirme faktöründe dikkate değer bir artış gözlediler. Bunun yanında, açılma alanı ve eşik alanında ise bir azalma gözlediler. Filmlerin element bileşimini ve yüzey morfolojisini SEM, TEM ve EDS ölçümleri ile incelediler. CNT'lerin üstünde ve duvarlarında CeB<sub>6</sub> nanopartiküllerini gösterdiler. Filmin TEM (Şekil 7 (a)) ve FESEM (Şekil 7 (b)) görüntüleri, CNT duvarlarının uçlarında ve yanlarında ve Si substratında CeB<sub>6</sub> nanoparçacıklarının büyümesini ortaya koydu[48].



The International Conference of Materials and Engineering Technology

**Şekil 9.**(a) CNT duvarlarındaki CeB<sub>6</sub> nanoparçacıklarının oklarla işaretlendiği CeB<sub>6</sub> kaplı CNT filminin TEM görüntüsü (b) CeB<sub>6</sub> kaplı CNT filminin FESEM görüntüleri Adopted from [48] @ 2014 AIP Publishing LLC.

### 2.5 LaB<sub>6</sub>-Alümina (Al<sub>2</sub>O<sub>3</sub>)

Alümina yüksek aşınma direnci, yüksek sıcaklıklarda iyi korozyon dayanımı, yüksek ergime sıcaklığı, düşük yoğunluk, iyi mekanik özellikleri yanı sıra düşük maliyet gibi ilgi çekici yönlere sahip olmasından dolayı yaygın kullanım alanına sahip bir malzemedir. Buna mukabil dezavantajları da mevcuttur. Bunların içinde en önemlisi ise düşük kırılma tokluğuna sahip olmasından dolayı gevrek ve kırılgan bir malzeme yapısına sahip olmasıdır. Alüminanın dezavantajı ise uygulama sıcaklıklarının sınırlı olmasıdır. Birçok araştırmacı, alüminadaki istenmeyen özelliklerinin elimine edilmesi amacıyla farklı bir malzeme ile takviye edilerek yeni bir kompozit malzeme oluşturulmasını hedeflemişlerdir [49,50].

LaB<sub>6</sub>-Al<sub>2</sub>O<sub>3</sub> seramik tozları partikül takviyesi olarak uygun aday olabilir ve seramik veya metalik matris kompozitlerin mikroyapısal ve mekanik özelliklerine katkıda bulunabilirler. Tekoğlu ve diğerlerinin yaptığı bu çalışmada, saf LaB<sub>6</sub>-Al<sub>2</sub>O<sub>3</sub> nanokompozit tozları, ilk kez yüksek enerjili bilyeli öğütme, tavlama ve liç işlemlerinden oluşan bir yöntem ile sentezlediler. Frezeleme zamanının LaB<sub>6</sub> ve Al<sub>2</sub>O<sub>3</sub> fazlarının oluşumuna etkisi detaylı mikroyapısal özellikler açısından incelediler[51].

#### 2.6 Diğer Kompozitler

LaB<sub>6</sub>@Si0<sub>2</sub>/Au kompozit nanoparçacıkları, sitratla kaplanmış Au nanoparçacıklarının, silika ile kaplanan LaB<sub>6</sub> nanoparçacıklarının yüzeyine birleştirilmesiyle başarılı bir şekilde Lai ve diğerleri tarafından imal edildi[52]. Lu ve diğerleri tarafından yapılan farklı bir çalışmada ise, TaC-LaB<sub>6</sub> kompozit seramikleri sıcak presleme ile hazırladılar. LaB<sub>6</sub>, TaC seramiklerinin yoğunlaştırılmasını kolaylaştırmak için TaC ile reaksiyona girebilir.LaB<sub>6</sub> ilavesinin TaC-LaB<sub>6</sub> kompozitlerin faz bileşimleri, mikroyapı ve mekanik özellikleri üzerindeki etkilerini incelediler. TaC-LaB<sub>6</sub>kompozitlerinin mekanik özellikleri LaB<sub>6</sub> içeriğine göre değişmiştir[53].

# 3. SONUÇ

Kompozitlerin sertliği, yüzey morfolojisi, optik, deşarj özellikleri, kırılma dayanaklığı, mikroyapı ve mekanik özelliklerini incelediler. $REB_6$ 'lar  $X^{IV}B_2$  ( $X^{IV}$ -Ti, Zr, Rf ve Hf), SiC, MgO, CNT ve Al<sub>2</sub>O<sub>3</sub> gibi malzemelerle kompozit oluşturmasının avantajları; yüksek mukavemet, kolay şekillendirebilmesi, üstün elektriksel özelliklere sahip olması, ısıya ve ateşe dayanıklılık gibi özellikler sıralanabilir. Dezavantajı ise; malzemelerin değişik doğrultularda çeşitli mekanik özellikler göstermesi, tokluğunun (dayanıklılık) ve süneklikliğin düşük oluşu, kompozit malzeme için eğilme mukavemet değerlerinin farklılıklar göstermesi, kompozit malzemelerin üretim maliyetinin yüksek olması gibi nitelikler söylenebilir. $REB_6$ 'ların  $X^{IV}B_2$  ile kompozit oluşturması



#### Referanslar

- 1. 1.Shishevan, F. A., Acar, V., Akbulut, H., & Seydibeyoğulları, M. Ö. Reçinesine Grafen ve Karbon Nanotüp Eklenmesinin Karbon Elyaf Takviyeli Kompozitlerin Mekanik Özellikleri ve Darbe Davranışları Üzerindeki Etkileri, **2015**.
- Buytoz, S.,&Yilmaz, O..Influence of thermal properties on microstructure and adhesive wear behaviour of Al/Al<sub>2</sub>O<sub>3</sub> MMCs. Materials science and technology,2006,22(6): 687-697.
- 3. Öztürk, M., "SiC ilaveli alümina seramik kompozitler", Yüksek Lisans Tezi, Sakarya Üniversitesi Fen Bilimleri Enstitüsü, Sakarya.2007. 4-77.
- Yastımoğlu, F.,& Özkan, A. Tekrarlanan Yükler Altında Kompozit Malzemelerin Yapılarının İncelenmesini Amaçlayan Deney Aygıtı Tasarımı. Düzce Üniversitesi Bilim ve Teknoloji Dergisi,2017, 5(1): 56-66.
- 5. Ashbrook, R. L. Directionally solidified ceramic eutectics. Journal of the American Ceramic Society, **1977**, 60(9-10): 428-435.
- Waku, Y., Nakagawa, N., Wakamoto, T., Ohtsubo, H., Shimizu, K., & Kohtoku, Y. A ductile ceramic eutectic composite with high strength at 1,873 K. Nature,1997, 389: 49–52.
- 7. E.C Dickey, V.P. Dravid, P.D. Nellist, D.J. Wallis, S.J. Pennycook, Acta Mater, **1998**, 46(5): 1801–1816
- 8. Loboda, P. I.Features of structure formation with zone melting of powder boron-containing refractory materials. Powder Metallurgy and Metal Ceramics, **2000**, 39(9-10):480-486.
- Paderno, Y., Paderno, V., & Filippov, V. Some peculiarities of eutectic crystallization of LaB<sub>6</sub>-(Ti, Zr) B<sub>2</sub> alloys. Journal of Solid State Chemistry, **2000**, 154(1): 165-167.
- 10. Chen, C.M., Zhang, L.T., & Zhou, W. C. Characterization of LaB<sub>6</sub>–ZrB<sub>2</sub> eutectic composite grown by the floating zone method. Journal of crystal growth, **1998**, 191(4): 873-878.
- 11. Paderno, V. N., Paderno, Y. B., Pilyankevich, A. N., Lazorenko, V. I., & Bulychev, S. I. The micromechanical properties of melted borides of rare earth metals. Journal of the Less Common Metals, **1979**, 67(2): 431-436.
- 12. Nakamoto, M., & Fukuda, K. Field electron emission from LaB<sub>6</sub> and TiN emitter arrays fabricated by transfer mold technique. Applied surface science, **2002**, 202(3-4):289-294.
- Bogomol, I., Nishimura, T., Vasylkiv, O., Sakka, Y., & Loboda, P. High-temperature strength of directionally reinforced LaB<sub>6</sub>–TiB<sub>2</sub> composite. Journal of Alloys and Compounds, **2010**,505(1): 130-134.
- 14. Bogomol, I., Nishimura, T., Vasylkiv, O., Sakka, Y., & Loboda, P. Microstructure and high-temperature strength of B<sub>4</sub>C–TiB<sub>2</sub> composite prepared by a crucibleless zone melting method. Journal of Alloys and compounds, **2009**, 485(1-2): 677-681.
- 15. Schwetz, K. A.Boron carbide, boron nitride, and metal borides. Ullmann's Encyclopedia of Industrial Chemistry,**1985**.

16. Kuznetsov, G. High temperature cathodes for high current density. Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, **1994**, 340(1):204-208.

The International Conference of Materials and Engineering Technology

- 17. Wang, S. C., Wei, W. C. J., &Zhang, L. T..Microstructural characterization of LaB<sub>6</sub>-ZrB<sub>2</sub> eutectic composites. In Key Engineering Materials.Trans Tech Publications, **2003**, Vol,(101-104).
- 18. Min, G. H., Gao, R., Yu, H. S., & Han, J. Mechanical properties of LaB<sub>6</sub>-ZrB<sub>2</sub>composites. In Key Engineering Materials.Trans Tech Publications.**2005**, Vol,(1630-1638).
- 19. Bogomol, I.,Nishimura, T., Nesterenko, Y., Vasylkiv, O., Sakka, Y., &Loboda, P..The bending strength temperature dependence of the directionally solidified eutectic LaB<sub>6</sub>– ZrB<sub>2</sub> composite. Journal of Alloys and Compounds,**2011**, 509(20):6123-6129.
- 20. Gao, R., Min, G., Yu, H., Zheng, S. Q., Lu, Q., Han, J., &Wang, W..Fabrication and oxidation behavior of LaB<sub>6</sub>-ZrB<sub>2</sub>composites. Ceramics International,**2005**,31(1):15-19.
- 21. Sokol, I.V. and Krosnova, T.V., "Composition of Titanium Boride Synthesis Products", Plenum Publishing Corporation, **1993**.
- 22. Weast, R. C. CRC Handbook of Chemistry and Physics 61st Ed 1980-1981. Chemical Rubber, **1981**.
- 23. Soloviova, T. O., Karasevska, O. P., Vleugels, J., &Loboda, P. I..Influence of annealing on crucible-free float zone melted LaB<sub>6</sub>-TiB<sub>2</sub> composites. Journal of Alloys and Compounds, **2017**, 729, 749-757.
- Nesmelov, D. D., Vikhman, S. V., Novoselov, E. S., Perevislov, S. N., &Ordan'yan, S. S. Structure, hardness and fracture toughness of arc-melted LaB<sub>6</sub>-TiB<sub>2</sub> eutectic alloy. In IOP Conference Series: Materials Science and Engineering.IOP Publishing,2019,Vol,(525).
- 25. S.P. Murarka, Refractory silicides for integrated circuits. Critical review, J. Vac. Sci. Technol, **1980**, 17: 775–792.
- 26. G. Medeiros-Ribeiro, D.A.A. Ohlberg, D.R. Bowler, R.E. Tanner, G.A.D. Briggs, R. Stanley Williams, Titanium disilicide nanostructures: two phases and their surfaces, Surf. Sci. 1999,431: 116–127
- 27. S.L.Zhou, J.X.Zhang, D.M.Liu, L.H.Bao, Properties of CeB<sub>6</sub> cathode fabricated by spark plasma reactive liquid phases intering method. J.Inorg. Mater, **2009**, 24(4):793–797.
- Sonber, J. K., Murthy, T. C., Sairam, K., Paul, B., & Chakravartty, J. K..Effect of TiSi<sub>2</sub> addition on densification of Cerium hexaboride. Ceramics International, 2016, 42(1):891-896.
- 29. Pierson, H.O. Handbook of Refractory Carbides and Nitrides. New Jersey, Noyes Publications, **1996**.
- 30. F. Wang, L. Cheng, Y. Xie, J. Jian, L. Zhang, Effects of SiC shape and oxidation on the infrared emissivity properties of ZrB<sub>2</sub>-SiC ceramics, J. Alloys Comp, **2015**, 625:1-7.
- 31. M.S. Asl, M.G. Kakroudi, S. Noori, Hardness and toughness of hot pressed ZrB<sub>2</sub>-SiC composites consolidated under relatively low pressure. J. Alloys Comp,**2015**,619: 481-487
- 32. I.Akin, M. Hotta, F.C. Sahin, O. Yucel, G. Goller, T. Goto, Microstructure and densification of ZrB<sub>2</sub>-SiC composites prepared by spark plasma sintering.J. Eur. Ceram. Soc., **2009**, 29: 2379-2385.
- 33. W.B. Tian, Y.M. Kan, G.J. Zhang, P.L. Wang, Effect of carbonnanotubes on the properties of ZrB<sub>2</sub>-SiC ceramics. Mater. Sci. Eng., **2008**, A 487: 568-573.
- 34. Yang, X., Wang, X., Wang, P., Hu, K., Li, Z., &Zhang, J..Spark plasma sintering of SiC-LaB<sub>6</sub> composite. Journal of Alloys and Compounds, **2017**, 704:329-335.
- 35. Zhu, Q.,Oganov, A. R., &Lyakhov, A. O..Novel stable compounds in the Mg–O system under high pressure. Physical Chemistry Chemical Physics,**2013**,15(20):7696-7700.

36. Shand, M. A. The chemistry and technology of magnesia .New York: Wiley-Interscience, **2006**, Vol, (210).

UWE dithe Bristol West

MIST

The International Conference of Materials and Engineering Technology

- 37. Deng J, Zeng BQ, Wang XJ, Lin ZL, Qi KC, Cao GC. IEEE Trans Electron Device Lett,**2013**,34:1026–8.
- 38. Deng, J., Zeng, B., Wang, Y., Wang, X., Lin, Z., Qi, K., & Cao, G. Fabrication and characteristics of LaB<sub>6</sub>-doped MgO protective layer for plasma display panels. Materials Letters.2014. 134:51-55.
- 39. De Volder, M. F. L., Tawfick, S. H., Baughman, R. H. and Hart, A. J., "Carbonnanotubes: present and future commercial applications", Science, 2013, 339:535-539
- 40. Dresselhaus, M. S., G. Dresselhaus and P. Avouris, "Carbonnanotubes", Preface. Springer, USA.,2000.
- 41. Velasco-Santos, C., Martinez-Hernandez, A.L., & Castano, V. M.. Carbonnanotubepolymernano composites: The role of interfaces. Composite Interfaces, 2005, 11(8-9):567-586.
- 42. W. Xiaoju, L. Zulun, Q. Kangcheng, C. Zexiang, W. Zhigang, and J. Yadong, J. Phys. D: Appl. Phys., 2007, 40:4775.
- 43. W. Wei, J. Kaili, W. Yang, L. Peng, L. Kai, Z. Lina, L. Qunqing, and F. Shoushan, Appl. Phys. Lett.,2006,89: 203112
- 44. Kumari, M., Gautam, S., Shah, P. V., Pal, S., Ojha, U. S., Kumar, A., ... & Tandon, R. P.. Improving the field emission of carbonnanotubes by lanthanum-hexaboride nano-particles decoration. Applied Physics Letters.,2012, 101(12):123116.
- 45. Patra, R., Ghosh, S., Sheremet, E., Jha, M., Rodriguez, R. D., Lehmann, D., & Zahn, D.R.T..Enhanced field emission from lanthanum hexaboride coated multiwalled carbonnanotubes: Correlation with physical properties. Journal of Applied Physics.,2014, 116(16): 164309.
- 46. M.L. Bauccio, ASM Engineered Materials Reference Book, ASM International United States of America, 1994.
- 47. Jha, M., Patra, R., Ghosh, S., & Ganguli, A. K.Vertically aligned cerium hexaboride nanorods with enhanced field emission properties. Journal of Materials Chemistry, 2012, 22(13), 6356-6366.
- 48. Patra, R., Ghosh, S., Sheremet, E., Jha, M., Rodriguez, R. D., Lehmann, D., ... & Zahn, D. R. T. Enhanced field emission from ceriumhexaboride coated multiwalled carbonnanotube composite films: A potential material for next generation electron sources. Journal of Applied Physics, **2014**, 115(9):094302
- 49. Torralba J. M., da Costa C. E., Velasco F..P/M Aluminum Matrix Composites: An Overview. Journal of Materials Processing Technology, 2003, 133: 203–206.
- 50. Çerezci T., Nikel Partikül Takviyeli Alümina Seramik Kompozitlerin Sentezi ve Karakterizasyonu, Yüksek Lisans Tezi, Sakarya Üniversitesi, Fen Bilimleri Enstitüsü, Sakarya.2008, 21604
- 51. Tekoğlu, E., İmer, C., Ağaoğulları, D., & Öveçoğlu, M. L..Synthesis of LaB6-Al<sub>2</sub>O<sub>3</sub>nanocompositepowdersviaballmilling-assistedannealing. Journal of materials science, 2018, 53(19):13538-13549.
- 52. Lai, B. H., Lin, Y. R., & Chen, D. H. Fabrication of LaB<sub>6</sub>@ SiO<sub>2</sub>/Au composite nanoparticles as a catalyst with near infrared photothermally enhanced activity. Chemical engineering journal, 2013, 223, 418-424.

53.Lu, Z.,Liu, L., Hou, Z., Geng, G., Wang, Y., Sun, W., ... & Chen, Y. Effects of LaB<sub>6</sub> content on microstructure and mechanical properties of TaC-LaB<sub>6</sub> composites. Materials Chemistry and Physics, 2018, 213:374-382.

469

# DESIGN OF A NEW REWINDING SYSTEM FOR CORELESS STRETCH FILM PRODUCTION

The International Conference of Materials and Engineering Technology

# HATİCE NİDA CİVAN\*, HADİ SAMET MUMCU, OSMAN YAYLA

<sup>1</sup>Selçuk İplik San. ve Tic. A.Ş., R&D Center, Gaziantep, TÜRKIYE

#### Abstract

Stretch film is the name given to the protective structure containing polymer raw material, which enables the product to be wrapped after tension adjustment. One of the stretch film production techniques is cast technology. Briefly, stretch film is produced by pouring molten linear low density polyethylene material (LLDPE) onto the cold cylinder by extruder. Stretch films are used in many different fields from food industry to packaged transportation. Stretch film is classified into two types which are home type stretch film and industrial type stretch film. Stretch film rolls used today are wound onto cellulosic tubes and after the end of the stretch rolls, the tubes are not reused. Industrial stretch film of 20 microns, 40 cm wide and 200 meters wrapped stretch roll weights nearly 1720 g and also has a heavy weight cellulosic tube with an approximate weight of 250 g. Maximum use of limited resources in pulp and paper industry is one of the challenges for millons of people around the world today. The world is warming up and the impact of future warming on the air conditioning system cannot be underestimated. The carbon footprint of both fossil fuel emissions and the products produced in nature can be reduced by cultivating and protecting forests. Paper products are increasingly consumed every year. In this study, using the newly designed innovative shaft, the stretch film can be wound without the use of heavy weight cellulosic tube. New solution is design of a mechanism that work on stretch film rewinding machines. A stretch rewinding mechanism was designed to produce for industrial stretch films to be wound without heavy weight cellulosic tube. Shipment of empty tubes is eliminated, time and raw material are saved by this environmentally friendly production.

Keyword: coreless stretch film, carbon footprint, rewinding machine.

#### **1. Introduction**

Today, for millions of people around the world, the maximum use of limited resources in the pulp and paper industry is one of the most important challenges [1]. The world is warming up and impact of future warming on the air conditioning system can not be underestimated. The carbon footprint of both fossil fuel emissions and the products produced in nature can only be reduced by cultivating and protecting forests [2]. The amount of trees spent as a result of paper consumption is seven per person on average. Millions of tons of paper and paper products are consumed every year. Developing the recycling technologies is one of the solutions [3]. With these effects, coreless stretch film production technology has been developing rapidly in recent years in terms of reducing costs, reducing the use of heavy weight cellulosic tube, and causing less environmental damage. Stretch film is widely used in furniture industry, glassware industry, white goods sector, and other business area. Industrial stretch film ensures the stability of packaged products. The materials placed in the cartons are wrapped with thick stretch film to prevent damage during transportation. Industrial type stretch film manufacturing companies make their production according to demand in desired thickness and size of stretch film production. Large-scale and thick stretch films are required for companies to produce large-scale materials, while small-scale materials such as glass industry needs smaller stretch films. Coreless stretch film production technology has been developing rapidly in recent years in terms of reducing costs, reducing the use of heavy weight cellulosic tube and causing less environmental damage.

The International Conference

Coreless film technology is an innovation which saves environmental resources for eco-conscious consumers and organizations with sustainability goals. Pallet can be optimized with maximum load holding to produce lightweight, coreless, and high performance stretch film. Elimination the use of the heavy weight cellulosic tube reduces cost of transporting and storing. 100% of produced product can be used. Utilization can be increased. Using the stretch film until the end provides the reduction of waste generated. Our company manufactures stretch films between 9-50 microns. Hand type, machine type, and jumbo type are available in the product range. Production can be made in the dimensions requested by the customer. Our company is capable of producing 70 tons of stretch film daily. In our company, in the first stage, industrial stretch film rolls are produced to have 17 kg roll weight. Then these rolls are rewinded onto heavy weight cellulosic tubes to obtain the requested stretch film quantities by rewinding machines. Rewinding machines cover approximately 10-20% of the production area (Figure 1). In this study newly designed system in semi-automated rewinding machine provided space saving. Additionally, time and raw material are saved by this environmentally friendly design.



Figure 1. Semi-automated rewinding machine

# 2. Materials and Methods

#### 2. 1 Design of the New System

Present machine rewinds the stretch film from jumbo roll with automatic cellulosic tube transferring. Machine stops mechanically when the heavy weight cellulosic tube is finished. Hot wire cuts stretch film. Shaft width is 600 mm and main motor is 4 Kw. Machine speed is adjustable

Technology

till 1200m/min. Heavy weight cellulosic tubes are changed in every 23 seconds. Jumbo roll width is 300-520mm. Heavy weight cellulosic tube diameter can be 38-50-76 mm. Required meter or weight of stretch film can be adjusted on machine.

The International Conference

Pneumatic shafts are used in different production systems. In this study, design process is done in two phase. Firstly a brand new pneumatic shaft is designed (Figure 2 - left hand side) and manufactured. Then, different samples are produced with this shaft. The stretch film samples produced with this shaft are assessed regarding stretch film visible winding quality, ease of separation of film from the shaft, micron diversity and maximum production speed. After the assessments, since the samples do not have the adequate quality levels, it is decided to design a new shaft to increase the quality level. Then, the later shaft (Figure 2 - right hand side) is designed and manufactured. With the later design, optimum quality levels regarding stretch film visible winding quality, ease of separation of film from the shaft, micron diversity and maximum production speed are obtained. The sample produced with the later shaft design is tested for elasticity and puncture resistance and the results are compared with the standard products which have different stretch film thickness values.

### **2.2 Test Methods**

The samples produced by the later shaft are tested for elasticity properties of stretch film products according to ASTM D882. In this elasticity properties of stretch film product are determined. The other test applied to stretch film products is protrusion puncture resistance. Protrusion puncture resistance of stretch wrap film test measures the maximum force required to pierce the film. Puncture test is done according to ASTM D5748.



Figure 2. Pneumatic shaft and designed shaft

#### **3. Results and Discussion**

In this study, tensile and puncture performance, stretch film visible winding quality, ease of separation of film from the shaft, micron diversity and maximum production speed were

Technology



investigated for newly designed systems. Experimental design of the study is given in Table 1 and Table 2. Test results are given in Table 3.

	Table 1. Experimental design of first phase with the first shaft design						
No	Technique Used	Bobbin Length (cm)	Thickness (µm)	Production rate (m/min)	Internal/ External Surface	Winding Length (m)	Quality result
1	-	40	11	250	Hexagonal /hexagonal surface	100	Low
2	greaseproof paper	40	11	250	Hexagonal /hexagonal surface	100	Low
3	oily silicone sprey	40	11	250	Hexagonal /hexagonal surface	40	Low
4	Greaseproof paper, oily silicone sprey	40	19	250	Hexagonal /hexagonal surface	40	Low
5	PVC	40	11	250	Not leave the shaft	200	Low
6	PVC, oily silicone sprey	40	19	250	Not leave the shaft	100	Low
7	PVC, oily silicone sprey, laminated	40	11	100	Not leave the shaft	200	Low
8	PVC, oily silicone sprey, laminated paper	40	11	100	Not leave the shaft	100	Low
9	Rubber	40	24	250	Hexagonal and distorted /hexagonal surface	40	Low
10	Rubber, oily silicone sprey	40	24	250	Hexagonal and distorted /hexagonal surface	40	Low
11	White apparatus	40	19	300	Hexagonal and distorted /ellipse surface	40	Low

	Table 2. Ex	perimenta	l design of se	cond phase wi	th the second	d shaft desig	'n
No	Technique Used	Bobbin Length (cm)	Thickness (µm)	Production rate (m/min)	Internal/ External Surface	Winding Length (m)	Quality result
1	Oily silicone sprey	40	16	600	Proper surface	100	High quality

The International Conference of Materials and Engineering Technology

a ı	C (	Thickness	Elongation	Puncture resistance
Sample	Core type	(µm)	(%)	(kgf)
<b>S</b> 1	Coreless stretch film	24	349.92%	3.36
<b>S</b> 2	Standard stretch film	24	351.05%	2.66
<b>S</b> 3	Standard stretch film	20	336.48%	2.35
<b>S</b> 4	Standard stretch film	23	354.83%	3.15

# 4. Conclusions

The main reason for the development of the study is that coreless stretch film usage is not widespread. Customer demand, economic factors, and especially environmental conditions have been strong reasons for the formation of the study. When all these aspects are considered, the design of the new system has been developed with prototype and related trials have been made. Stretch film tests were applied to the developed samples. According to the test data and test results, the following outcome were obtained.

The force applied by the speed of the machine, the design of the pneumatic shaft, the used technical with the shaft (spray, greaseproof paper, silicone, PVC, gray tape), stretch micron fineness were observed as factors affecting the stretch product without the heavy weight cellulosic tube. As the micron fineness decreases, the separation of the stretch film from the shaft becomes more difficult as the winding is stronger and the desired results could not be achieved. Design of shaft has been an important factor to make a difference in whether or not the stretch film separates from the shaft. Stretch film tests were compared to the test results. Test method for tensile properties, other test method for tensile properties and protrusion puncture resistance of stretch wrap film test results were between target values.

#### References

1. White, A., Cannell, M.G. and Friend, A.D., Climate Change Impacts on Ecosystems and The Terrestrial Carbon Sink: a New Assessment, Elsevier, 1999, pp. S21-S30

2. Carrere, R. and Lohmann, L., Pulping the South Industrial Tree Plantations and the World Paper Economy, London and New Jersey: Zed Books Ltd , 1996.

3. Oliveira, J.R.M., Silva, H.M.R.D., Pais, J.C., Different Solutions for Road Pavement Recycling, Nova Science Publishers Incorporated, 2013.

# BİR AĞIR VASITA HAVA KOMPRESÖRÜNÜN TERMODİNAMİK DAVRANIŞININ DENEYSEL İNCELENMESİ

The International Conference

ials and Engineering Technology

# MURAT ŞÜKRÜ AYDINER <sup>1</sup>, ERDEM ÜNÜVAR <sup>1</sup>, METE KALYONCU <sup>2</sup>

<sup>1</sup> Yıldız Pul Otomotiv Motor Parçaları Sanayi A.Ş. 42250 Konya/TURKEY

<sup>2</sup> Mechanical Eng. Department, Konya Technical University, Faculty of Engineering and Natural Sciences, 42250 Konya/TURKEY

# Özet

Bu çalışmada, bir ağır vasıta hava kompresörünün termodinamik davranışı deneysel olarak incelenmiş ve teorik olarak elde edilen verilerle karşılaştırılmıştır. Araçlarda pistonlu hava kompresörleri her geçen gün çok yaygın olarak kullanılmaktadır. Ağır vasıtalarda basınçlı hava üretme görevini üstlenen hava kompresörlerinin termodinamik davranışı birçok parametreye doğrudan bağlı olarak değişmektedir. Bu parametrelerin en başında hava kompresörünün hacmi, sıcaklığı, devir sayısı, set basıncı, soğutma sıvısı tipi ve sıcaklığı, çevre sıcaklığı gibi parametreler gelmektedir. Numune olarak alınan bir ağır vasıta hava kompresörünün gerçek çalışma şartlarını simule edebilen bir test sistemi kullanılarak, bir çevrim esnasında çeşitli şartlar altındaki termodinamik davranışı yapılan deneyler ile tespit edilmiştir. Farklı parametre değerlerinin hava kompresörünün termodinamik davranışını nasıl etkilediği yapılan deneyler sonucunda elde edilmiş ve sonuçlar teorik olarak hesaplanan değerler ile karşılaştırmalı olarak grafikler halinde sunulmuştur. Teorik olarak hesaplanan değerler ile deneysel olarak elde edilen verilerdeki farklılıklar tartışılmıştır.

**Anahtar kelimeler:** Pistonlu hava kompresörü, termodinamik davranış, deneysel inceleme, PV diyagramı, indikatör diyagramı.



# Giriş

Hava kompresörleri ile üretilen hava; kamyon, tır-yarı römork ve otobüslerde yaygın olarak kullanılmaktadır. Ortamdan alınan atmosferik hava sıkıştırılarak depolanmaktadır. Depolanan bu basınçlı hava fren sistemlerinde, süspansiyonlarda ve havaya ihtiyaç duyulan çeşitli bölgelerde kullanılmaktadır. Çevreci ve temiz bir enerji kaynağı olan basınçlı hava sistemi motorun üretmiş olduğu gücün yaklaşık %2lik kısmını kullanmaktadır.[1]

Pistonlu hava kompresörleri birçok mühendislik uygulamasında kullanılmaktadır. Burada diğer kompresör tiplerinden ayrılan yanı ise; aynı hacmi daha düşük güç ile daha yüksek hacme çıkarabilmesidir.[2]

Hava kompresörleri, basitçe emme ve havayı basınçlandırıp aktarma görevini üstlenmektedirler. Basınçlanan hava ise su ve yağ filtresinden geçerek tanklarda depolanır. Hava kompresöründen çıkan hava, tanka ulaştırılması esnasında soğuma gerçekleştirilir. Soğuma yeterli seviyede gerçekleşmez ise tankta soğumaya devam eden havada yoğuşma devam eder. Böylece tankta soğumaya devam eden hava, su birikmesine neden olur ve tanktan sonra eğer filtre kullanılmaz ise havayı kullanan sistemlere zarar verebilir.

Basınçlanan hava sıkışma anında 300°Clere kadar sıcaklık farkı oluşturabilmekte ve yüksek verime çıkmasının yanında yüksek termal etkileri olabilmektedir. Bu basınçlanma ve termal etkiler beraberinde komple sistem olan hava kompresörünün farklı parametrelerinin de etkilenmesine/etkilemesine sebep olmaktadır.

Hava kompresörleri farklı set basınç değerlerinde ayarlanabilmektedir. Farklı devir ve hız ile çalışan sistemler olan hava kompresörleri genel olarak 600 ile 3000dev./dk. arasında 8-12,5 bar set basınç değerlerinde çalışabilmektedir. Bu çalışmada ise aracın yoğun çalıştığı düşünülen devri ile genel olarak araçlar üzerinde fabrika basınç set değeri olan 10-10.5 set değerleri referans alınarak çalışmalar yapılmıştır. Burada temel hedef kompresörün benzer şartlar altında referans değerler değiştirilmeden termal etkiler incelenmek istenmiştir. Uygulamalı deney ile yapılacak olan test sonuçları ise ana sanayi firmasının üretmiş olduğu aynı hacme sahip hava kompresörünün katalog değerleri ile kıyaslanmış ve sonuçlar yorumlanmıştır. Şekil 1. de örnek bir farklı hızlara ait güç değerlerinin verildiği grafik görülmektedir. Bu grafik aynı zamanda testlerimizin yapıldığı hava kompresörüne ait performans değerleridir.

Technology



The International Conference of Materials and Engineering Technology

Şekil 1 OEM firmasının yayınlamış olduğu devir-güç grafiği

Günümüzde testler ve test merkezleri çok yaygınlaşmıştır. Özellikle otomotiv alanında teknolojinin ve imkanların gelişmesiyle birlikte araçların daha stabil konforlu gitmeleri hedeflenmektedir. Bunların başında da motor ve diğer dinamik parçaların testlerinin yapılması öncelik taşımaktadır.[3] Ağır vasıta araçlar için hava kompresörleri ise içten yanmalı motorlara benzerliğinden dolayı test parametreleri ya da elde edilecek grafikler benzerlik göstermektedir. En ortak özelliği ise tıpkı motorlar gibi pistonlu mekanizmaya sahip olmasıdır. Bu sebeple piston çalışma yapısını incelenebilmesi adına basınç-hacim (P-V) grafikleri de test cihazı üzerinden alınmıştır.[4] Böylece farklı bir parametrenin aynı şartlar altında grafiğe etkisi incelenmiştir.

Hava kompresörünün temel parametresi olan hava, Emiş hattı hava sıcaklığı, kompresör sıkıştırma oranı ve hacmi gibi etmenlerden dolayı farklı termal etkilere maruz kalmaktadır. Ayrıca soğutma sıvısının sıcaklığının farklı olması da havanın debi miktarına ve kompresörün çekmiş olduğu gücü etkilediği düşünülmektedir. Bu çalışmada yapılan deneyler ile de bu düşüncenin doğruluğu incelenmiştir.

## Materyal ve Yöntem

### Önemli Performans Parametreleri

Çalışmanın temel hedeflerinden olan sıcaklık parametresi, hava kompresörünün performansına etkisi olması ve sisteme doğrudan etki eden diğer parametrelerin sabit tutulmak istenmesi sebebiyle; öncelikli olarak deney düzeneğinden elde edilecek parametreler belirlenmiştir. Bu bağlamda sistem üzerinde kontrol edilecek öncelikli parametreler:

The International Conference of Materials and Engineering

Technology

<u>Motor Hızı</u>: Ağır vasıta hava kompresörlerinin birçoğu araç motorundan ya doğrudan ya da dolaylı olarak tahrik almaktadır. Bu sebeple aracın devri ile doğru orantılı olarak hava kompresörünün devri de değişmektedir. 600-3000dev./dk. arasında değişen motor hızı kompresör üzerinde de dişli oranına bağlı olarak genellikle bu seviyededir. Araç devri ise, hava debisi ve tüketilen güç miktarını doğrudan etkilemektedir. Bu sebeple kompresör devri deneylerimizde sabit tutulmuştur.

<u>Tank Basınç değeri</u>: Tank basıncı, yani hava kompresörünün basınçlandırmış olduğu havaya karşı basınç; hava kompresörünün tükettiği güç ve debiyi doğrudan etkileyen bir diğer parametredir. Örneğin, tank basıncı ne kadar yüksek olursa hava kompresörünün tüketmiş olduğu güç o kadar artarken; birim zamanda ya da periyotta üretilen hava debisi o kadar az olur. Hava kompresörünün performans analizinde termodinamik etkiyi inceleyebilmek için bu parametre de motor hızı gibi sabit tutulacaktır.

<u>Soğutma Sıvısı Sıcaklığı (Su Sıcaklığı</u>): Soğutma sıvısı araç motoru ile hava kompresörü ortak kullanmaktadır. Soğutma sıvısının debi miktarı ise araç motorunun devri ile doğru orantılıdır. Yani araç motorunun devri (sıvının debisi), sıvının cinsi (antifriz oranı) ve sıvının sıcaklığı havanın sıkıştırma sıcaklığı dahil soğuma hızının belirlenmesinde doğrudan etkilidir. Bu çalışmada ise havanın soğuma karakteri ve bu durumun debi ile güç gibi parametrelere etkisinin incelenmesi için yapılacak olan deneylerde bu parametre değişkenlik göstermiştir.



he International Conference

Şekil 2 Deneylerin yapıldığı test ortamı

Sistemden kontrolü sağlanarak gözlemlenecek diğer veriler ise:

<u>Hava Kompresörü Güç Bilgisi (Motor Gücü)</u>: Ana sanayi (OE) hava kompresörü üreticisi firmaların öncelikli olarak paylaştığı katalog değerlerinden birisidir. Araç motorundan çekilen gücün fazla olması yakıt tüketimini arttırması, konforlu sürüşü etkilemesinin yanında, kompresör tarafında çekilen gücün hava debisi ile oranı kompresörün verimi hakkında bilgi vermektedir.

<u>Hava Kompresörü Debi Bilgisi</u>: Ağır vasıta araçlarda hava kompresörlerinin en önemli parametresi havadır. Havanın debi miktarı ise aynı hacme sahip kompresörlerin dış etkenlere bağlı performans parametresini belirlemektedir. Yani aynı şartlar altında iki aynı hava kompresörünün performansını belirleyici en önemli parametrelerden birisidir. Bu çalışmada ise hava debisi ve motordan çekilen güce bakılacaktır. Böylece aynı şartlar altında bir parametrenin değişimi (soğutma sıvısının sıcaklığı) ile hava debisi ve kompresörün çekmiş olduğu gücün değişimlerinin tespitine göre yorumlama yapılabilecek ve OE katalog değerleriyle bu sonuçların karşılaştırmaları yapılmıştır.

<u>Hava Kompresörü Sıkıştırma Sıcaklığı Bilgisi</u>: Hava kompresörü bir çevrim süresi 10ms ile 2ms arasında tamamlayabilmektedir (600-3000dev./dk.). Bu çevrimler esnasında sıkıştırılabilir bir faz olan havanın, kompresörün içyapısı ve çevre şartları sebebiyle birçok parametreden etkilenmesi mümkündür. Sıkıştırılan havanın termodinamik yapısı gereği sabit kütle ve azalan hacme bağlı olarak basınç ve sıcaklığı yükselmektedir. Sıkışma esnasında meydana gelen bu sıcaklık, basınçlanma sonrası hat üzerinde ilerlerken hızla soğumaya başlamaktadır. Yüksek sıcaklık sonrası soğuma ise moleküllerin hızını azaltarak basınç seviyesini bir miktar azaltmakta, daha önemlisi birimi zamandaki debi miktarını etkilemektedir. Yapılan deneyler ile ise bu hava debisi ve

Technology

aterials and Engineering



kompresörün çekmiş olduğu güç miktarına etkisi incelenmiştir. Ayrıca sıkışma anındaki sıcaklık değişimi de grafikler ile incelenmiştir.[2, 5]

# Deneyin Yapılması ve Deney Şartları

Yapılacak olan deney için öncelikli olarak şartlar belirlenmiştir. Bu bağlamda öncelikli olarak 636cc hacme sahip bir kompresör belirlenmiştir. Belirlenen hava kompresörünün çalışma şartları simule etmek amacıyla tam otomatik bir test sistemi hazırlanmıştır. Yapılacak bu çalışma için iki farklı deney planı hazırlanmıştır

Deney-1: Soğutma sıvısı sıcaklığı ortam sıcaklığı olan 22°C olarak belirlenmiştir. Çevre şartları ve kompresörün çalışmasını etkileyen diğer tüm parametreler deney-2 ile aynı tutulmuştur.

Deney-2: Soğutma sıvısı sıcaklığı kompresör hararet sıcaklık aralığı olan 85-95°C olarak belirlenmiş ve diğer tüm şartlar deney-1 ile aynı tutulmuştur.

Kompresör test cihazı olanaklarıyla 600-3000dev./dk. aralığında tıpkı araç üzerindeki gibi çalıştırılabilmektedir. Her iki deney için de 1750dev./dk. sabit hız kullanılarak deney parametreleri aynı tutulmaya çalışılmıştır. Burada hedeflenen parametre, sıcaklığın debi ve güç üzerindeki etkisinin olması da bu fikri güçlendirmiştir. Böylece hedeflenen çıktılara en kısa ve mümkün deneyler ile ulaşılmış, teorik ile kıyaslamasını olanaklı kılmıştır.


Şekil 3 Pistonlu bir mekanizmanın bir çevriminde olması gereken teorik grafiğin görünümü [6]

Tank set basınç değeri ise araçlarda genel olarak 10,5 bar seviyelerine set edilmektedir. Bu sebeple ısınmanın etkisini daha net tanımlayabilmek için 10 bar seviyesinde test basınç değeri tercih edilmiştir. [7]

Ortam sıcaklığı ise oda şartlarında olup her iki deney için de 20-24°C arası tespit edilmiştir.

Yukarıda verilen parametre ve belirlenen değerler ışığında:

1750dev./dk. motor hızı ve bu hızla orantılı yağ ve soğutma sıvısı hızı kullanılmıştır.

10bar seviyesinde basınç sabit tutularak öncelikle soğuk sonrasında soğutma sıvısının rezistans marifeti ile ısıtmak kaydı ile sıcak olarak deneylerimiz gerçekleştirilerek verilerimiz sistem üzerinden toplanmıştır.

Şekil-3 ile verilen örnek bir P-V grafiğinde görüldüğü üzere aşağıda Şekil 6 ve Şekil 7 de deney-1 ve deney-2'ye ait P-V grafiği ve bu grafik anında alınan debi güç ve sıcaklık değerleri görülmektedir



The International

Con

aterials and Engineering Technology

UWE Bristol

Şekil 4 Örnek bir silindir basıncına ait P-V grafiği (1800dev./dk., 11,58bar) [8]



Şekil 5 Hazırlanan deney ortamında oluşturulmuş, bir çevrime ait grafiğin gösterimi



Şekil-4 incelendiğinde numerik olarak oluşturulmuş 1800dev./dk.'ya ait 11,58bar set basınç anında kompresörün bir çevrimini gösteren grafik görülmektedir. Şekil-5'te ise 1750 dev./dk., 11,58bar basınç anındaki deney sonucu görülmektedir. Şekil-4 ve Şekil-5 ayrı ayrı incelendiğinde ise grafiklerin emme ve basınçlanma anındaki karakterlerinin benzerlik gösterdiği gözlemlenecektir. Bu durum sistemimizdeki deneysel tasarımın doğruluğu hakkında bilgi vermektedir.

The International Con

als and Engineering Technology



Şekil 6 Deney-1 sonunda elde edilen P-V grafiği ve çevrim anında elde edilen performans değerleri



The International Conference

terials and Engineering Technology

Şekil 7 Deney-2 sonunda elde edilen P-V grafiği ve çevrim anında elde edilen performans değerleri

# Deney Sonuçlarının İncelenmesi ve Tartışma

Deney sonuçları incelendiğinde, her iki deney neticesinde de benzer grafikler elde edilmiştir. Fakat elde edilen verileri incelediğimizde üç farklı çıktı parametrenin de belirgin şekilde farklılık gösterdiği görülmüştür. Aşağıda verilen tablo-1 incelenirse grafikler arasındaki farklar belirgin şekilde fark edilecektir.



Tablo 1 Soğutma sıvısı sıcaklığının performans parametreleri üzerindeki termodinamik etkisi

Sıkıştırma Sıcaklığı(°C)		Hava Debisi (m <sup>3</sup> /dk.)	Tüketilen Güç (kW)
Deney-1	221,01	0,6197	6,194
Deney-2	279,07	0,5725	5,540
Katalog Değerleri	Bilinmiyor	0,6263	6,164

Deney sonuçlarında görülmüştür ki, hava kompresörünün soğutma sıvısı sıcaklığının yüksek olması havanın çıkış sıcaklığının yüksek olmasına sebep olmaktadır. Bu durum hava debisinin azalmasına sebep olmaktadır. Ayrıca tüketilen güç miktarında da düşüş gözlemlenmektedir.

Katalog değerleri, maalesef testlerin hangi şartlar altında verildiği göstermemektedir. Yapılan test sonuçları incelendiğinde ise soğutma sıvısının soğuk olduğu yani deney-1 ile elde edilen değerlerin daha yakın olduğu gözlemlenmiştir.

Sıkıştırma anında deney-1 için ortam sıcaklığı, 20°C'lerden 220°C'lere kadar çıktığı; deney-2 için 20°C'lerden 280°C'lere varan bir sıcaklık yükselmesinin olduğu

gözlemlenmiştir.

Oluşan P-V grafikleri incelendiğinde ise deney-1 ve deney-2 arasında belirgin bir fark olmadığı, fakat deney çıktıları arasında belirgin bir fark olduğu görülmüştür.

# Yorumlar

Ağır vasıta araçlar üzerinde bulunan hava kompresörleri, araç motorundan soğutma sıvısı, hava gibi parametreleri ortak kullanması sebebiyle araç hararet sıcaklığı olan 85-95°C seviyelerinde soğutma sıvısını kullanmak zorundadır. Yapılacak yenilikçi çalışmalar ile birlikte araç üzerinde revizyonlar yapılarak araçta soğutma sıvısının bağımsız olarak bağlanması ve daha düşük sıcaklıklar ile çevrim yapılması kompresörün verimini olumlu yönde etkileyecektir. Yapılacak kapsamlı bir hesaplama ve fizibilite çalışması sonrası ağır vasıta hava kompresörleri daha verimli hale getirilebilir.

Günümüz yeni nesil ağır vasıta araçlarda elektrik motorundan harici olarak tahrik alabilen sistemlerin olduğunu düşünürsek bu tarz bir çalışmanın yapılarak hava kompresörleri için daha verimli çalışma ortamlarının oluşması sağlanabilir. Ayrıca hava kompresörlerinin daha soğuk soğutma suyu ile çalışması ile birlikte sıcaklık farkının daha az oluşacağı ve bu durumun hat üzerinde daha az yoğuşmaya sebep olması beklenmektedir. Böylece hava ile çalışan ağır vasıta bileşenlerinin bu durumdan daha az etkilenmesi beklenmektedir.

The International Conference of Materials and Engineering Technology

# Referanslar

- 1. Wang, X.F., et al., *Affect of Power Consumption Decline of Air Compressor on Oil Saving of Heavyduty Vehicle*. 20139th Ieee Vehicle Power and Propulsion Conference (Vppc), 2013: p. 493-497.
- 2. Stouffs, P., M. Tazerout, and P. Wauters, *Thermodynamic analysis of reciprocating compressors*.
- 3. International Journal of Thermal Sciences, 2001. 40(1): p. 52-66.
- **4.** Öztürk, F.Ö.N., *Otomotiv Yan Sanayii Tasarım Yeteneğinin Değerlendirilmesi*. Uludağ Üniversitesi-Mimarlık Mü-hendislik Fakültesi Dergisi, 2005. **Cilt 10**: p. 1.
- 5. Tang, B., et al., *Thermal performance analysis of reciprocating compressor with stepless capacity control system*. Applied Thermal Engineering, 2013. **54**(2): p. 380-386.
- 6. Roskosch, D., V. Venzik, and B. Atakan, *Thermodynamic model for reciprocating compressors with the focus onfluid dependent efficiencies*. International Journal of Refrigeration-Revue Internationale Du Froid, 2017. 84: p. 104-116.
- Lin, J.M., et al., *Finite-time thermodynamic modeling and analysis of an irreversible Miller cycle working on a four-stroke engine*. International Communications in Heat and Mass Transfer, 2014. 54: p. 54-59.
- 8. Subramanian, K.S.G., *Reed Valve Dynamics of Reciprocating Compressor*. International Journal of Engineering Research & Technology (IJERT), 2016. **5**(9): p. 5.
- **9.** Farzaneh-Gord, M., et al., *Thermodynamic analysis of natural gas reciprocating compressors based on real and ideal gas models.* International Journal of Refrigeration-Revue Internationale Du Froid, 2015. **56**: p. 186-197.

# ELECTROCHEMICAL CORROSION BEHAVIOR OF HEAT-TREATED 2205 DUPLEX STAINLESS STEEL IN CHLORIDE SOLUTION

The International Conference

aterials and Engineering Technology

# **HUSEYIN ZENGIN<sup>\*1</sup>**

<sup>1</sup> Karabuk University, Engineering Faculty, Department of Metallurgical and Materials Engineering, Karabuk, TURKEY.

#### Abstract

In this study, influence of heat treatment routes on the corrosion behavior of 2205 duplex stainless steel was investigated. Two different solution heat treatments were applied to the as-received sample. First, both samples were heated at 1200 °C for 1 h. Then, one of them was taken from the furnace and cooled in air whereas the other was kept in the furnace and cooled down to 970 °C at a cooling rate of 0.05 °C/s, followed by water quenching. The microstructure investigations were carried by optical microscope after applying standard metallographic procedure and electrolytic etching. The electrochemical corrosion tests were conducted by potentiodynamic polarization test in 3.5 NaCl solution at room temperature. The microstructure of the as-received sample consisted of disorderly distributed austenite and ferrite phases whereas the heat-treated samples exhibited much more ordered and equiaxed blocks of austenite and ferrite phases. The electrochemical corrosion tests results showed that pitting corrosion resistance of 2225 duplex stainless steel was improved by solution heat treatment. The heat-treated sample that quenched from 970 °C exhibited higher pitting potential than the air-cooled sample. The pitting corrosion resistance of the 2225 duplex stainless steels was determined in the following order: As-received < Air-cooled < Quenched.

Keywords: Duplex stainless steel, Heat treatment, Microstructure, Corrosion, Pitting

# **1. Introduction**

Duplex stainless steels (DSS) are composed of austenite and ferrite phases and thus, they possess the properties of both ferritic and austenitic stainless steels [1]. They exhibit excellent combination of mechanical and corrosion properties. Generally, the optimal properties can be obtained when austenite and ferrite phases present equally in the microstructure, allied with no presence of third phase [2,3]. In recent years, DSSs have been extensively used in various applications, mainly in marine and oil-gas applications [3].

Although DSSs display a good corrosion resistance, when they are exposed to high chloride environment at high temperatures, they still suffer from pitting corrosion. A recent study revealed that the critical pitting temperature of 2205 DSS is between 40-45  $\Box$ C, regardless of the concentration of NaCl [4]. Numerous studies also investigated the critical potential breakdown of the passivation films and pitting corrosion of 2205 DSSs in various chloride solutions [5–7]. However, few studies have

focused on the effect of heat treatment on the corrosion properties of 2205 DSS. Luo et al. [8] reported that solution heat treatment improved the stability of passive film and decreased the precursor areas for pitting formation, resulting in an improvement on the pitting corrosion resistance of 2205 DSS. Naghizadeh and Moayed [9] showed that solution annealing at 1250 °C resulted in a 8 °C fall in the critical pitting temperature of 2205 DSS.

The International Conference of Materials and Engineering

In this paper, the effects of heat treatment routes on corrosion resistance of 2205 DSS were investigated in 3.5 % NaCl solution. The relationship between the commercially used and heat-treated 2205 DSS (air-cooled and quenched) was clarified.

# 2. Materials and Methods

The 2205 duplex stainless steel was supplied from the market as sheet form. The as-received DSS was produced by combination of casting, hot rolling and cold rolling. Samples with a dimension of 10 mm x 10 mm x 3 mm were cut for metallographic and corrosion characterizations. Two different solution heat treatments were applied to the as-received sample. Firstly, both samples were heated at 1200 °C for 1 h. Then, one of them was taken from the furnace and cooled in air whereas the other was kept in the furnace and cooled down to 970 °C at a cooling rate of 0.05 °C/s, followed by water quenching.

For microstructural investigations, all samples were mechanically ground with 240, 400, 600, 800, 1000, 1200 and 2000 grit emery papers followed by polishing with 6  $\mu$ m and 1  $\mu$ m diamond paste. Then, the samples were electrolically etched in a mixture of 40 gr NaOH and 100 mL distilled water at 2 V for 10 s.

The samples for corrosion test were mounted in epoxy resin with brass wire connection. The electrochemical testing system consisted of a graphite rod as counter electrode, a saturated calomel electrode (SCE) as reference electrode and the sample with exposed area of 0.25 cm<sup>2</sup> as working electrode in a working solution of 3.5% NaCl. The polarization curves were obtained by Gamry model PC4/300 mA potentiostat/galvanostat with DC105 corrosion analysis controlled by a computer. After monitoring open circuit potential for 600 s, the specimens were polarized from -0.5 V to +1.5 V with respect to  $E_{OC}$ . The potential value where a sharp increase in the current density occurred, was used to determine the pitting corrosion potential.

# **3. Results and Discussion**

Fig. 1 illustrates the optical micrographs of the as-received and heat-treated steels. The austenitic and ferritic phases are designated as A and F, respectively in the figures. It can be seen that the microstructure of the as-received sample consisted of disorderly distributed and elongated (caused by rolling) austenite and ferrite phases. However, heat-treated samples displayed much more ordered and equiaxed blocks of austenite and ferrite phases. Additionally, the air-cooled sample several tiny austenite phases having complex geometry with sharp corners. This formation of austenite phases was not observed in the quenched sample. This is because the quenching process prevented further phase transformation and maintained the microstructure at 970 °C.

Technology



Figure 1 Optical micrographs of 2205 DSS (a,b) as-received, (c,d) air-cooled from 1200 °C and (e,f) quenched from 970 °C.

Fig. 2 shows the potentiodynamic polarization curves of the as-received and heat-treated 2205 DSSs. In the anodic region, all the samples exhibited similar corrosion potential and a passive region, following a sharp increase in their current densities, indicating that pitting corrosion occurred at high voltages. The passive region lasted until 300-400 mV. However, above this potential values, breakdown of austenite passivation occurred and austenite phase started to exhibit pitting corrosion, while the ferrite was still passive. As the potential was increased further, ferrite phase started to show pitting. The potential values at the starting point of ferrite pitting corrosion changed after heat treatment. The heat-treated samples showed ferrite pitting at higher potential values than that of as-received sample. The approximate pitting potentials of the as received, air-cooled and quenched samples were found as follow, respectively, 1114, 1197 and 1220 mV, indicating a gradual pitting corrosion resistance. Similar findings have been reported elsewhere [1].

489

The potentiodynamic test results indicated that the protective passive film on the as-received sample was weaker than the solution heat-treated samples. Furthermore, the quenched sample showed the most stable passive film and better pitting corrosion resistance although the potential values were similar to the air-cooled sample. It has been reported that the pitting mostly nucleated on the interface of austenite and ferrite [1, 2]. As shown in Fig. 1, the as-received sample have much larger austenite and ferrite interface due to the complex geometry of the constituent phases. Therefore, it can be deduced that the more pitting occurred on these interfaces and reduced overall pitting corrosion potential. Besides, the preserved microstructure and more equiaxed blocks of phases in the quenched sample likely provided lower pitting nucleation sites and improved the pitting corrosion resistance. Fig. 3 shows the electrochemical impedance spectroscopy results of the studied samples. It can be seen that none of the alloys exhibited a complete capacitive loop, in which very small differences were observed. This indicated that the alloys showed similar corrosion mechanisms.

The International



Figure 2 Potentiodynamic polarization curves of the as-received and heat-treated 2205 DSS.

Technology





# 4. Conclusions

The microstructure and potentiodynamic polarization tests of the as-received, air-cooled and quenched samples were investigated in this study. The microstructure of the as-received sample consisted of disorderly distributed austenite and ferrite phases whereas the heat-treated samples exhibited much more ordered and equiaxed blocks of austenite and ferrite phases. The pitting corrosion resistance of the steels was determined in the following order: As-received < Air-cooled < Quenched.

# References

- 1 Gholami, M.; Hoseinpoor, M.; Moayed, M. H. A Statistical Study on the Effect of Annealing Temperature on Pitting Corrosion Resistance of 2205 Duplex Stainless Steel. Corrosion Science, **2015**, 94, 156–164. https://doi.org/10.1016/j.corsci.2015.01.054.
- Chen, T. H.; Yang, J. R. Effects of Solution Treatment and Continuous Cooling on σ-Phase Precipitation in a 2205 Duplex Stainless Steel. Materials Science and Engineering: A, 2001, 311 (1), 28–41. https://doi.org/10.1016/S0921-5093(01)00911-X.
- Haghdadi, N.; Laleh, M.; Kosari, A.; Moayed, M. H.; Cizek, P.; Hodgson, P. D.; Beladi, H. The Effect of Phase Transformation Route on the Intergranular Corrosion Susceptibility of 2205 Duplex Stainless Steel. Materials Letters, 2019, 238, 26–30. https://doi.org/10.1016/j.matlet.2018.11.143.
- 4 Liu, C.; Gong, M.; Xingwen, Z. Pitting Corrosion of 2205 Duplex Stainless Steel at High Concentrations of NaCl Solution. International Journal of Electrochemical Science, **2018**, 13, 7432–7441. https://doi.org/10.20964/2018.08.41.

Technology



The International Conference of Materials and Engineering Technology

- 6 Deng, B.; Jiang, Y.; Gong, J.; Zhong, C.; Gao, J.; Li, J. Critical Pitting and Repassivation Temperatures for Duplex Stainless Steel in Chloride Solutions. Electrochimica Acta, 2008, 53 (16), 5220–5225. https://doi.org/10.1016/j.electacta.2008.02.047.
- 7 Peguet, L.; Gaugain, A.; Dussart, C.; Malki, B.; Baroux, B. Statistical Study of the Critical Pitting Temperature of 22-05 Duplex Stainless Steel. Corrosion Science, 2012, 60, 280–283. https://doi.org/10.1016/j.corsci.2012.03.025.
- Naghizadeh, M.; Moayed, M. H. Investigation of the Effect of Solution Annealing Temperature on Critical Pitting Temperature of 2205 Duplex Stainless Steel by Measuring Pit Solution Chemistry. Corrosion Science, 2015, 94, 179–189. https://doi.org/10.1016/j.corsci.2015.01.051.
- 10 Hussain, E. A. M.; Robinson, M. J. Erosion–Corrosion of 2205 Duplex Stainless Steel in Flowing Seawater Containing Sand Particles. Corrosion Science, **2007**, 49 (4), 1737–1754. https://doi.org/10.1016/j.corsci.2006.08.023.

# EFFECT OF DIFFERENT INTERMETALLIC COMPOUNDS ON CORROSION RESISTANCE OF Mg-AI MAGNESIUM ALLOYS

The International Conference

aterials and Engineering Technology

# **HUSEYIN ZENGIN<sup>\*1</sup>**

<sup>1</sup>Karabuk University, Engineering Faculty, Department of Metallurgical and Materials Engineering, Karabuk, TURKEY.

#### Abstract

This study aims to investigate the effects of different intermetallic compounds which provided by adding different alloying elements on corrosion properties of Mg-4Al (wt%) magnesium alloys. Strontium (Sr), silicon (Si) and a mixture of cerium (Ce) and lanthanum (La) were employed separately at 1 wt% to ensure formation of intermetallic compounds. These alloying elements were selected in this study due to their known positive effects on the improvement of mechanical properties especially at elevated temperature. The alloys were melted by induction furnace under controlled argon gas atmosphere. A preheated steel mould was used to cast the alloys. The microstructure characterizations were conducted by optical and SEM microscopies. Immersion and electrochemical corrosion tests were carried out for corrosion investigations. Microstructure characterizations showed that Mg-4Al alloy contained divorced Mg<sub>17</sub>Al<sub>12</sub> intermetallic phase whereas after the additions of Ce/La, Sr and Si, the intermetallic compounds were mostly seen as Al<sub>11</sub>(Ce,La)<sub>3</sub>, Al<sub>4</sub>Sr, and Mg<sub>2</sub>Si, respectively. Both immersion and electrochemical corrosion tests revealed that all the alloying elements deteriorated the corrosion resistance of Mg-4Al alloy. However, the detected intermetallic phases showed different degree of corrosion reduction depending on the alloying elements. The detrimental effect of these elements decreased in the following order: Ce/La, Sr and Si.

Keywords: Magnesium alloys, Microstructure, Corrosion, Electrochemistry

# **1. Introduction**

Mg-Al alloys are considered as the most commercially used magnesium alloys due to their good combination of strength and ductility [1]. Al, which has a high solid solubility in Mg (~12 wt%) can provide a very effective solid solution strengthening [2, 3]. On the other hand, Al-containing Mg alloys are prone to show low strength values at elevated temperatures due to the formation of  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> phase with low thermal stability [4]. Therefore, many research studies have focused on employing new alloying elements with low solid solubility in Mg and accordingly, new type of magnesium alloys containing thermally stable intermetallic phases have been developed. In this regard, Mg-Al-RE (RE: rare earth elements), Mg-Al-Sr and Mg-Al-Si alloy systems, also denoted as AE, AJ and AS, respectively, have come to the forefront since they possess high strength at elevated temperatures. For example, Bai et al. [5] showed that La addition improved the mechanical properties of Mg-4Al alloy at both room and elevated temperatures due to the formation of Al<sub>11</sub>La<sub>3</sub> intermetallic phase. Another study showed that a mixture of Ce/La addition to AM60 magnesium alloy improved the corrosion and wear resistance [6]. It is widely agreed that the formation of Mg<sub>2</sub>Si intermetallic in



the Mg-Al-Si alloy system is the main reason for the improvement of strength at elevated temperatures. Blum et al. [7] reported that Mg-Al-Si alloys displayed the best creep resistance among the Mg-Al-Zn, Mg-Al-Mn and Mg-Al-RE systems due to the thermally stable Chinese script-like Mg<sub>2</sub>Si phases. Additionally, in recent years, new types of high creep-resistant Mg-Al-Sr based magnesium alloys, such as AJ52 and AJ62 have also been proposed [8]. Sr and Al form thermally stable Al4Sr phase, together with Mg,Al-Sr binary intermetallics, which are considered as the reason of the improvement of strength.

The International Conference of Materials and Engineering Technology

Many studies have been carried on Mg-Al based alloys with alloying elements that have high tendency to form hard second phases so far. Furthermore, these studies have mainly investigated the mechanical properties at room and elevated temperatures. However, very limited studies have focused on the corrosion properties of the Mg-Al based alloys, containing additions of 1 wt% Ce/La, Sr and Si. In the previous study, we investigated the mechanical properties of these alloys. Thus, the aim of the present study is to clarify the effects of these alloying elements on the corrosion properties of Mg-4Al alloy.

# 2. Materials and Methods

The Mg-4Al, Mg-4Al-1Ce/La, Mg-4Al-1Si and Mg-4Al-1Sr (wt%) alloys were produced by permanent mould casting. Pure Mg and Al ingots and Al-30Si, Al-15Sr, Mg-30Ce and Mg-30La master alloys were melted in an induction furnace under controlled  $CO_2+1\%SF_6$  mixture gas. After the melt was stirred at 750 °C for 2 min by a mechanical stirrer and held at this temperature for 10 min, it was poured into a steel mould. Finally, the cast ingots having a diameter of 42 mm and a length of 320 mm were obtained. Microstructure characterizations were carried out by scanning electron microscopy (SEM). The metallographic samples were prepared by mechanical grinding and polishing. After the samples were etched with 2% nital solution.

The corrosion tests were conducted by constant immersion and electrochemical polarization tests. For the immersion corrosion test, the samples were cut from the half of the radius of the samples, followed by grinding and polishing. After that, the samples were immersed in 3.5 wt% NaCl solution at room temperature for 60 h. The corrosion products were removed by chromic acid solution. The weight loss measurements were made and the corrosion rates were calculated. The electrochemical corrosion tests were carried out by a Gamry model PC4/300 mA potentiostat/galvanostat with DC105 corrosion analysis at a scan rate of 1 mVs-1, starting from -0.25 V (vs. Eoc ) to +0.25 V (vs. Eoc ). The obtained polarization curves were used to estimate the corrosion current densities ( $i_{corr}$ ), and corrosion potentials ( $E_{corr}$ ) by Tafel extrapolation method. A classical three-electrode cell with a graphite rod as counter electrode, a saturated calomel electrode (SCE) as reference electrode and the sample as working electrode was used for the electrochemical corrosion tests.

# **3. Results and Discussion**

Fig. 1 shows the SEM micrographs of the alloys. All the microstructures consisted of intermetallic phases (white) with different morphologies and  $\alpha$ -Mg matrix phase (dark). Mg-4Al alloy contained low amount of divorced and globular-like intermetallic phases. However, after the addition of 1 wt%

alloying elements, the fraction of intermetallic phases considerably increased. This indicated that the alloying elements resulted in a formation of different intermetallic phases. It can be seen that the Ce/La addition led to formation of discontinuous strip-like second phases. Sr addition also resulted in a formation of islands of rods and globular-like second phases, in which some of them constituted a blocky shape. Furthermore, Si addition gave rise to a formation of very uniquely shaped second phase particles similar to Chinese-script shape. In the literature, similar findings were reported by numerous studies, which identified as Chinese script Mg<sub>2</sub>Si phase. In our previous studies [9, 10], the XRD and SEM characterizations with EDX analysis of the present alloys revealed that the second phase particles in the Mg-4Al alloy corresponded to  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> intermetallic phase. The strip-like second phase particles in the Mg-4Al-1Ce/La alloy accounted for Al<sub>11</sub>(Ce,La)<sub>3</sub>. On the other hand, the second phase particles in the Mg-4Al-1Sr alloy consisted of Al<sub>4</sub>Sr and (Mg,Al)<sub>17</sub>Sr<sub>2</sub> intermetallic phases. Thus, it can be deduced that the alloying elements of Ce/La mixture, Sr and Si showed a significant tendency to form new intermetallic phases in Mg-Al based magnesium alloys and these intermetallics can play a remarkable role on the both mechanical and corrosion properties.

The International Conference

aterials and Engineering Technology



**Figure 1** SEM micrographs of the alloys: (a) Mg-4Al, (b) Mg-4Al-1Ce/La, (c) Mg-4Al-1Sr and (d) Mg-4Al-1Si.

Fig. 2 presents the immersion corrosion test results of the alloys. It can be seen that the Mg-4Al alloy showed the lowest corrosion rate among the studied alloys. Thus, it can be inferred that the formation of thermally stable second phase particles in the Mg-4Al alloy considerably deteriorated the corrosion resistance. Mg-4Al-1Si alloy exhibited the second-best corrosion performance among the studied alloys. According to our previous study on the mechanical properties of the present alloys [9], Mg-4Al-Si alloy exhibited the best mechanical properties at both room and elevated temperatures. Based on that result and considering there is a low difference between the corrosion rates of Mg-4Al and Mg-4Al-1Si alloys, the deteriorated corrosion performance after 1 wt% Si addition can be admissible.



However, it can be seen in Fig. 2 that the Mg-4Al-1Ce/La alloy showed the poorest corrosion resistance and the corrosion rate of Mg-4Al-1Sr alloy doubled that of Mg-4Al alloy. This indicates that the intermetallic phases in the Mg-4Al-1Ce/La and the Mg-4Al-1Sr alloys led to an increase in the corrosion kinetics. The coarser Al<sub>11</sub>(Ce,La)<sub>3</sub>, Al<sub>4</sub>Sr and (Mg,Al)<sub>17</sub>Sr<sub>2</sub> intermetallic phases created higher microgalvanic effect between the intermetallics and the matrix phases. Therefore, these alloys exhibited poor corrosion resistance. On the other hand, it was reported that  $\alpha$ -Mg and Mg<sub>2</sub>Si phase had the same corrosion potential values of -1.65 V [11]. Hence, the Chinese script Mg<sub>2</sub>Si phase did not show a strong microgalvanic effect and accordingly slightly reduced the corrosion resistance of the Mg-4Al alloy. Fig. 3 shows the SEM micrographs of the alloys after the immersion tests. It can be seen that Mg-4Al-1Ce/La and Mg-4Al-1Sr alloys showed much deeper corrosion pits on their surfaces whereas the corrosion attacks were not strong in the Mg-4Al and Mg-4Al-1Si alloys.





Figure 3 SEM micrographs after the immersion tests for 60 h of the alloys: (a) Mg-4Al, (b) Mg-4Al-1Ce/La, (c) Mg-4Al-

Fig. 4 represents the potentiodynamic polarization curves of the alloys. The corrosion current densities  $(i_{corr})$ , corrosion potentials  $(E_{corr})$  and corrosion rates (mm/y) of the alloys are also given in Table 1. It can be seen that all the alloy showed the similar trend in their cathodic and anodic branches with increased cathodic kinetics during the initial stages of the corrosion. The  $i_{corr}$  values and the corrosion rates in mm/y, which calculated from the  $i_{corr}$  values, indicated the lowest corrosion resistance was found for the Mg-4Al-1Sr alloy, which almost doubled the other alloys. All the other alloys exhibited similar values of corrosion rates. This indicated that Sr addition gave rise to a significantly high cathodic corrosion kinetics, which is the main reason of higher  $i_{corr}$  values.



Figure 4 Potentiodynamic polarization curves of the alloys.

Sample	icorr	Ecorr	Corrosion Rate
	$(\mu A/cm^2)$	(V)	(mm/y)
Mg-4Al	129	-1.32	2.9
Mg-4Al-1Ce/La	110	-1.25	2.5
Mg-4Al-1Sr	219	-1.25	5
Mg-4Al-1Si	115	-1.32	2.6

Table 1 Electrochemical corrosion test results of the alloys.

# 4. Conclusions

The microstructure of Mg-4Al alloy consisted of  $\alpha$ -Mg matrix phase and divorced Mg<sub>17</sub>Al<sub>12</sub> intermetallic phase while after the additions of Ce/La, Sr and Si, the intermetallic compounds were mostly seen as Al<sub>11</sub>(Ce,La)<sub>3</sub>, Al<sub>4</sub>Sr, and Mg<sub>2</sub>Si, respectively. Immersion and electrochemical corrosion tests showed that the corrosion resistance of Mg-4Al alloy was deteriorated by the additions of alloying elements. The second phase particles displayed different degree of reduction in the corrosion rate depending on the type of alloying elements. The detrimental effect of these elements decreased in the following order: Ce/La, Sr and Si.

Technology



1 Bamberger, M.; Dehm, G. Trends in the Development of New Mg Alloys. *Annu. Rev. Mater. Res.*, **2008**, *38* (1), 505–533. https://doi.org/10.1146/annurev.matsci.020408.133717.

The International Conference of Materials and Engineering Technology

- 2 Dahle, A. K.; Lee, Y. C.; Nave, M. D.; Schaffer, P. L.; StJohn, D. H. Development of the As-Cast Microstructure in Magnesium–Aluminium Alloys. *J. Light Met.*, 2001, *1* (1), 61–72. https://doi.org/10.1016/S1471-5317(00)00007-9.
- 3 Zengin, H.; Turan, M. E.; Turen, Y.; Ahlatçi, H.; Sun, Y. Influence of Titanium Addition on Wear Properties of AM60 Magnesium Alloy. *Int. J. Mater. Metall. Eng.*, **2016**, *10* (6), 767–771.
- 4 Hort, N.; Huang, Y.; Kainer, K. U. Intermetallics in Magnesium Alloys. *Adv. Eng. Mater.*, 2006, 8 (4), 235–240. https://doi.org/10.1002/adem.200500202.
- 5 Bai, J.; Sun, Y.; Xue, F.; Qiang, J. Microstructures and Creep Properties of Mg–4Al–(1–4) La Alloys Produced by Different Casting Techniques. *Mater. Sci. Eng. A*, **2012**, *552*, 472–480. https://doi.org/10.1016/j.msea.2012.05.072.
- 6 Chenghao, L.; Shusen, W.; Naibao, H.; Zhihong, Z.; Shuchun, Z.; Jing, R. Effects of Lanthanum and Cerium Mixed Rare Earth Metal on Abrasion and Corrosion Resistance of AM60 Magnesium Alloy. *Rare Met. Mater. Eng.*, **2015**, *44* (3), 521–526. https://doi.org/10.1016/S1875-5372(15)30031-X.
- Blum, W.; Zhang, P.; Watzinger, B.; Grossmann, B. v; Haldenwanger, H. G. Comparative Study of Creep of the Die-Cast Mg-Alloys AZ91, AS21, AS41, AM60 and AE42. *Mater. Sci. Eng. A*, 2001, *319–321*, 735–740. https://doi.org/10.1016/S0921-5093(00)02016-5.
- Baril, E.; Labelle, P.; Pekguleryuz, M. Elevated Temperature Mg-Al-Sr: Creep Resistance, Mechanical Properties, and Microstructure. *JOM*, 2003, 55 (11), 34–39. https://doi.org/10.1007/s11837-003-0207-7.
- 9 Zengin, H.; Turen, Y.; Elen, L. A Comparative Study on Microstructure, Mechanical and Tribological Properties of A4, AE41, AS41 and AJ41 Magnesium Alloys. *J. Mater. Eng. Perform.*, **2019**, *28* (8), 4647–4657. https://doi.org/10.1007/s11665-019-04223-8.
- 10 Zengin, H.; Turen, Y.; Ahlatci, H.; Sun, Y.; Karaoğlanli, A. C. Influence of Sn Addition on Microstructure and Corrosion Resistance of AS21 Magnesium Alloy. *Trans. Nonferrous Met. Soc. China*, **2019**, *29* (7), 1413–1423. https://doi.org/10.1016/S1003-6326(19)65048-X.
- 11 Song, G.; Atrens, A. Understanding Magnesium Corrosion—A Framework for Improved Alloy Performance. *Adv. Eng. Mater.*, **2003**, *5* (12), 837–858. https://doi.org/10.1002/adem.200310405.

# DESIGN AND ANALYSIS OF MANHOLE COVER FOR TRAILERS

The International Conference of Materials and Engineering Technology

# ISMAIL BOGREKCI<sup>\*1</sup>, PINAR DEMIRCIOGLU<sup>1</sup>, DECAN ALPARGUN<sup>2</sup>, EMRE CEYLAN<sup>2</sup>, KADİR BOZKURT<sup>2</sup>

<sup>1</sup> Aydin Adnan Menderes University, Engineering Faculty, Mechanical Engineering Department, Aydin, TURKEY. <sup>2</sup> Ok Kardesler Treyler San. ve Tic. A.S., R&D Center, Aydin, TURKEY.

#### Abstract

Loading and unloading for trailers are carried out using manhole covers. The traditional designs of the manhole covers are far behind when the potential efficiency and ergonomic advancements are considered. Design improvements in leakproofness and ergonomy are crucial. Design and manufacturing approach are implemented traditionally, when conventional manhole covers are designed and manufactured. Conventional manhole covers are tested in this type of manufacturing cycle until the final product specifications are satisfactory. In this study, a manhole cover for trailers was designed, analyzed, manufactured and tested in an attempt to address efficiency and ergonomic shortcomings of conventional versions. Finite Element Analysis was implemented additionally to specified manufacturing cycle so as to optimize the design and reduce the manufacturing cost.

Solid state modelling was utilized to design manhole cover in 3D by SolidWorks CAD software. FEA was used in order to investigate the stressed, strains, displacements and safety factor of relevant critical sections. Improved manhole covers were manufactured using both casting and machining along with two testing rigs in order to test the performance of manhole covers in terms of leakproofness and durability under operational conditions. The test results indicated that manhole covers did not suffer from any leakage during experimental analyses and no deformations were formed up to 3.5 bars of pressure. However, when the pressure reached 4 bars, the cast manhole cover exhibited cracks.

Keyword: Manholes, Manhole Cover, Design, FEA Analysis, Casting.

# **1. Introduction**

Dry bulk carrier semi-trailer is used for transporting solid materials. Dry bulk carrier semi-trailer vehicles carrying materials such as flour, grain, silica sand, powder cement are generally filled from the top of the vehicles via filling caps. This filling opening is named 'manhole' conventionally and the lid covering the manhole is called 'manhole cover'. Manhole cover is considered to be the most important component for trailer structure due to the vital duty of preventing overflow along with



The International Conference of Materials and Engineering Technology

In this article, characteristics of manhole cover are examined in order to propose an improved design. Firstly, benchmarking was implemented with already available manhole covers, literature search was carried out following the benchmarking process and an improved manhole cover was designed utilizing 3D CAD software, finally the prototype production was completed. Hydraulic pressure and leakproofness tests were performed in addition to finite element analyses in order to examine the behavior of the manhole cover design.

The Finite Element Analysis (FEA) or called Finite Element Method (FEM) at times is a numerical technique which is addressed to obtain approximate results for more complex geometries or field applications. FEA can be described as dividing complex structures with excessive contours into a set of certain tiny structures or elements. The aforementioned ability is generally referred as meshing or discretization, making analyses of irregular, delicate or complex geometries solvable. Rather than numerical solutions, this type of approach is considered to be more effective in analyzing a variety of engineering problems [1-4].

This study inspected a rare kind of manhole cover designed for special utility tanks or namely trailers. Manhole covers are considered mostly for sewers or relevant piping applications in the literature. Structural strength, alternative materials and design along with various analysis methods are expressed referring to manhole covers.

Park's study investigated steel manholes with height adjusting property in order to replace traditional cast iron manhole covers. Their main motivation was to match the upper surface of the manhole with the relevant surface for convenient utility and improved safety. In addition to aforementioned criterion, performance of the steel manhole cover was inspected through ANSYS software along with experimental load tests. Under 450kN of force, authors claimed a deformation of maximum 2.7mm without any residual deformation due to inexertion of the forces [5].

Jinbao et al expressed manhole cover process technology including structural durability, material choice and forming methods Primary purpose of the authors was to inspect the defects and appropriate injection variables of the process. Resin characteristics and formation of defects were evaluated with respect to flow parameters [6].

Li et al studied the overload issues of manhole covers considering failure causes. In order to improve the maximum load carrying capacity, analyses were carried out utilizing a special type of software called I-DEAS. The safety of the design would be ascended as well according to the authors. Production technology besides type of cover is among optimization variables for their study. As a result the authors claimed that the strengthening of manhole covers was possible with their approach especially for certain type of manhole covers [7]. Li and Sun concentrated on the structure of manholes, and the manhole inspected by the authors was made out of polymer matrix composites. The composite manhole was investigated utilizing ANSYS software in order to assess static performance. The authors proposed that according to the results, practical implementation for real life conditions was possible as the basic requirements were satisfied. Cost efficiency was evaluated in their study as well [8].

The International Conference of Materials and Engineering Technology

Tijsseling et al described the characteristics and physics of moving manholes under harsh environmental conditions such as heavy rainfall or flood. This type of phenomena was named as dancing manhole cover and the authors underlined the dangers posed by this movement as well. Assessment of the physics of this phenomenon was attempted utilizing simple spring-mass-dashpot model. As a result, authors claimed that the driving mechanism behind dancing manhole cover phenomena was obtained by a rather simplified mathematical model [9].

Saldarriaga et al researched the basic behavior junction manholes of sewers under supercritical flow conditions. Different parameters such as Froude number and filling ratio were tested with an implemented physical model. Wave formations at both ends of the stream along with structural patterns were evaluated in their study. This particular flow type was expressed in detail and wave height, wave types and forming conditions were inspected in order to better understand hydraulic behavior of similar applications [10].

# 2. Materials and Methods

In the benchmarking study, it was observed that the clamping arms of the manhole covers used in the current situation were made with the help of passes and it was necessary to turn the passes several times in order to open and close the manhole cover. It has also been observed that the gasket that prevents material from escaping from inside the vehicle is parallel to the surface and may cause leakage. A new design was developed and a product was developed to minimize the errors seen in benchmarking. In the literature, this type of locking system is called cam & latch.

Conceptual design and three-dimensional modeling of the manhole cover was made using Solidworks. Modeling is done by working on clamping arms, gaskets and pressing surface angle to solve the problem of ergonomics and leakproofness. Static structural analysis was performed in order to see the stress behavior of the product design and to provide input to the design with Finite Element Analysis method. Mesh was formed on the geometry of the manhole cover and neckring. Mechanical properties of Etial 171 material were defined. Hydrostatic pressure was applied considering the internal test pressure 3Bar. As a result of the applied pressure, the strength of the cover is evaluated by looking at the yield stress.



Figure 1. The Manhole Cover

The prototype was produced by casting and applying machining processes. Hydraulic pressure and leakproofness tests were performed on the prototype and the behavior of the prototype manhole cover was examined.

According to the calculations based on EN 13445-5 Unfired Pressure Vessels Part5: Inspection and Testing standard, a minimum 3Bar pressure was applied to the manhole cover placed on the test bench. Worst case 3.5Bar was determined. The applied pressure lasted 30 minutes. In this way, it was examined how the pressure inside the vehicle affects the cover. The test was carried out on the hydrostatic test device, not on the vehicle. This makes it possible to test without causing any security problems.

Material leakproofness at the edge of the manhole cover was examined by applying pressure between 3 and 3.5Bar written in the literature. In this test, the test apparatus used for the hydraulic pressure test was used and after the pressure was applied, the manhole cover was checked with eye and material leakage from the manhole cover was examined.

# **3. Results and Discussion**

In this study, especially the manhole cover used in the dry bulk carrier semi-trailer has been handled, modeled in Solidworks, structural analysis has been made, prototype production has been made and then subjected to hydraulic pressure and leakproofness test.

The analysis was completed in Solidworks Simulation. The results were obtained by applying the above-mentioned boundary conditions. The results were evaluated in von Mises stress distribution (Figure 2). The criteria were determined as 75% of the yield strength of the material. When the results are examined, the design is safe and durable since the stress values are below the passing criteria, yield stress.



Figure 2. The Manhole Cover Stress Distribution-Von Mises Stress

In the pressure test (Figures 3 and 4), the pressure applied was increased from 3Bar to 4.5Bar and the behavior of the manhole cover under this pressure was examined. No deformation was observed up to the standard 3.5Bar. When the pressure of 4.5Bar was applied, the manhole cover was broken. Although our lowest case value is 3.5Bar, the manhole cover is broken at 4.5Bar. These results show that the strength of the manhole cover is quite high. Likewise, there was no nonconformity in the leak test.



Figure 3. The Manhole Cover Testing



Figure 4. The Manhole Cover Testing Pressure Setup

**4. Conclusions:** Manhole cover design is conducted. The manhole cover design is evaluated using FEA analysis. Manhole cover is produced using both casting and machining. The prototype manhole cover was tested. The results from FEA analyses and tests indicated that the designed manhole cover is strong enough in terms of strength and leakage free.

The International Conference of Materials and Engineering Technology

**Acknowledgments:** The authors warmly acknowledge the funding, support and contributions of OK Kardeşler Trailers Inc. and its R&D Center. Also Authors appreciate the support of Mechanical Engineering Department, Engineering Faculty at Aydin Adnan Menderes University.

# **References:**

- 1. Madenci, E., and Guven, I. The Finite Element Method and Applications in Engineering Using ANSYS. New York, USA: Springer Science+Busness Media, LLC, 2007.
- 2. Klein, M., Thorenz, B., Lehmann, C., Boehner, J., Steinhilper, R., Integrating New Technologies and Materials by Reengineering: Selected Case Study Results. Procedia CIRP, Vol 50, 2016, 147-152.
- 3. Uchibori, D., Ashikaga, S., Deguchi, T., Yanagi, S. Development of a Manhole Cover with Pattern for Improved Durability and Visibility of Worn Condition. DOI: 10.2208/jscejcei.75.1, 2019.
- 4. Ch, K., HPark, J.H. Development of a height-adjustable manhole. Journal of Achievements in Materials and Manufacturing Engineering, 2006, Vol 14, Issue 1-2.
- 5. Park, W.C. The Development of Height Adjustable Steel Manhole Cover. Journal of the Korea Academia-Industrial cooperation Society, 2018, 19(9): 581-586.
- 6. Jinbao, L., Shaoqin, Z. and Lili, C. Application of RTM Process in FRP Inspection Manhole Lid, Fiber Reinforced Plastics/Composite, 2003-02.
- 7. Li, H.P., Liang, C.Y. and Qi, M. The Structure Optimization Design of Manhole-cover with Software I-DEAS", Journal of Hebei University of Technology, 2005-06.
- 8. Li, S. and Sun, K. The Finite Element Analysis and Configuration Optimization on Polymer Matrix Composites Manhole Cover. China Resources Comprehensive Utilization, 2008-03.
- 9. Tijsseling, A. S., Vasconcelos, J. G., Hou, Q. and Bozkuş, Z. Dancing Manhole Cover: A Nonlinear Spring-Mass System. Proceedings of the ASME 2019 Pressure Vessels and Piping Division Conference PVP2019, July 14-19, TX, USA, 2019.
- 10. Saldarriaga, J., Bermudez, N. and Rubio, D. P. Hydraulic Behavior of Junction Manholes under Supercritical Flow Conditions. Journal of Hydraulic Research, 2012, 50(6): 631-636.



'he International Conference of Materials and Engineering Technology

# MIKAIL ASLAN<sup>1</sup>, ABDULAZIZ KAYA<sup>1</sup>, HALIL IBRAHIM KURT<sup>1</sup>, NECIP FAZIL YILMAZ<sup>2</sup>

<sup>1</sup>Metalurji ve Malzeme Mühendisliği Bölümü, Mühendislik Fakültesi, Gaziantep Üniversitesi, Gaziantep, TÜRKİYE. <sup>2</sup>Makine Mühendisliği Bölümü, Mühendislik Fakültesi, Gaziantep Üniversitesi, Gaziantep, TÜRKİYE.

#### Özet

Bu çalışmada, Aluminyum-Magnezyum oksit (MgO) kompozitler toz metalurjisi yöntemiyle üretilmiştir. Kompozitlerde MgO güçlendirici olarak ağırlıkça %10 oranında ilave edilmiş olup güçlendiricinin ortalama çapı <40 nm boyutundadır. Kompozitlerin mikroyapıları optik mikroskop ve taramalı elektron mikroskobu aracılığıyla incelenmiştir. Kompozitlerin teorik yoğunlukları karışım kanununa göre ölçülen yoğunlukları Arşimed prensibine göre araştırılmıştır. Teorik yoğunluk değerleri ve ölçülen yoğunluk değerleri kullanılarak ürünlerin porozite içeriği belirlenmiştir. Ayrıca kompozitlere sertlik testi uygulanarak mekanik özellikleri araştırılmıştır.

Anahtar Kelimeler: Al, Kompozit, MgO, Toz metalurjisi

#### 1. Giriş

Kompozitler, başka şekilde elde edilemeyen özelliklerin bir kombinasyonunu elde etmek için iki veya daha fazla malzeme veya faz birlikte kullanıldığında üretilir. Sıra dışı sertlik, kuvvet, ağırlık, yüksek sıcaklık performansı, korozyon direnci veya iletkenlik kombinasyonları verecek şekilde kompozit malzemeler üretilebilirler. Kompozitler, ayrıca farklı malzemelerin sinerji içinde nasıl çalışabileceğini vurgulamaktadır. Makro ölçekte kompozit olan bir malzeme örneği çelik betonarmedir. Mikro ölçekte olan kompozitler arasında karbon veya cam elyaf takviyeli plastikler gibi malzemeler bulunur [1]. Birçok kompozit malzeme sadece iki fazdan oluşur; bunlardan biri sürekli olan ve genellikle dağınık faz olarak adlandırılan diğer fazı çevreleyen matris olarak adlandırılır. Kompozitlerin özellikleri, kurucu fazların özelliklerine, nispi miktarlarına ve dağınık fazın geometrisine bağlı olarak değişiklik gösterirler [2]. Dağılmış veya güçlendirici faz, lifler, partiküller veya diğer çeşitli geometriler halinde olabilirler. Fazlar genellikle birbirlerinde çözünmezler, ancak arayüzlerinde güçlü bir uyumluluk olması gerekir [3].

Metal matriks kompozitler (MMC), silisyum karbür veya grafit gibi bir seramik malzemenin parçacık veya lifleri ile güçlendirilmiş alüminyum veya magnezyum gibi düşük yoğunluklu bir metal matristen oluşur. Saf metallere kıyasla, MMC'ler daha yüksek spesifik güç ve sertlik sunmak; daha yüksek çalışma sıcaklığı ve aşınmaya karşı daha yüksek direnç ve bu özellikler belirli bir uygulamaya uyacak şekilde uyarlanabilirler [4]. Bu malzemelerin polimer-matris kompozitlere göre avantajlarından bazıları, daha yüksek çalışma sıcaklıkları, yanmazlık ve organik sıvılar tarafından bozulmaya karşı daha fazla direnç içermeleridir [2]. Bu tür kompozitlerin ideal özelliklere sahip olarak imal edilmesi için; işleme tekniğinin, takviye birleştirmesinin yüksek hacimli fraksiyonunu sağlaması, takviyenin

düzgün bir şekilde dağılması ve matris ile mekanik özellikleri bozan istenmeyen ara-yüz reaksiyonları olmadan takviye fazı arasında kabul edilebilir bir yapışmayı temin etmesi gerekir. Çeşitli alaşımlar ve takviye malzeme kombinasyonlarını kullanan MMC'yi sentezlemek için argon gazı altında ergiyik karıştırma/ vorteks yöntemi, toz metalurjisi, sızma (infiltrasyon), döküm vb. gibi bir dizi işleme tekniği geliştirilmiştir [5].

The International Conference of Materials and Engineering Technology

Mevcut çeşitli matris malzemeleri arasında, alüminyum ve alaşımları MMC'lerin üretiminde yaygın olarak kullanılmaktadır. Bunun nedeni alüminyum bazlı MMC'lerin hafif, ekonomik açıdan uygun, çeşitli işleme teknikleriyle üretime uygun ve yüksek mukavemet ve iyi korozyon direncine sahip olmasıdır. Alüminyum metal matris kompozitlerde kullanılan bazı önemli takviye malzemeleri parçacıklı, kılsı veya elyaf formunda olan karbon / grafit, silisyum karbür, alümina, zirkonya ve zirkondur [6].

Magnezyum oksit mekanik dayanımı, toksik olmaması ve antibakteriyel aktivitesi gibi değerli özellikleri sayesinde büyük ilgi çekmektedir. Yangın ve korozyona karşı yüksek direnciyle birlikte düşük hidrasyon hassasiyetine sahip olması, magnezyum oksite eşsiz özellikler kazandırır ve bu durum magnezyum oksitin çok çeşitli uygulamalarda kullanılmasına neden olur [7]. Magnezyum oksitin, bu özellikleri sayesinde alüminyum metal matris kompozitlerde kullanılabilecek uygun bir takviye malzemesidir.

Bu çalışmada, Al - MgO kompozit üretiminde karıştırma, sıkıştırma ve sinterleme işlemlerinden oluşan geleneksel toz metalurjisi tekniği uygulanmıştır ve elde edilen kompozitlerin mikroyapısı ve mekanik özellikleri incelenmiştir.

# 2. Malzeme ve Yöntem

Al-MgO kompozitleri, matris malzemesi olarak %99,80 saflıkta alüminyum ve takviye olarak MgO nanoparçacıkları kullanılarak toz metalürjisi yöntemiyle üretilmiştir. Alüminyum tozu parçacıkları ve magnezyum oksit nanopartikülleri ((Nanografi A.Ş.) tarafından tedarik edilmektedir.) Al - MgO nanokompozit için hammadde olarak kullanıldı.

Takviye ve matris tozlarını tarttıktan sonra, tozlar bir beherin içine konuldu ve karıştırma işlemi, 30 dakika boyunca etanol içerisinde manyetik bir karıştırıcı kullanılarak gerçekleştirildi. Elde edilen süspansiyon oda sıcaklığında etanolun uzaklaşması için yeterli süre bekletilerek toz karışımı tamamen kurutuldu. Ardından kompozitlerin mikroyapı, yoğunluk ve sertlik incelemelerini gerçekleştirmek amacıyla 30 mm çapındaki silindirik numuneler  $\approx$ 190 MPa presleme basıncında tek yönlü olarak preslenmiştir. Sinterleme sıcaklığının özelliklere etkisini incelemek için ağırlıkça %10 magnezyum oksit içeren kompozitler hazırlandı.

Preslenen numuneler farklı sıcaklıklarda (500, 550, ve 600°C) hava atmosferinde 5 saat boyunca sinterlenmiştir. Numunelerin kütle yoğunluğu, Arşimet yöntemi kullanılarak ölçüldü. Sinterlenen numunelerdeki sinterlemeyi ve faz dağılımını görebilmek için Optik Mikroskop ve Taramalı Elektron Mikroskobu ile incelemiştir. Sertlik ölçümleri Rockwell skalası kullanılarak ölçüldü ve sonuçlar Brinell skalasına çevrildi.

# 3. Bulgular ve Tartışma

Farlı derecedeki sinterleme sıcaklıklarının ağırlıkça %10 MgO nano parçacığı içeren AlMgO kompozitine etkisi Şekil 1 verilmiştir. Üretilen kompozit toz metalürji yöntemiyle üretildiğinden dolayı oluşan gözenek sayısı fazladır. Malzemede oluşan gözenekler malzemenin mekanik





Şekil 1. AlMgO kompozitlerin farklı sıcaklıklardaki sinterlenmiş mikro yapıları

Al-MgO metal matriks kompozitin mikroyapısal analizi SEM ile yapılmıştır ve Şekil 2 de faklı sinterleme sıcaklıklarında ki kompozitin SEM görüntüleri verilmiştir. Elde edilen sonuçlara göre sinterleme süresince yüzeyde bulunan MgO nanoparçacıkları malzemenin içerisine doğru yayılım göstermektedir. Bunun sonucu olarak MgO nanoparçacıkları özellikle yüzeyde homojen olarak dağılmamaktadır. Bunun nedeni MgO nanoparçacıklarının yüzey/hacim oranının ve özkütlesinin Al



mikro parçacıklarından büyük olmasından dolayı kaynaklanabilir çünkü spesifik yüzey alanındaki artış parçacıklar arasındaki sürtünmeyi artırarak parçacıkların homejen olarak dağılmasını engellemektedir. Şekil 2 de görüldüğü gibi ayrıca MgO parçacıklarının kümelenmesine veya topaklanmasına sebep olmaktadır. Diğer yandan toz metalurjisiyle elde edilen bu kompozit döküm yoluyla elde edileydi kümelenmenin daha fazla olacağını tahmin etmek zor değildir. SEM analizinden elde edilen diğer bir sonuç AlMgO kompozitinin sinterlemeden sonra yüzey pürüzlülüğü artmaktadır (şekil 2d' ye bakınız).

aterials and Engineering Technology



Şekil 2. AlMgO kompozitlerin değişik sıcaklıklardaki sinterleme sonucu SEM yapıları

Sinterlenme sıcaklığının özkütle ve gözeneklik üzerine etkisi Şekil 3-5' te verilmektedir. Beklenildiği gibi sinterleme sıcaklığının artmasıyla özkütle artmakta ve gözeneklik oranı azaltmaktadır çünkü sinterleme sıcaklığı atomik difüzyon hızını artırarak kompozitin ıslanabirliği ve sinterleme yeteneğini artırmaktadır. Bunun sonucu olarak kompozitteki gözenek oranları azalmıştır. Kompozitin yoğunluğundaki en küçük artış oranı 550 den 600 °C ye geçişte görüldü(Şekil 3' e bakınız). Sinterleme



sıcaklığının artması gözenek yüzde oranlarının azalmasına sebep olması özkütlenin doğrudan gözenek sayısı ile ilişkili olduğu bilinen bir gerçektir. Bu çalışmada aynı sonuca varılmıştır. Gözenek oranında en büyük azalma 500 den 550 °C ye geçişte görüldü(Şekil 4' e bakınız). Malzemenin gözenek oranını en aza indirmek için uygulanan sinterleme süresini dikkate alarak 600 °C en uygun sinterleme sıcaklıklarından biri olabileceği sonucuna varılmıştır. Ayrıca, göreceli özkütle değerleri hesaplandı(Şekil 5'e bakınız).



Şekil 3. AlMgO kompozitinin değişik sıcaklıklardaki sinterleme sonucu özkütleleri



Şekil 4. AlMgO kompozitinde sinterleme sıcaklığının gözenikliliğe etkisi.



Şekil 5. AlMgO kompozitinde sinterleme sıcaklığının göreceli yoğunluğa etkisi

Sinterleme sıcaklığının mekanik özellikler üzerine etkisi Şekil 6' da değerlendirilmiştir. Sinterleme işlemiyle gözenek oranının azalmasına rağmen mekanik özelliklerde bir iyileşme görülmemektedir. Al matriksin sertliğini artırıcı MgO nanoparçacıklarının yüzeyde daha önce belirtilen sebeplerden dolayı azalmasından ve yüzeyde mevcut olan MgO nanoparçacıklarının homojen dağılmamasından dolayı malzemenin sertliği sinterleme işlemiyle artmamaktadır. Bazı bölgelerde özellikle tane sınırları üzerinde görülen topaklanmalar sinterleme süresince tane sınırları arasında zayıf bağlara sebep olduğundan istenilen mekanik özellikler elde edilmemiştir. Ek olarak sinterleme süresi mekanik özelliklerin zayıflamasına sebep olmuş olabilir.



Şekil 6. AlMgO kompozitinde sinterleme sıcaklığının göreceli yoğunluğa etkisi

# 4. Sonuçlar

Bu çalışmada, karıştırma, sıkıştırma ve sinterleme işlemlerinden oluşan geleneksel toz metalurjisi tekniği uygulanarak Al-MgO kompozitleri üretildi. Takviye edici olarak MgO nanoparçacığı (ortalama çapı <40 nm) ağırlıkça %10 oranında ilave edildi. Kompozitlerin porozite oranları teorik

ve deneysel ölçümlerden elde edilmiştir. Ayrıca farklı sıcaklıklarda sinterlenme sonucu elde edilen AlMgO kompozitlerin sertlik değerleri hesaplanmıştır. Elde edilen sonuçlar aşağıda belirtilmiştir:

• Sinterlemeden önce üretilen kompozitte gözenek sayısı fazladır. Gözenek sayısının fazla olması MgO nanoparçacıkları ile Al tozları arasındaki erime noktası ve basınç dayanımının çok farklı olmasından dolayı kaynaklanmaktır. Seramik MgO parçacıkları yüksek gözenekliğe sebep olan parçacıkların düzenlenmesine, deformasyonuna ve difüzyonuna engel teşkil etmektedir.

The International Conference of Materials and Engineering Technology

- Sinterleme süresince yüzeyde bulunan MgO nanoparçacıkları malzemenin içerisine doğru yayılım göstermektedir. Bunun sonucu olarak MgO nanoparçacıkları özellikle yüzeyde homojen olarak dağılmamaktadır. Bunun nedeni MgO nanoparçacıklarının yüzey/hacim oranının ve özkütlesinin Al mikro parçacıklarından büyük olmasından dolayı kaynaklanabilir çünkü spesifik yüzey alanındaki artış parçacıklar arasındaki sürtünmeyi artırarak parçacıkların homejen olarak dağılmasını engellemektedir.
- Beklenildiği gibi sinterleme sıcaklığının artmasıyla özkütle artmakta ve gözeneklik oranı azaltmaktadır çünkü sinterleme sıcaklığı atomik difüzyon hızını artırarak kompozitin ıslanabirliği ve sinterleme yeteneğini artırmaktadır. Bunun sonucu olarak kompozitteki gözenek oranları azalmıştır.
- Sinterleme işlemiyle gözenek oranının azalmasına rağmen mekanik özelliklerde bir iyileşme görülmemektedir

#### Referanslar

- 9. Askeland, D. R. ve Fulay, P. P., Essentials of Materials Science and Engineering. 2nd ed. **2009**, Cengage Learning. New York.
- 10. Callister, W. D., Materials Science and Engineering An Introduction. 7th ed. **2007**, John Wiley & Sons, Inc. New York.
- 11. Groover, M. P., Fundamentals of modern manufacturing: materials processes, and systems. 4th ed. **2010**, John Wiley & Sons, Inc. New York.
- 12. Kumar, V. M. ve Venkatesh, C. V comprehensive review on material selection, processing, characterization and applications of aluminium metal matrix composites. Materials Research Express, **2019**, 6: 072001.
- Manu, K. M. S., Raag, L. A., Gupta, M. ve Pai, B. Liquid Metal Infiltration Processing of Metallic Composites: A Critical Review. Metallurgical And Materials Transactions B., 2016, 47B: 2799–2819.
- 14. Rajan, T. P. D., Pillai, R. M., ve Pai, B. C. Reinforcement coatings and interfaces in aluminium metal matrix composites. Journal of Materials Science, **1998**, 33: 3491–3503.
- 15. Pilarska, A. A., Klapiszewski, L. ve Jesionowski, T. Recent development in the synthesis, modification and application of Mg(OH)2 and MgO: A review. Powder Technology, **2017**, 319: 373–407.

# AL-MG-SIC KOMPOZITLERIN GÖRÜNÜR YOĞUNLUKLARININ TAGUCHI ANALIZI

The International Conference

aterials and Engineering Technology

# ABDULAZIZ KAYA<sup>1</sup>, MIKAIL ASLAN<sup>1</sup>, NECIP FAZIL YILMAZ<sup>2</sup>, HALIL IBRAHIM KURT<sup>1</sup>

<sup>1</sup>Metalurji ve Malzeme Mühendisliği Bölümü, Mühendislik Fakültesi, Gaziantep Üniversitesi, Gaziantep, TÜRKİYE. <sup>2</sup>Makine Mühendisliği Bölümü, Mühendislik Fakültesi, Gaziantep Üniversitesi, Gaziantep, TÜRKİYE.

# ÖZET

Bu çalışmanın amacı üretilen metal matrisli kompozitlerde işlem parametrelerin Taguchi ve ANOVA yaklaşımı kullanılarak araştırılması ve analiz edilmesidir. Bu amaçla toz metalürjisi yöntemiyle üretilen kompozitlerin Arşimet prensibine göre elde edilen görülür yoğunluk değerleri Minitab programı kullanılarak araştırılmıştır. Deneysel tasarım yapılması ve sonuçların etkileşimi ve uyumu kullanılan analiz ile değerlendirilmiş olup izlenen yolun ve yapının doğruluğunun yüksek olduğu gözlenmiştir.

Anahtar Kelimeler: Taguchi, Toz metalürjisi, Kompozit, SiC

# 1.Giriş

Kompozit malzemeler, olan iki veya daha fazla farklı malzemenin sistematik bileşimiyle elde edilen yeni malzemelerdir [1]. Matrisin özelliklerinin yanısıra, güçlendiricinin morfolojisi, özellikleri, oranı ve dağılımı kompozitin özelliklerinin büyük ölçüde etkilemektedir [2-4]. Yani, güçlendirici ile matrisin uyumluluğu da önemli bir konudur. SiC takviyeli kompozitler, düşük ısıl genleşme katsayısı, yüksek özgül mukavemet ve ısıl iletkenliği sayesinde elektronik alanlardaki ambalajlarda, havacılık, askeri ve otomotiv gibi birçok endüstriyel uygulamada yaygın olarak kullanılmaktadır [5-7].

Komozit üretiminde birçok yöntem kullanılmakta olup bu yöntemler birçok avantaj ve dez avantaja sahiptir [8-10]. Toz metalurisi yöntemi üretim yöntemini hızlılığı, hurda miktarının azaltılması, geniş kompozisyon aralığında çalışma ve talaşlı işleme gerek duymadan üretim gibi önemli avantajlara sahip bir yöntemdir [11, 12]. Bütün üretim yöntemlerinde karşılaşılan ve engellenmeye çalışılan en önemli sorunlardan biri porozite içeriği olup buda doğrıdan yoğunlukla ilgilidir. Yeni mühendislik çalışmalarında en önemli hedeflerden biri ise malzemenin iyi olan özelliklerinin korunarak zayıf özelliklerini geliştirmek ve tüm bunları yaparken minimum yoğunluk değerleri elde etmektir. Bu nedenler son yıllarda en çok çalışan konu alüminyum ve artan düzeyde magnezyum alaşımlarıdır [13, 14].



Bu çalışmada, toz metalürji yöntemiyle üretilen alüminyum matrisli kompozitlerin yoğunlukları Taguchi ve ANOVA yöntemleriyle analiz edilmesi amaçlanmıştır.

# 2. Malzeme ve Yöntem

Bu çalışmada Al-Mg-SiC kompozitler toz metalürjisi yöntemiyle üretilmiş olup kompozitlerin görünür yoğunlukları Taguchi yöntemiyle incelenmiştir. Kompozitlerin yoğunlukları, Arşimet prensibine göre değerlendirilmiştir. Teorik yoğunluklar ve ölçülen yoğunluklar kullanılarak görünür yoğunluk değerleri hesaplanmıştır [15]. Daha sonra görünür yoğunluk değerleri Taguchi ve ANOVA yöntemi kullanılarak analiz edilmiştir [16, 17]. Kullanılan giriş ve çıkış parametreleri Tablo 1'de verilmiştir.

SiC	Basınç (MPa)	Sinterleme sıcaklığı (ºC)	Sinterleme süresi (dk)	Görünür yoğunluk (gr/cm <sup>3</sup> )
15	260	300	30	2,510
15	260	300	90	2,445
15	260	500	30	2,359
15	260	500	90	2,541
15	520	300	30	2,538
15	520	300	90	2,439
15	520	500	30	-
15	520	500	90	2,574
30	260	300	30	2,619
30	260	300	90	2,539
30	260	500	30	2,547
30	260	500	90	2,473
30	520	300	30	2,595
30	520	300	90	2,474
30	520	500	30	-
30	520	500	90	2,542

Tablo 1	. Sistemde	kullanılan	parametrele	er
		Ranannan	parametre	· <b>I</b>

Taguchi L16(2\*\*4) ortogonal dizi tasarımı kullanılmıştır. Bu tasarım 2 seviye ve 4 faktör olarak karşımıza çıkmaktadır. S/N oranı (Signal/ Noise) için "Smaller is better" fonksiyonu kullanılmıştır.

# 3. Bulgular ve Tartışma

Veri grupları arasındaki farklılıkları veya benzerlikleri düzeltmek için kullanılan istatistiksel bir araç olan deneysel (DOE) ve varyans analizi (ANOVA), işlem parametrelerinin çıktı değeri üzerindeki etkilerini ortaya çıkarmak için yoğun bir şekilde kullanılmaktadır. Deneylerin tasarlanmasında Minitab yazılımı ve Taguchi yöntemi kullanılmıştır. S/N oranındaki sinyal ve gürültü istenen ve



istenmeyen değerlerdir. Bu oran, sürecin performansını ve önemini hesaplamak için yaygın olarak kullanılır. Şekil 1 ve 2, görünür yoğunluk değerleri için temel etki ve S/N oranlarını ve değişimlerini göstermektedir.



Şekil 1. Görünür yoğunluk değeri için temel etki değişkenleri



Şekil 2. Test (Görünür yoğunluk) değeri için S/N oranı

S/Nratio değeri ne kadar yüksek olursa, o kadar doğru veri ve herhangi bir giriş değişkeni ile çıkış parametresi arasındaki yakın ilişki anlamına gelir. Verilen grafikler ışığında, düşük yoğunluk elde edebilmek için SiC oranının %15, Basınç 260 MPa, Sıcaklık 500 °C ve zaman 90 dk olarak tespit edilmiştir. Bu varsayım ANOM analizi (Şekil 3) ile doğrulanmıştır. Sonuçlar benzer çalışmalarda da görülmektedir [18, 19].






Tablo 2, deney değişkenlerinin Delta ve Rank (sıra) değerlerini göstermektedir. Delta S/N deki maksimum ve minimum arasındaki fark olup Rank ise üretilen kompozitlerin yoğunlukları üzerinde hangi değişkenin daha etkili olduğunu göstermektedir. Sic oranın yoğunluk üzerinde en büyük etkiye sahipken sıcaklık diğer değişkenlere göre en düşük etkiye sahip olduğu görülebilir.

Tablo 2. Degişkenlerin etkisi								
	SiC	Basınç	Sıcaklık	Zaman				
Delta	0,055	0,023	0,014	0,024				
Rank	1	3	4	2				

Şekil 4 parametrelerin birbiri üzerindeki etkiler göstermektedir. Örneğin, sistemde sıcaklık değerinin artması zaman değerinin azalmasıyla doğru orantılıdır. Düşük yoğunluk için, 520 MPa basınç, 500°C sıcaklık ve 90 dk zaman kullanılmalıdır.



UWE Bristol

Şekil 4. Etkileşim grafiği

Bağlantı eşitliğini ifade etmek gerekirse sistem tarafında bu eşitlik "*Yoğunluk* = 2,43 + 0,00364 SiC + 0,000104 Basınç - 0,000024 Sıcaklık- 0,000468 Zaman" olarak hesaplanmıştır. Giriş değişkenlerin uyumluluğuna ait grafik Şekil 5' de verilmiştir.



Şekil 5. Uyumluluk Grafiği

Genel anlamda uyumluluk değişimleri büyük benzerlik göstermektedir. Standart sapma değeri SiC oranı için minimum seviyede olduğu ve basınç için maksimum seviyededir. P-değerleri ise 0,005 den küçük olmakta ve sistem değişkenlerinin anlam seviyesini göstermektedir. Beklediğimiz gibi bu değişkenlerin birbiri ile uyumu anlamlı olduğunu söyleyebiliriz.

The International Conference of Materials and Engineering Technology

### 4.Sonuçlar

Bu çalışmada toz metalürjisi yöntemiyle üretilen Al-Mg-SIC kompozitlerin görünür yoğunluk değerleri Taguchi ve ANOVA yöntemiyle incelenmiştir. Kompozitlerin yoğunlukları, Arşimet prensibine göre değerlendirilmiştir. Daha sonra görünür yoğunluk değerleri Taguchi ve ANOVA yöntemiyle yöntemi kullanılarak analiz edilmiştir. Düşük yoğunluk değerleri elde etmek için verilen parametreler ışığında hangi parametrelerin kullanılması gerektiği belirlenmiştir. Bu parametrelerin birbiri üzerinde etkileşiminin de etkili olduğu görülmüştür. Bu sonuçlar tasarımın güvenilir ve uyum içerisinde olduğunu göstermiştir.

#### Referanslar

- 1. Hashim, J., L. Looney, and M. Hashmi, Metal matrix composites: production by the stir casting method. Journal of Materials Processing Technology, 1999. **92**: p. 1-7.
- 2. Metal matrix composites reinforced by nano-particles—a review. Metals, 2014. 4: p. 65.
- 3. Tjong, S.C., Novel nanoparticle-reinforced metal matrix composites with enhanced mechanical properties. Advanced engineering materials, 2007. **9**(8): p. 639-652.
- 4. Tjong, S.C., 8 Processing and Deformation Characteristics of Metals Reinforced with Ceramic Nanoparticles, in Nanocrystalline Materials (Second Edition), S.-C. Tjong, Editor. 2014, Elsevier: Oxford. p. 269-304.
- 5. Molina, J.M., et al., Pressure infiltration of liquid aluminium into packed SiC particulate with a bimodal size distribution. Acta Materialia, 2002. **50**(2): p. 247-257.
- 6. Zhang, Q., X. Ma, and G. Wu, Interfacial microstructure of SiCp/Al composite produced by the pressureless infiltration technique. Ceramics International, 2013. **39**(5): p. 4893-4897.
- 7. Geiger, A.L., D.P.H. Hasselman, and P. Welch, Electrical and thermal conductivity of discontinuously reinforced aluminum composites at sub-ambient temperatures. Acta Materialia, 1997. **45**(9): p. 3911-3914.
- 8. Abbasipour, B., B. Niroumand, and S.M. Monir Vaghefi, Compocasting of A356-CNT composite. Transactions of Nonferrous Metals Society of China, 2010. **20**(9): p. 1561-1566.
- 9. Abdizadeh, H., P. Vajargah, and M. Baghchesara, Fabrication of MgO nanoparticulates reinforced aluminum matrix composites using stir-casting method. KOVOVE MATERIALY-METALLIC MATERIALS, 2015. **53**(5): p. 319-326.
- Anvari, S.R., F. Karimzadeh, and M.H. Enayati, Wear characteristics of Al–Cr–O surface nano-composite layer fabricated on Al6061 plate by friction stir processing. Wear, 2013. 304(1–2): p. 144-151.



The International Conference

of Materials and Engineering Technology

- 12. ANGELO, P.C. and R. SUBRAMANIAN, POWDER METALLURGY: SCIENCE, TECHNOLOGY AND APPLICATIONS. 2008: PHI Learning.
- Jia, H., X. Feng, and Y. Yang, Effect of grain morphology on the degradation behavior of Mg-4 wt% Zn alloy in Hank's solution. Materials Science and Engineering: C, 2020. 106: p. 110013.
- 14. Zhang, Y.H., et al., Grain refinement of hypoeutectic Al-7wt.%Si alloy induced by an Al–V– B master alloy. Journal of Alloys and Compounds, 2020. **812**: p. 152022.
- 15. Kurt, M., SiC ve Al2O3 takviyeli Al-Mg kompozitin toz metalurjisi yöntemi ile üretiminin araştırılması, in Metallurgical Engineering. 2017, Fırat University.
- 16. Roy, R.K., A primer on the Taguchi method. 2010: Society of Manufacturing Engineers.
- Rama, R.S. and G. Padmanabhan, Application of Taguchi methods and ANOVA in optimization of process parameters for metal removal rate in electrochemical machining of Al/5% SiC composites. International Journal of Engineering Research and Applications (IJERA), 2012. 2(3): p. 192-197.
- 18. Yilmaz, N.F., et al., Experimental and theoretical analysis of the welding process parameters for UTS with different methods. Materials Research Express, 2018. **6**(1): p. 016524.
- 19. Rosa, J.L., et al., Electrodeposition of copper on titanium wires: Taguchi experimental design approach. Journal of materials processing technology, 2009. **209**(3): p. 1181-1188.

# TÜMÜYLE ELEKTRİKLİ BİNEK TİPLİ BİR ARAÇTA YUVARLANMA DİRENCİ DEĞİŞİMİNİN İVMELENME PERFORMANSI VE TRANSMİSYON KAYIPLARINA ETKİSİ ÜZERİNE BİR ÇALIŞMA

The International Conference

aterials and Engineering Technology

### M. AKİF KUNT \*1

<sup>1</sup> Dumlupınar Üniversitesi, Tavşanlı MYO, Motorlu Araçlar ve Ulaştırma Teknolojileri :, Kütahya, TÜRKİYE.

### Öz

Elektrikli araçların günümüzde halen istenilen düzeyde kullanılmamasının en önemli sebebi sınırlı menzilleridir. Elektrikli araçların menziline etki eden en önemli faktörlerden birisi yuvarlanma direnci etkisidir. Lastik tasarımına ve çevresel koşullara bağlı olarak değişebilen yuvarlanma direnç kuvveti değişimi fren ve batarya performansıyla birlikte menzili de etkilemektedir. Bu çalışmada binek tipli tümüyle elektrikli bir araç için ADVISOR taşıt simülasyon programı kullanılarak iki farklı yuvarlanma direncinde elektrikli taşıtın ivmelenme ve transmisyon kayıpları incelenmiştir. Sürüş çevrimine göre ortalama yuvarlanma direnç kuvvetleri arasında 2.1 kat fark meydana gelmiştir. Düşük yuvarlanma dirençli lastiklerin hızlanma süresi 0-96.6 km/h hızlar arasında 0.4 saniye, 64.4-96.6 km/h hızlar arasında 0.2 saniye, 0-137 km/h hızlar arasında ise 0.5 saniye azalmıştır. Ayrıca frenleme enerjisi düşük yuvarlanma dirençli lastiklerde % 7.3 daha fazla meydana gelmiştir.

Anahtar kelimeler: Yuvarlanma direnci, transmisyon kayıpları, sürüş çevrimi, ivmelenme, rejenerasyon

#### 1.Giriş

Otomobil üretim teknolojilerinin gelismesi ile üretilen otomobillerin sayısı da artmaktadır. Bu durum otomobillerden beklentileri artırarak enerji gereksiniminin daha fazla artmasına neden olmuştur. Dünya üzerindeki enerji rezervlerinin daha verimli kullanılması ve taşıt kaynaklı kirlenmenin azaltılabilmesi için elektrikli araçlar önemli bir alternatiftir. Çevre duyarlılığı ve verimlilik bakımından elektrikli araçlar son zamanlardaki en uygun taşıtlardır [1]. EA'ların yaygınlaşmasını etkileyen en önemli faktörler; uzun şarj süresi ve araçların kısa menzil mesafeleridir [2]. EA'ların menzillerinin artırılmasında en önemli faktörler taşıt ön izdüşüm alanı, batarya enerji yoğunluğu ve yuvarlanma direnç katsayısıdır [3]. Elektrikli araçlarda enerji kullanımını daha verimli hale getirmek için uygulanabilecek yöntemlerden biri de araçların kullanımı sırasındaki kayıpları azaltmaktır. Araçlar üzerinde etkisi olan direnç kuvvetleri enerji kaybına neden olmaktadır. Bu kuvvetlerden belki de en önemlisi yuvarlanma direnç kuvvetidir [4]. Yuvarlanma direnci ile taşıtın enerji tüketimi arasında doğrudan bir bağlantı vardır [5]. Yuvarlanma direnci sürüş halindeki bir taşıtın oluşturduğu enerjinin %25'ini harcamaktadır. Bu durum elektrikli araçların menzillerinin artırılması konusunda yuvarlanma direncini oldukça önemli bir konuma getirmektedir. Özellikle düşük taşıt hızlarında yuvarlanma direnç kuvveti enerji kayıpları açısından büyük önem kazanmaktadır [6]. Yuvarlanma direncinin taşıttaki diğer enerji kayıpları arasındaki durumu Şekil 1' de gösterilmiştir.



Şekil 1. Taşıt enerji kayıplarının yüzde gösterimi [6]

Yuvarlanma direncine etki eden temel faktörler kullanım koşulları, hız, şişirme basıncı ve yüktür. Bu faktörler frenleme, tahrik ve tutunma durumları dikkate alınarak optimum bir değerde tutulursa elektrikli otomobillerde enerji tasarrufu sağlanarak menzil artırılabilir. Lastik yuvarlanma direncinin standart ölçüm teknikleri Amerikan Otomobil Mühendisler Odasının SAE J1269 ve SAE J1270 standartlarında yer almaktadır. SAE J1269 standardı, laboratuvar ortamında pnömatik binek, hafif kamyon ve karayolu kamyonu ve otobüs lastiklerinin yuvarlanma direncinin ölçülmesi için hazırlanmıştır. Standart serbest kaymalı lastiklerin kaymasız ve sabit eğim açılı durumda çalışması koşulları için geçerlidir. SAE J2452 standartlarına göre de yuvarlanma direnç kuvveti hesaplanabilmektedir.

### 2. Elektrikli aracın modellenmesi

Elektrikli aracın modellenmesi amacıyla ADVISOR (Advanced Vehicle Simulator) programı kullanılmıştır. Gerçek sürüş şartlarında çevresel kararlılığın (yol eğimi, yuvarlanma direnç katsayısı, çevre sıcaklığı vb) sağlanmasında karşılaşılan zorluklar sebebiyle simülasyon yoluyla analiz yapılmasına karar verilmiştir. Bu program yeni araçların gelişimine hızlandırmak için ABD hükümeti tarafından kurulan Ulusal Yenilenebilir Enerji Laboratuarı (NREL-National Renewable Energy Laboratory) tarafından oluşturulmuş bir simülasyon programıdır. Program daha sonra otomobil üreticileri ve üniversitelerin katılımıyla güncellenmiş ve yeni özellikler eklenmiştir [7]. ADVISOR, programın kullanımını kolaylaştırmak amacıyla, grafiksel kullanıcı ara yüzünden de yararlanmaktadır. Araç tipi ve bileşenlerinin seçiminde kullanılan tanımlama ekranı Şekil 2'de gösterilmiştir.

/ehicle Input			Load File g	gm_ev	1_in			V		Auto-Size		
			Drivetrain Co	evers	/	tvp	e l	×		max	Scale peak	ma
		5	Vehicle		~ '	2	~	VEH_SMCAR	×	pm	- CII	(3)
	-		Fuel Converter			2		fc options				
1.			Exhaust Aftertr		1	2		EX_CI		#of	V nom	
1			Energy Storage	rint	~ '	NiZn	~	ESS_NIZN22_temp	v	25	307	
			Energy Storage 2			2		ess 2 options				
-			Motor		~ '	2	~	MC_AC124_EV1	v	105	0.95	
	-		Motor 2		1	2		motor 2 options				
			Starter		1	?		starter options				
			Generator		1	?		gc options				
			Transmission	man	v '	man	×	TX_1SPD	v		0.93	
Componen	Plot Selection		Transmission 2		1	2		trans 2 options				
energy_sto 💙	ess Rint	~	Clutch/Torq. Co			2		clutch/torque conv				
	Battery Resistance:		Torque Coupling		1	2		TC_DUMMY				
	rcel NIZh battery, tested at NRt		Wheel/Axle	Crr	*	Crr	×	WH_SMCAR_REG	¥			
12 An Ever						the second second		and a state of the second				
12 12	26C Discha	rge	Accessory	C	Y _	Co	Y	ACC_HYBRID	~			
12 0.1	26C Discha	rge rge	Accessory Acc Electrical	C	× 1	Co	×	ACC_HYBRID acc elec options	~			

Şekil 2. ADVISOR giriş ekranı

Sürüş çevrimi olarak maksimum hızı 50 km/h olan ve düşük hızlarda kullanılan NYCC (New York City Cycle) sürüş çevrimi seçilmiştir. Sürüş çevrimine ait özellikler Tablo 1'de gösterilmiştir.

Tablo1. Sürüş çevriminin özellikleri.								
Veriler	Değerler	Birimler						
Süre	598	S						
Mesafe	1.9	km						
Maksimum hız	44.58	km/h						
Ortalama hız	11.41	km/h						
Maksimum ivme	2.68	$m/s^2$						
Maksimum yavaşlama ivmesi	-2.64	$m/s^2$						
Rölanti süresi	210	S						
Duraklama sayısı	18							



#### 2.1 Aracın Hareketine Etki Eden Kuvvetler

Aracın hareketine etki eden temel kuvvetler çevresel faktörlerinde etkili olduğu direnç kuvvetleri ve direnç kuvvetlerini karşılamak üzere motor tarafından üretilen gücün tekerleklere kadar iletilmesini sağlayan tahrik (çekiş) kuvvetidir. Direnç kuvvetleri denklem 1'de gösterilen lastiklerdeki yuvarlanma direnci ( $F_y$ ), aerodinamik (hava) sürtünme ( $F_h$ ), aracın aşması gereken atalet direnci ( $F_a$ ) ve eğim direnci ( $F_e$ )'dir. Aracın tahrik edilebilmesi için tüm bu dirençlerin aşılması gerekmektedir.

he International Conference of N

$$F_t = F_y + F_h + F_a + F_e$$
(1)  

$$F_y = f_{ro}G$$
(2)

(2) no' lu denklemde ( $f_{ro}$ ) yuvarlanma direnç katsayısını, (G) taşıt ağırlığını ifade etmektedir. Yuvarlanma direnci taşıtın hızlanma performansına önemli ölçüde etki etmektedir.

#### 2.2 Yuvarlanma direnç katsayısı

Yuvarlanma direnci SAE J2452 standartlarına göre hesaplanmıştır. SAE J2452 lastik yuvarlanma direncinin hesaplanması için hazırlanmış bir standarttır. Daha önce kullanılan SAE J1269 standardı lastiğin çalışma koşullarında kararlı durumda olduğunu kabul ederken SAE J2452 standardı farklı taşıt yükü, lastik basıncı ve hızlarının yuvarlanma direncine etkisini belirleyebilmektedir. Bu standarda göre yuvarlanma direnç kuvveti:

$$F_r = P^{\alpha} Z^{\beta} (a + bV + cV^2)$$

(8) no'lu bağıntıda (P) lastik basıncını, (Z) lastiğe düşeyde tesir eden yükü, (V) taşıt hızını, (a, b, c) model için hesaplama katsayılarını göstermektedir. (a), (b) ve (c) katsayılarının değeri sırasıyla yuvarlanma direncinin değerine göre Tablo'2 de verilmiştir.

Tablo 2. SAE J2452 test standartlarına	göre	yuvarlanma	direnç	kuvveti	hesap	lama	katsay	ıları.
--	------	------------	--------	---------	-------	------	--------	--------

	a	b	с
Yuvarlanma direnci (düşük)	0.0682	2.32*10(-4)	1.2 * 10 (-6)
Yuvarlanma direnci (yüksek)	0.159	3.44*10 <sup>(-4)</sup>	1.25 * 10 (-6)

### 3. Materyal ve Metot

Modellemesi yapılan elektrikli taşıtın parametreleri Tablo 3'te verilmiştir. ADVISOR taşıt simülasyon programı kullanılarak sonuç grafikleri oluşturulmuştur. SAE J2452 standartlarına göre 2 farklı yuvarlanma direnç kuvveti elde edilmiştir. Elektrikli taşıtların şehir içi sürüşlerinde daha verimli olması sebebiyle dur-kalk sayısı fazla olan NYCC (New York City Cycle) sürüş çevrimi seçilmiştir. NYCC sürüş çevrimine göre 1 sürüş çevriminde meydana gelen hızlanma performansları benzetilmiştir. Aracın durgun hava içerisinde hareket ettiği ve yol eğiminin olmadığı kabul edilmiştir. Lastik basıncı ve lastiğe düşeyde tesir eden yük sürüş çevrimi boyunca değişmemektedir.



Veriler	Değerler	Birimler
Taşıt kütlesi	1465	kg
Tekerlek yarıçapı	0.3	m
Yerçekimi ivmesi	9.81	$m/s^2$
Hava yoğunluğu	1.204	kg/m <sup>3</sup>
Rejeneratif frenleme oranı	30	%
İnvertör verimi	95	%
Konvertör verimi	95	%
Batarya verimi	95	%
Diferansiyel redüksiyon oranı	10	
Elektrik motor verimi	95	%
Lastik ölçüleri	205/60 R15	
Batarya modül sayısı	26	
Batarya kapasite	80	Ah

### Tablo 3. Tașıta ait model parametreleri.

The International Conference

of Materials and Engineering Technology

### 4. Simülasyon Sonuçları

Şekil 3'te sürüş çevrimine göre zaman bağlı mesafe değişimi görülmektedir. 18 adet duraklama sayısına sahip olan sürüş çevrimi 598 saniye sürmektedir. Çevrim tamamlandığında 1.9 km yol edilmiş olup rölanti süresi toplam 210 saniyedir.



Şekil 3. NYCC çevrimine göre zamana bağlı mesafe değişimi

Şekil 4'te zaman bağlı taşıt hızı değişimi gösterilmiştir. Seçilen sürüş çevrimi dur-kalk sayısı fazla olan bir çevrimdir. Bu nedenle hızlanma ivmesi 2.68 m/s<sup>2</sup>, yavaşlama ivmesi ise -2.64 m/s<sup>2</sup> olarak gerçekleşmiştir. Sürüş sırasında maksimum hız 44.58 km/h olarak gerçekleşmektedir. Çevrimin ortalama hızı 11.41 km/h'dir.



Şekil 4. NYCC çevrimine göre zamana bağlı taşıt hızı değişimi



The International Conference

aterials and Engineering Technology



Şekil 5. NYCC çevrimine göre zamana yuvarlanma direnç kuvvetinin değişimi

Tablo 4'te simülasyon sonucu elde edilen bazı sonuç çıktıları gösterilmiştir. 1 sürüş çevrimi için düşük yuvarlanma dirençli lasti5 % 6.25 yakıt ekonomisi sağlamıştır. 0-96.6 km/h hızlar arasında hızlanma süresinde düşük yuvarlanma dirençli lastik kullanılması durumunda 0.4 saniye azalma meydana gelmiştir. 64.4-96.6 km/h hızlar arasında hızlanma süresinde düşük yuvarlanma dirençli lastik kullanılması durumunda 0.2 saniye azalma meydana gelmiştir. 0-137 km/h hızlar arasında hızlanma süresinde düşük yuvarlanma dirençli lastik kullanılması durumunda 0.5 saniye azalma meydana gelmiştir. Advisor programı bu hızlanma performansındaki değerlendirmeleri ileri yönlü iterasyon yaparak hazırlamaktadır. Frenleme sırasında gereken enerjinin yüksek yuvarlanma dirençli lastiklerde daha fazla absorbe edilmesi sebebiyle frenleme enerjisi düşük yuvarlanma dirençli lastiklerde % 7.3 daha fazla meydana gelmiştir. Ayrıca 1 sürüş çevrimi için tahrik durumunda aerodinamik direnç dışında diğer aktarma organları kayıpları yüksek yuvarlanma dirençli lastiklerde daha fazladır.



The International Conference of Materials and Engineering Technology

### Şekil 6'da rejenerasyon sırasında yuvarlanma dirençlerine ait enerji kayıplarını göstermektedir. Rejenerasyonlu frenleme sırasında düşük yuvarlanma dirençli lastik daha fazla enerji tüketmiştir. Transmisyon ve motor kayıpları da bataryaların daha yoğun deşarj olmaları yüksek yuvarlanma dirençli lastiklere göre sebebiyle daha yüksektir. Tahrik sırasında ise yüksek yuvarlanma dirençli lastiğin enerji tüketimini fazla olması güç aktarma organlarındaki kayıpları artırmıştır.



(Düşük yuvarlanma dirençli lastik)



Şekil 6. Frenleme durumunda taşıt enerji kayıpları

Şekil 7'de tahrik sırasında yuvarlanma dirençlerine ait enerji kayıplarını göstermektedir. Tahrik sırasında düşük yuvarlanma dirençli lastik % 4.4 daha düşük miktarda enerji tüketmiştir. Transmisyon ve motor kayıpları da yuvarlanma direncinin daha az oluşması sebebiyle yüksek yuvarlanma direncine sahip lastiklere göre daha düşüktür. Tahrik sırasında ise düşük yuvarlanma dirençli lastikte daha yüksek rejenerasyon kazanımı olması sebebiyle batarya şarj yoğunluğu artmış ve % 8.4 daha fazla batarya kaybı meydana gelmiştir. Batarya enerji kaybının sebebi rejenerasyon işlemi sırasında bataryada meydana gelen şarj-deşarj yoğunluğunun fazla olmasıdır. Aerodinamik direnç kuvveti değişmemiştir.



The International Conference

aterials and Engineering Technology

Şekil 7. Tahrik durumunda taşıt enerji kayıpları

#### 5. Sonuçlar

Seçilen sürüş çevrimine göre yuvarlanma direnç kuvvetleri arasında 2.1 kat fark meydana gelmiştir. Yuvarlanma direnci düşük lastiğin 0-96.6 km/h hızlar arasında hızlanma süresinde 0.4 saniye, 64.4-96.6 km/h hızlar arasında hızlanma süresinde 0.2 saniye, 0-137 km/h hızlar arasında ise 0.5 saniye azalma meydana gelmiştir. Yüksek yuvarlanma dirençli lastiklerde frenleme sırasında gereken enerjinin daha fazla absorbe edilmesi sebebiyle frenleme enerjisi düşük yuvarlanma dirençli lastiklerde % 7.3 daha fazla meydana gelmiştir. Yüksek yuvarlanma dirençli lastiklerde transmisyon ve motor kayıpları bataryaların daha yoğun deşarj olmaları göre sebebiyle daha yüksektir. Rejenerasyon kazanımının yüksek olması sebebiyle düşük yuvarlanma direncine sahip lastiklerde batarya şarj yoğunluğu artmış ve % 8.4 daha fazla batarya kaybı meydana gelmiştir.

#### Kaynaklar

1. Kunt, M.A., Tümüyle elektrikli binek tipli bir aracın Advisor tabanlı modellenmesi ve aerodinamik direnç değişiminin batarya performansına etkisi üzerine bir çalışma, ISASTECH 2019, 5-6 Eylül 2019, Ankara, Türkiye.

The International Conference of Materials and Engineering Technology

- 2. Huang Q., Li J. and Chen Y., Control of Electric Vehicle. Urban Transport and Hybrid Vehicles, InTech, Chengdu. 2010.
- 3. Yuan X., Li L., Gou H. and Dong T. Energy and environmental impact of battery electric vehicle range in China. Applied Energy, 2015, 157: 75-84.
- 4. SAE Standartları Testleri. https://www.eurolab.com.tr/sektorel-test-ve-analizler/endustriyel-testler/sae-standartlari-testleri. 19.09.2019.
- 5. Tat, M.A., Özenç, F., Otomobil lastiklerinde yuvarlanma direncini etkileyen faktörlerin ve standart yuvarlanma direnci ölçüm tekniklerinin incelenmesi, Mühendis ve Makine, 2007, 48(572):16-72.
- Kunt M.A., Advisor based modelling of the effect of rolling resistance on regenerative braking in all-electric passenger cars, El-Cezerî Journal of Science and Engineering, 2019, 6(3): 847-855.
- 7. Nokian Tyres plc, Finland, European Tyre School Project. Supported by Leonardo da Vinci Programme of European Commision. www.tut.fi/plastics/tyreschool/index.html.
- 8. Wipke K. B., Cuddy M. R., and Burch S. ADVISOR 2.1: A User-friendly advanced power train simulation using a combined backward/forward approach. IEEE Transactions on Vehicular Technology, 1999, 48(6), 1751-1761.

# AL-MG-AI2O3 KOMPOZITLERIN YAPAY SİNİR AĞLARI YAKLAŞIMI İLE POROZİTE İÇERİĞİNİN ARAŞTIRILMASI

The International Conference of Materials and Engineering Technology

### HALIL IBRAHIM KURT<sup>1</sup>, ENGIN ERGUL<sup>2</sup>, NECIP FAZIL YILMAZ<sup>3</sup>, MURAT ODUNCUOGLU<sup>4</sup>

<sup>1</sup>Metalurji ve Malzeme Mühendisliği Bölümü, Mühendislik Fakültesi, Gaziantep Üniversitesi, Gaziantep, TÜRKİYE <sup>2</sup>İzmir Meslek Yüksekokulu, Dokuz Eylül Üniversitesi, İzmir,TÜRKİYE. <sup>3</sup>Makine Mühendisliği Bölümü, Mühendislik Fakültesi, Gaziantep Üniversitesi, Gaziantep, TÜRKİYE. <sup>3</sup>Fizik Bölümü, Yıldız Teknik Üniversitesi, İstanbul, TÜRKİYE.

#### Özet

Esnek/Yumuşak hesaplama, esnek bilişim tekniklerinin ve araçlarının hem günlük hem de gelişmiş uygulamalara kullanımı önemlidir. Esnek/Yumuşak hesaplamanın uygulamalarını ve tekniklerini diğer multi-disiplinlerle ilişkilendirerek, mühendislik karşılaştırmaları, uzantıları ve yeni uygulamaları teşvik eden birleştirici bir platform görevi görür. Bu çalışmada da, bir mühendislik çalışması olan toz metalürjisi yöntemiyle üretilen Al-Mg-Al<sub>2</sub>O<sub>3</sub> kompozitlerin görünür porozite değerlerinin esnek hesaplama yöntemi olan yapay sinir ağları (ANN; YSA) yöntemiyle incelenmiştir. Deneysel/ölçülen görünür porozite değerleri eğitim ve test verisi olarak ayrılmıştır. Kullanılan verilerin ortalama karasel hataları (MSE), ortalama mutlak hata (MAE) ve korelasyon katsayısı (R) kullanılarak araştırılmıştır. Elde edilen sonuçların eğitim testinde yüksek test kısmında kabul edilebilir olduğu görülmüştür. Sistem parametreleri değiştirilerek daha yüksek tutarlılık oranlarının elde edilebileceği sonucuna varılmıştır.

Anahtar Kelimeler: ANN, Toz metalürjisi, Kompozit, Alümina

#### 1. Giriş

Yumuşak hesaplama, kendini esnek hesaplama tekniklerine dayalı sistem çözümlerine adanmıştır. Yumuşak bilişim teknolojilerinde önemli sonuçların hızlı bir şekilde yayılmasını, evrimsel algoritmalar ve genetik programlama, sinir bilimi ve sinir ağları sistemindeki araştırmaların birleştirilmesini sağlar[1]. Yapay sinir ağları (YSA), insan beyninin özelliklerinden olan öğrenme yolu ile yeni bilgiler türetebilme, yeni bilgiler oluşturabilme ve keşfedebilme gibi yetenekleri, herhangi bir yardım almadan otomatik olarak gerçekleştirebilmek amacı ile geliştirilen bilgisayar sistemleridir [2]. Yapay sinir ağları; insan beyninden esinlenerek, öğrenme sürecinin matematiksel olarak modellenmesi uğraşı sonucu ortaya çıkmıştır. Bu nedenledir ki, bu konu üzerindeki çalışmalar ilk olarak beyni oluşturan biyolojik üniteler olan nöronların modellenmesi ve bilgisayar sistemlerinde uygulanması ile başlamış, daha sonraları bilgisayar sistemlerinin gelişimine de paralel olarak birçok alanda kullanılır hale gelmiştir. YSA'lar birçok hücreden meydana gelir ve bu hücreler eş zamanlı olarak çalışarak karmaşık işlevleri yerine getirir. Diğer bir değişle karmaşık işlevler birçok hücrenin eş zamanlı çalışması ile meydana getirilir. Süreç içerisinde bu hücrelerden her hangi biri işlevini yitirse dahi sistem güvenli bir şekilde çalışmasına devam edebilir. Eğitim esnasında kullanılan nümerik bilgilerden, problemin genel özellikleri elde etmesi ve böylelikle eğitim sırasında kullanılmayan girdiler için de, anlamlı yanıtlar üretebilmesidir [3]. Özellikle mühendislik uygulamalarında zaman ve malzeme tasarrufu ile üretim maliyetlerini azaltmada ve devam eden süreçlerde işlem parametrelerinin etkileşimi konusunda büyüm önem taşımaktadır [4-8]. Bu çalışmanın amacı üretilen metal matrisli kompozitlerde işlem parametrelerin araştırılması, analiz edilmesi ve ileri süreçlerden yaşanabilecek kayıpları minimum seviyeye indirilmeye çalışılmasıdır.

The International Conference of Materials and Engineering Technology

#### 2. Malzeme ve Yöntem

Bu çalışmada Al-Mg-Al<sub>2</sub>O<sub>3</sub> kompozitler toz metalürjisi yöntemiyle üretilmiştir. Üretilen kompozitlerin görünür porozite değerleri yapay sinir ağları yöntemiyle incelenmiştir. Kompozitlerin yoğunlukları, Arşimet prensibine göre değerlendirilmiştir. Teorik yoğunluklar ve ölçülen yoğunluklar kullanılarak görünür porozite değerleri hesaplanmıştır [9]. Daha sonra görünür porozite değerleri yapay sinir ağları yöntemi kullanılarak analiz edilmiştir. Kullanılan giriş ve çıkış parametreleri Tablo 1'de verilmiştir.

Al <sub>2</sub> O <sub>3</sub>	Basınç (MPa)	Sinterleme sıcaklığı (ºC)	Sinterleme süresi (dk)	Görünür Porozite (%)
15	260	300	30	24,53
15	260	300	60	28,56
15	260	300	90	25,74
15	260	400	30	19,08
15	260	400	60	20,89
15	260	400	90	25,55
15	260	500	60	22,80
15	260	500	90	27,96
15	520	300	30	15,02
15	520	300	60	18,37
15	520	300	90	18,79
15	520	400	30	18,35
15	520	400	60	15,50
15	520	400	90	17,99
15	520	500	30	26,94
15	520	500	60	21,78
15	520	500	90	20,41
30	260	300	30	25,86
30	260	300	60	27,17

**Tablo 1.** Sistemde kullanılan parametreler

	The International Conference of Mee								
20	260	200	00						
30	260	300	90	24,46					
30	260	400	30	21,91					
30	260	400	60	27,63					
30	260	400	90	27,07					
30	260	500	60	26,45					
30	260	500	90	27,71					
30	520	300	30	22,23					
30	520	300	60	20,18					
30	520	300	90	21,64					
30	520	400	30	18,66					
30	520	400	60	20,05					
30	520	400	90	20,60					
30	520	500	30	27,47					
30	520	500	60	25,70					
30	520	500	90	21,99					

ICME

Engineering Technology

Bu değerler 0-1 aralığında =(mak-min)/fark ile normalize edilmiştir. Yapay sinir ağları yaklaşımında verilerin %60 eğitim geri kalanı test verisi olarak ayarlanmıştır. Yapay sinir ağlarında 4 giriş ve 1 sonuç verileri, 1 gizli tabaka içinde 10 nöron yapısı ile Sigmoid LM fonksiyonu kullanılmıştır.

### 3. Bulgular ve Tartışma

UWE Bristol

Yapay sinir ağları yaklaşımında sistemin analizi ortalama karasel hata (MSE), ortalama mutlak hata (MAE) ve korelasyon katsayısı (R) kullanılarak değerlendirilmiştir. Şekil 1-4'de eğitim datalarına ait deneysel ve ANN sonuçları gösterilmiştir.





Şekil 2. Eğitim setine ait MSE değerleri a) Normalize edilmiş b) Normalize edilmemiş





UWE Bristol

Şekil 3. Eğitim setine ait MAE değerleri a) Normalize edilmiş b) Normalize edilmemiş



Şekil 4. Eğitim setine ait yüzde hata değişimi

Deneysel ve ANN sonuçlarına ait porozite değerleri birbiri ile uyum içerisindedir. Normalize edilmiş ve edilmemiş MSE ve MAE değerleri oldukça düşük olduğu görülmüştür. Eğitim testine ait maksimum hata %11 olarak elde edilmiş olup R değeri 0.954572 olarak hesaplanmıştır. Bu değerler eğitim serinde güvenilir sonuçlar elde edildiğini göstermektedir. Şekil 5-8'de test datalarına ait deneysel ve ANN sonuçları gösterilmiştir.



UWE Bristol







Şekil 6. Eğitim setine ait MSE değerleri a) Normalize edilmiş b) Normalize edilmemiş





Şekil 7. Eğitim setine ait MAE değerleri a) Normalize edilmiş b) Normalize edilmemiş



Şekil 8. Eğitim setine ait yüzde hata değişimi

Test seti deneysel ve ANN sonuçlarına ait porozite değerleri birbiri ile uyum içerisinde olduğu görülmektedir. Normalize edilmiş ve edilmemiş MSE ve MAE değerleri kabul edilebilirdir. Test setine ait maksimum hata %24 olarak elde edilmiş olup R değeri 0,799893 olarak hesaplanmıştır. R<sup>2</sup> değeri (0,6398) ise sistem tahmin sonucunu göstermektedir. Bu değerler test serinde güvenilir sonuçlar elde edildiğini göstermekle birlikte eğitim testine göre daha düşük analiz sonuçları oluşması deneysel değişkenlere bağlanabilir ve sistem parametrelerinde değişiklik yapılarak daha yüksek oranda tutarlılık elde edilebilir.



### 4. Sonuçlar

Bu çalışmada toz metalürjisi yöntemiyle üretilen Al-Mg-Al<sub>2</sub>O<sub>3</sub> kompozitlerin görünür porozite değerleri yapay sinir ağları yöntemiyle incelenmiştir. Kompozitlerin yoğunlukları, Arşimet prensibine göre değerlendirilmiştir. Teorik yoğunluklar ve ölçülen yoğunluklar kullanılarak görünür porozite değerleri hesaplanmıştır. Daha sonra görünür porozite değerleri yapay sinir ağları yöntemi kullanılarak analiz edilmiştir. Eğitim testine ait maksimum MSE=6, MAE=2,5, % hata=11 ve R=0,95 olarak belirlenirken, Test seti için maksimum MSE=25, MAE=5, R değeri 0,799893, ve % hata=24 olarak ve R<sup>2</sup> değeri ise 0,6398 olarak hesaplanmıştır. Bu sonuçlar sistemin yeteri kadar güvenilir olduğunu ve sistem değişkenleri kullanılarak daha iyi sonuçlar elde edilebileceğini göstermiştir.

The International Conference of Materials and Engineering Technology

#### Referanslar

- 1. Di Nola, A., Soft Computing, A Fusion of Foundations, Methodologies and Applications. 2007, Springer, New York.
- 2. Bhadeshia, H.K.D.H., Neural Networks in Materials Science. ISIJ International, 1999. 39: p. 966--979.
- 3. Haykin, S.S., Neural Networks: A Comprehensive Foundation. 1994: Macmillan.
- 4. Neural networks modeling of shear strength of SFRC corbels without stirrups. Applied Soft Computing, 2010. 10(1): p. 135.
- 5. Fatigue response and constitutive behavior modeling of poly(ethylene terephthalate) unreinforced and nanocomposite fibers using genetic neural networks. Polymer Composites, 2012: p. n/a.
- 6. Altinkok, N. and R. Koker, Modelling of the prediction of tensile and density properties in particle reinforced metal matrix composites by using neural networks. Materials & Design, 2006. 27(8): p. 625-631.
- Amirjan, M., et al., Artificial Neural Network prediction of Cu–Al2O3 composite properties prepared by powder metallurgy method. Journal of Materials Research and Technology, 2013. 2(4): p. 351-355.
- 8. Çetinel, H., et al., Artificial neural network-based prediction technique for wear loss quantities in Mo coatings. Wear, 2006. 261(10): p. 1064-1068.
- 9. Kurt, M., SiC ve Al2O3 takviyeli Al-Mg kompozitin toz metalurjisi yöntemi ile üretiminin araştırılması, in Metallurgical Engineering. 2017, Fırat University.

## BURSTING STRENGTH ANALYSIS OF KNITTED FABRICS FROM MULTIFILAMENT/MICROFILAMENT CORE-SPUN YARNS

The International Conference of Materials and Engineering Technology

## ESİN SARIOĞLU<sup>\*1</sup>, GÜLBİN FİDAN<sup>2</sup>

<sup>1</sup> Gaziantep University, Faculty of Fine Arts, Department of Fashion and Textile Design, Gaziantep, TURKEY. <sup>2</sup> Gaziantep University, Naci Topçuoglu Vocational School, Machinery and Metal Technologies Department, Gaziantep, TURKEY.

#### Abstract

A microfiber is defined as the filament/fiber fineness lower than 1 dtex linear density. Microfibers have several advantages such as very soft hand, shrink resistance, protection against weather (wind, cold, rain etc), superabsorbent, environmentally friendly etc. Microfibre can be produced as staple or filament form according to the usage area. The core spun yarn production method is used to obtain yarn with optimum properties from two or more different fibers having different properties. Basically, staple sheath fiber is wound around the filament core component. This paper covers the bursting strength and distention of knitted fabrics produced from multifilament/microfilament corespun yarns. For this purpose five different type of drawn textured polyester filaments at different filament fineness (3.06, 1.15, 0.76, 0.57 and 0.33 dtex) were used to produce cotton covered Ne 16/1 core-spun yarns. Ring spinning system was used for the production of core-spun yarns and all parameters were kept constant. Besides, 100% cotton ring spun yarn was also produced as control group of the study. Single jerseys knitted fabrics were produced at three different fabric density (tight, medium and loose) from these yarn samples. Bursting strength and distention properties of fabric samples were determined and results were evaluated statistically. Results showed that filament fineness and fabric density have statistically significant effect on bursting strength and distention properties at 95% confidence interval.

Keyword: microfilament, core-spun yarn, single jersey fabric, bursting strength.

#### **1. Introduction**

Core-spun yarns are widely used in the textile industry. Core-spun yarn consists of two components such as sheath and core. Basically staple sheath fiber is wrapped around the core component. The core component is composed of staple yarn or filament. Sheath fiber is responsible for comfort and the core component contributes strength and functional properties [1-2]. According to the core component type a core-spun yarn is classified as elastic core-spun (elastane core) and rigid core-spun yarns (filament or staple core). Core spun yarns can be produced by various spinning systems [1-9]. Recent years the properties of filament core-spun yarns have been investigated by various researchers [10-14]

In the scope of this study reveals the filament fineness parameter of core-spun yarns and fabric density parameter on bursting strength and distention of single jersey knitted fabrics. Results were statistically investigated in order to determine the significant effect of parameters analyzed.



### 2. Materials and Methods

As core materials, drawn textured polyester filament yarns with different filament fineness were used and the physical properties of polyester filament yarns are indicated in Table 1. As a sheath fiber combed cotton (Co) fiber with 30 mm length, 4.5 micronaire and 34 g/tex strength was chosen.

Fiber Classification	Decitex/	Fiber Count	Tenacity	Elongation
	Filament	(dtex/f)	(cN/dtex)	(%)
Medium	110/36	3.06	4.11	20.97
Fine	110/96	1.15	3.77	21.04
	110/144	0.76	3.69	18.85
Micro	110/192	0.57	3.77	17.16
	110/333	0.33	4.17	19.44

**Table 1.** Physical properties of polyester filament yarns [2].

All Ne 16/1 core-spun yarn types and 100% Co yarn were produced with ring spinning system. Production parameters ( $\alpha e=3.9$ , spindle speed 8000 rpm, combed cotton roving count Ne 0.6) were kept constant in order to evaluate only the filament fineness variable. The physical and mechanical properties of Ne 16/1 yarn samples are given in Table 2.

	Table 2. Yarn properties.										
Filament Fineness (dtex)	Tenacity (grf/den)	Elongation (%)	Unevenness (CVm%)	Thin Places (-40%/km)	Thick Places (+50%/km)	Neps (+200%/km)	Hairiness (Uster H)				
100% Co	1.88	7.41	10.04	1.71	4.08	5.53	6.73				
3.06	1.72	9.03	9.36	0.00	6.84	7.89	7.25				
1.15	1.70	8.68	9.29	0.26	4.34	6.71	8.03				
0.76	1.89	8.87	9.26	0.13	3.90	4.63	7.44				
0.57	1.75	8.24	9.23	0.13	7.63	6.71	7.44				
0.33	2.05	9.22	9.30	0.00	8.75	9.03	7.59				

Knitted fabrics from these yarns were manufactured with different level of fabric density such as tight, medium and loose by a 3.5" gauge, 22 fein and one feeder sample circular knitting machine at  $20\pm2$  rev/min production speed. For further process, fabrics were scoured and dyed at the same process parameters.

Before bursting strength and distention test, fabrics were conditioned at standard atmosphere condition for 24 hours. Bursting strength test was achieved with James Heal bursting strength tester according to the BS EN ISO 13938-2 standard at 50 cm<sup>2</sup> test area in order to determine bursting strength and distention in a mean time a test specimen to burst falls within  $(20\pm5)$  seconds [15]. Results were evaluated statistically at 95% confidence interval by means of SPSS package program. Meanwhile, Student-Newman-Keuls (SNK) multiple comparison analysis was achieved in order to evaluate the difference between subgroups of filament fineness and fabric density.

### 3. Results and Discussion

The structural properties of single jersey knitted fabric are shown in Table 3. In terms of filament fineness and fabric density variable on bursting strength and distention, Figure 1 illustrates bursting strength bar graphs.

Table 3. Structural properties of fabric samples.							
Filament Fineness (dtex)	Fabric Density	Thickness (mm)	Weight (gsm)	Wale Density (WPC)	Course Density (CPC)		
100% Co		0.65	247.60	13.67	16.667		
3.06		0.68	265.54	13.00	17.333		
1.15	Ticht	0.69	263.48	13.00	17.333		
0.76	Tight	0.68	258.78	13.00	17.667		
0.57		0.69	265.54	13.00	18.000		
0.33		0.66	255.54	13.33	17.333		
100% Co		0.70	232.31	13.00	15.667		
3.06		0.74	244.37	13.00	15.667		
1.15	Madian	0.70	235.84	12.67	15.000		
0.76	Medium	0.70	235.84	13.00	15.000		
0.57		0.68	232.60	12.00	15.000		
0.33		0.67	227.31	12.67	15.000		
100% Co		0.73	207.61	12.00	14.000		
3.06		0.72	223.49	12.33	13.333		
1.15	Tasas	0.71	212.61	12.33	12.667		
0.76	Loose	0.73	206.43	12.33	12.000		
0.57		0.68	202.32	12.67	12.000		
0.33		0.68	192.32	12.33	12.333		

As seen in Figure 1, from tight to loose fabric density bursting strength of fabric samples also decreases. By increasing fabric density leads to also rising yarn density in a unit area (increasing wale and course density) of the fabric. When filament fineness variable is taken into consideration, from medium (3.06 dtex) to fine (1.15 dtex) filament fineness, the bursting strength was determined as slightly decrease. This can due to the fact that filament tenacity affects fabric strength directly (Table 1). Besides, it is seen that the bursting strength of knitted fabric made from microfilament core-spun yarns increases when filament fineness. Variance analysis result of bursting strength is shown in Table 4.



Figure 1. Bursting strength of fabric samples

Table 4. V	Variance analysi	s test results of	bursting	strength.
------------	------------------	-------------------	----------	-----------

Source	Sum of Squares	df	Mean Square	$\mathbf{F}$	Sig.
Corrected Model	31181.372 <sup>a</sup>	17	1834.198	22.650	0.000
Intercept	5699395.831	1	5699395.831	70379.163	0.000
Filament fineness (A)	5531.145	5	1106.229	13.660	0.000
Fabric density (B)	24014.791	2	12007.396	148.274	0.000
A*B	1635.435	10	163.544	2.020	0.060
Error	2915.327	36	80.981		
Total	5733492.530	54			
Corrected Total	34096.699	53			
a. R Squared = $0.914$ (Adjusted R Squared = $0.874$ )					

As illustrated in Table 4, both filament fineness and fabric density have statistically significant effect on bursting strength of single jersey fabric. Also, it was determined that the interaction of filament fineness and fabric density (A\*B) has no statistically significant effect on bursting strength at 0.05 confidence level. SNK test result belongs to filament fineness and fabric density variables for bursting strength are indicated in Table 5.

#### Table 5. SNK test results for bursting strength.

Parameter:	SNK	Parameter:	SNK
<b>Filament Fineness</b>	Ranges	Fabric Density	Ranges
100% Cotton	311.6667a	Loose	299,2722a
1.15	318.5444a	Medium	324,4333b
0.76	319.6222a	Tight	350,9222c
3.06	327.5222b		
0.57	328.2000b		
0.33	343.7000c		

Technology

tials and Engineering

According to the Table 5, we can say that there is no difference between bursting strength of single jersey fabrics made of 100% Co, 1.15 and 0.76 dtex filament finenesses. And, it was determined that bursting strength of fabric with cotton covered 3.06 and 0.57 dtex drawn textured polyester are similar that is there is no difference between two variables. Among these, fabric from 0.33 dtex core spun yarn differs from others and the highest bursting strength value was obtained from this fabric. When the fabric density variable is taken into consideration, all parameters were found to be different from each other.

The International Conference

The bursting distention bar graphs of knitted fabrics are shown in Figure 2. The highest bursting distention of 100% Co knitted fabrics with different fabric density was obtained from loose fabric density. In terms of filament fineness, single jersey fabric made of 1.15 and 0.33 dtex drawn textured polyester filament core-spun yarns show similar trend from tight to loose fabric density. According to the variance analysis result, all parameters and the interaction of parameters (A\*B) are found to have a statistically significant effect on bursting distention of single jersey knitted fabric (Table 6).



Figure 2. Bursting distention of fabric samples

Source	Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	149.675 <sup>a</sup>	17	8.804	8.179	0.000
Intercept	110622.682	1	110622.682	102763.200	0.000
Filament fineness (A)	76.837	5	15.367	14.276	0.000
Fabric density (B)	10.663	2	5.332	4.953	0.013
A*B	62.174	10	6.217	5.776	0.000
Error	38.753	36	1.076		
Total	110811.110	54			
Corrected Total	188.428	53			
a. R Squared = $0.794$ (Ad	)				

Table 6. Variance analysis test results of bursting distention.

Technology

of Materials and Engineering



-	5			
Parameter:	SNK	Parameter:	SNK	
<b>Filament Fineness</b>	Ranges	Fabric Density	Ranges	
0.33	43.8444a	Loose	44.6333a	
0.57	44.7889a	Medium	45.5500b	
0.76	44.9667a	Tight	45.6000b	
1.15	45.0444a			
100% Cotton	45.1778a			
3.06	47.7444b			

#### **Table 7.** SNK test results for bursting distention.

Results from SNK analysis revealed that there is no difference between filament fineness in terms of bursting distention except 3.06 dtex filament fineness (Table 7). In addition, results showed that the bursting distention of fabric with medium and tight fabric density does not differ statistically.

#### 4. Conclusions

In this study, core-spun yarns composed different filament fineness i.e. medium, fine and microfilament were manufactured by modified ring spinning system. Single jersey knitted fabric samples were produced from these yarns at different fabric density. Bursting strength and distention properties were determined and evaluated statistically.

Results are summarized below;

- The highest bursting strength of single jersey fabric was obtained at tight fabric density. Between filament fineness variables including 100% Co yarn, fabric with 0.33 dtex core-spun yarn showed the highest bursting strength. It was found that filament fineness and fabric density had statistically significance effect on bursting strength.
- Results showed that the bursting distention of fabrics with core-spun yarns differ from 100% Co yarn at different fabric density from tight to loose. The highest bursting distention was obtained at tight and medium fabric density according to the SNK results at 0.05 confidence level. According to the SNK results it was determined that there is no difference between filament fineness variables except 3.06 dtex.

#### References

- [1] Shi, FJ and Xuling J, Modelling the Tensile Properties of Modal/Polyurethane Core spun Stretch Yarn, Fibres & Textiles in Eastern Europe. 2012, 20 (3 /92):30-32.
- [2] Sarioğlu, E and Babaarslan, O, A Study on Physical Properties of Microfilament Composite Yarns, Journal of Engineered Fibers and Fabrics. 2016, 11(3):90-98.
- [3] Pourahmad, A and Johari, MS, Comparison of the properties of Ring, Solo, and Siro Corespun Yarns, The Journal of the Textile Institute. 2011, 102 (6):540-547.
- [4] Hua, YR, Yuan, X and Yuan, WS, Comparison and Analysis of Rotor-spun Composite Yarn and Sirofil Yarn, Fibres & Textiles in Eastern Europe. 2010, 18 (1/78):28-30.
- [5] Örtlek, HG, Influence of Selected Process Variables on the Mechanical Properties of Corespun Vortex Yarns Containing Elastane, Fibres & Textiles in Eastern Europe. 2006, 14 (3 /57):42-44.



The International Conference of Materials and Engineering Technology

- [7] Dhouib, AB, El-Ghezal, S and Cheikhrouhou, M, A Study of the Impact of Elastane Ratio on Mechanical Properties of Cotton Wrapped Elastane-core Spun Yarns, Journal of Textile Institute. 2006, 97 (2):167172.
- [8] Ramachandran, R and Vigneswaran, C, Tenacity and Breaking Extensiton of Cotton Covered Copper Open-end Friction-spun Yarns, Indian Journal of Fiber & Textile Research. 2009, 34:179-182.
- [9] Altas, S and Kadoglu, H, The Influence of Some Spinning Parameters on Strength Properties of Core Filament Dref-3 Yarns, Tekstil ve Konfeksiyon. 2009, 2:93-96.
- [10] Lou, CW, Process of Complex Core Spun Yarn Containing a Metal Wire, Textile Research Journal. 2005, 75(6):466-473.
- [11] Shanbaz, B, Nawaz, SM and Ali, R, Spinning Performance and Yarn Properties of Multiple Filament Polyester/Cotton Core Yarn, Pakistan Journal of Applied Sciences. 2002, 2 (3):324-326.
- [12] Rameshkumar, C, Rengasamy, RS and Anbumani, N, Studies on Polyester/Waste Silk Corespun Yarns and Fabrics, Journal of Industrial Textiles. 2009, 38 (3):191-203.
- [13] Jeddi, AA, Johari, MS and Merati, AA, A Study of the Structural and Physical Properties of Cotton-covered Nylon Filament Core-spun Yarns, The Journal of Textile Institute. 1997, 88(1):12-20.
- [14] Erez, E and Çelik, P. A Research on The Parameters of The Affecting Yarn Properties of Cotton-Polyester Rigid Core-Spun Yarns, Tekstil ve Konfeksiyon. 2014, 24(2):195-201.
- [15] ISO 13938-2:1999- Textiles-Bursting properties of fabrics-Part 2: Pneumatic method for determination of bursting strength and bursting distension.

# COMPARISON OF FIVE DEVELOPED POWER CYCLES IN THE FRAME OF WASTE HEAT RECOVERY

The International Conference of Materials and Engineering Technology

### ALPEREN TOZLU<sup>1</sup>, AYŞEGÜL ABUŞOĞLU<sup>2</sup>, EMRAH ÖZAHİ<sup>\*2</sup>

<sup>1</sup>Bayburt University, Engineering Faculty, Mechanical Engineering Department, Bayburt, TURKEY. <sup>2</sup>Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY.

#### Abstract

A thermodynamic comparison of five developed power cycles which are gas turbine (GT), Kalina (KAL), organic Rankine (ORC), gas turbine-Kalina (GT-KAL) and gas turbine-organic Rankine (GT-ORC) fed by the waste heat of an actual power plant system in Gaziantep is presented in this paper. The waste heat has a temperature and a pressure of 566 °C and 1.9 bar, respectively. In order to compare the thermodynamic performance of the power cycles, various parametric studies were performed by using the effect of the values of the pressure ratio of the cycles, the temperature of the exhaust gas, the logarithmic mean temperature difference of the heat exchangers and the turbine inlet and the outlet pressures in the given ranges. The net power output of GT, KAL, ORC, GT-KAL and GT-ORC were found to be 1095 kW, 955 kW, 585 kW, 1508 kW and 1594 kW, respectively. The thermodynamic results showed that the GT-KAL cycle has the best thermodynamic performance in terms of energetic and exergetic efficiencies such as 19.71% and 40.53%, respectively, as well as in terms of the highest net power production of 1594 kW from the available exhaust gas.

**Keyword:** waste heat recovery, gas turbine, Kalina, organic Rankine, energetic efficiency, exergetic efficiency.

#### **1. Introduction**

There are many parameters in power plants that should be take into account for improvements of system efficiencies. The efficiency improvement is a big problem for the companies, which are not only related with technical reasons, but also it is fundamentally related with economic reasons. Due to this, all possibilities to improve the total efficiencies of the systems should be utilized. Therefore, it is possible to develop thermodynamic systems with power production methods that can be added to existing power plants. In this manner, the gas turbine, the organic Rankine and the Kalina cycles are adapted to an actual power plant, which is located in Gaziantep in order to increase its total efficiency. In order to power production, all gas turbine systems are operated by means of Brayton cycle principles.

Gas turbines can be classified into three groups, which are open cycle in which air is working fluid, closed cycle with air or other fluid as working fluid and semi-closed cycle. In addition to this, heat sources such as fossil, biomass, nuclear and solar are other parameters for classification of closed cycle gas turbines. Low-grade heat sources of gas turbine and diesel engine exhausts are recently considered to supply global energy requirement due to its significant energy potential [1-4].

Organic Rankine cycle (ORC) is also an important low-grade thermal energy recovery technology because of its small-scale feature. ORC can be used to all kinds of low-temperature heat sources including geothermal energy, solar energy, biomass energy, and especially waste heat energy [5-7].

The International Conference of Materials and Engineering Technology

In addition to these, two power cycles, Kalina cycle can be considered as another alternative power production method for increasing the overall system efficiency. Kalina cycle was designed for a new cycle called as the Kalina using a bottoming cycle instead of the Rankine cycle in combined cycle power plants in 1989. Usage of an ammonia-water mixture instead of water as working fluid was a possible way to improve the efficiency of steam turbine processes [8-9].

#### 2. Materials and Methods

In this study, renewable and sustainable models adapted to the actual system are considered from the point of view of their thermodynamic analyses. All adapted models are designed so as to produce more electrical power in addition to the existing power production capacity of the Gaziantep Municipal Solid Waste Power Plant by utilizing the exhaust gases of the plant as a heat source. The temperature, pressure and mass flow rate of the exhaust gases of the plant are 566.7 °C, 1.9 bar and 16 kg/s, respectively. System descriptions of all models are given and then thermodynamic analyses of all models are performed with respect to their design parameters [1, 4, 7]. The schematics of designed models are given in figures from 1 to 5.

A gas turbine heat exchanger (GTHE), a gas turbine (GT), a recuperator (REC), a low pressure compressor (LPC), a high pressure compressor (HPC), a precooler (PRE) and an intercooler (INT) are used for the gas turbine cycle. Organic Rankine cycle is consisted of an evaporator (EVAP), an ORC turbine (OT), an ORC condenser (OC) and an ORC pump (OP). Lastly, in order to create the Kalina cycle, a generator (GEN), a separator (SEP), a turbine (KT), a valve (VAL), a low temperature recuperator (LTR), a condenser (KCON), a pump (KP) and a high temperature recuperator (HTR) are used [1, 4, 7].

The last two cycles, GT-ORC and GT-KAL, are composed of gas turbine, Kalina and organic Rankine cycles. The working fluids used in gas turbine, organic Rankine and Kalina cycles are supercritical CO<sub>2</sub>, toluene and ammonia-water mixture, respectively.



Figure 1. Schematic layout of the gas turbine cycle



Figure 2. Schematic layout of the organic Rankine cycle



Figure 3. Schematic layout of the Kalina cycle


Figure 4. Schematic layout of the gas turbine-organic Rankine cycle



Figure 5. Schematic layout of the gas turbine-Kalina cycle

# 3. Results and Discussion

UWE Bristol

The heat transfer rates, the work, the exergy destructions and the exergy efficiencies of all subcomponents are evaluated by means of the governing equations related with continuity, energy and exergy equations. Thus, the energy and exergy efficiencies of each cycle are evaluated [1, 4, 7]. The thermodynamic results for each cycle are given in table from 1 to 5.

The International Conference

of Materials and Engineering Technology

Table 1. Thermodynamic results of the gas turbine cycle								
Component	$\dot{Q}_{(kW)}$	$\dot{W}_{(kW)}$	$\dot{E}x_{F}$ (kW)	$\dot{E}x_{P}$ (kW)	$\dot{E}x_{D}(\mathrm{kW})$	E (%)		
GTHE	2730	-	1946	1391	555.6	71.5		
GT	-	1463	1517	1463	53.69	96.5		
REC	5726	-	1948	1880	68.12	96.5		
PRE	723.3	-	75.87	14.23	61.64	18.8		
LPC	-	231.5	231.5	202.6	28.97	87.5		
HPC	-	136.7	136.7	118.3	18.45	86.5		
INT	1394	-	186.9	49.82	137.1	26.7		
	Energy efficiency (%)		34.1					
GI System	Exergy effic	iency (%)		56.3				

**Table 2.** Thermodynamic results of the organic Rankine cycle

Component	$\dot{Q}$ (kW)	Ŵ (kW)	$\dot{E}x_F$ (kW)	$\dot{E}x_{P}$ (kW)	$\dot{E}x_D$	$\mathcal{E}(\%)$	
EVAP	7226	-	3725	2294	1430	61.6	
OT	-	590.8	647.7	590.8	56.91	91.21	
OC	5558	-	1652	168.9	1483	10.22	
OP	-	6.237	6.237	5.53	0.7062	88.68	
ODC System	Energy efficiency (%)			8.09			
OKC System	Exergy effi	Exergy efficiency (%)	15.69				

**Table 3.** Thermodynamic results of the Kalina cycle
  $\dot{Q}$  (kW)  $\dot{W}$  (kW)  $\dot{E}x_F$  (kW)  $\dot{E}x_{P}$  (kW)  $\dot{E}x_{D}$  (kW) Component  $\mathcal{E}(\%)$ GEN 996.4 8197 3952 2956 74.79 KΤ 976 1056 976 80.17 92.41 -LTR 954.2 52.72 901.5 5.525 5836 \_ KCON 7.749 20.99 227 1.627 6.123 \_ KP 21.4 21.36 13.65 7.711 63.89 \_ HTR 3535 1049 355 693.6 33.85 -Energy efficiency (%) 11.65 KC Exergy efficiency (%) 24.15

https://ticmet.org

			Ŭ	<u> </u>		
Component	$\dot{Q}$ (kW)	$\dot{W}$ (kW)	$\dot{E}x_F$ (kW)	$\dot{E}x_{P}$ (kW)	$\dot{E}x_D$ (kW)	$\mathcal{E}(\%)$
GTHE	2758	-	1964	1405	559.3	71.52
GT	-	1509	1565	1509	55.65	96.44
REC	5734	-	1949	1857	92.05	95.28
PRE	724.3	-	72.89	14.25	58.64	19.55
LPC	-	234.7	234.7	205.3	29.37	87.48
HPC	-	142.2	142.2	123	19.25	86.46
INT	1403	-	187.3	50.15	137.1	26.78
EVAP	4791	-	1959	1233	725.8	62.94
ОТ	-	469.1	536.2	469.1	67.06	87.49
CON	4406	-	779.1	134.2	644.9	17.22
OP	-	93.83	93.83	82.3	11.53	87.72
	Energy effi	ciency (%)		1	8.76	
GT-ORC	Exergy effi	ciency (%)		3	8.43	

Table 4. Thermodynamic results of the gas turbine-organic Rankine cycle

The International Conference

of Materials and Engineering Technology

UWE Bristol

**Table 5.** Thermodynamic results of the gas turbine-Kalina cycle

Component	$\dot{Q}$ (kW)	$\dot{W}$ (kW)	$\dot{E}x_F(\mathrm{kW})$	$\dot{E}x_{P}$ (kW)	$\dot{E}x_D$ (kW)	$\mathcal{E}(\%)$
GTHE	2758	-	1964	1405	559.3	71.52
GT	-	1509	1565	1509	55.65	96.44
REC	5734	-	1949	1857	92.05	95.28
PRE	724.3	-	72.89	14.25	58.64	19.55
LPC	-	234.7	234.7	205.3	29.37	87.48
HPC	-	142.2	142.2	123	19.25	86.46
INT	1403	-	187.3	50.15	137.1	26.78
GEN	4840	-	1968	1676	291.8	85.17
KT	-	469.7	496.7	469.7	26.96	94.57
HTR	1705	-	614.9	114.9	500.1	18.68
LTR	2177	-	347.9	109	238.8	31.34
КР	-	8.506	8.506	7.847	0.6593	92.25
KCON	899.3	-	70.19	6.446	63.74	9.184
CT KAL Cuelo	Energy efficiency (%)			19		
GT IMIL Cycle	Exergy efficiency (%)			40		

#### 4. Conclusions

The renewable and the sustainable models that can be adapted to the actual system in order to utilize the exhaust gas of the Gaziantep Municipal Solid Waste Power Plant as an energy source have been analyzed in terms of thermodynamic aspects.

The International Conference of Materials and Engineering Technology

The results are summarized as follows;

- The net power output, the energy and the exergy efficiencies of GT cycle were found to be 1095 kW, 34.1 % and 56.3 %, respectively.
- The net power outputs obtained from the ORC system in using toluene is 584.6 kW. The exergy and energy efficiencies of the ORC system is calculated as 15.69 % and 8.09 %, respectively.
- The thermodynamic analysis of Kalina cycle has shown that, the net power output, the energy and the exergy efficiencies are 954.6 kW, 11.65 % and 24.15 %, respectively.
- As a result of the thermodynamic analysis carried out for GT-ORC, the net power output, the energy and the exergy efficiencies are found to be 1508 kW, 18.76 % and 38.43 %, respectively.
- The thermodynamic analysis of GT-KAL cycle, the net power output, the energy and the exergy efficiencies are calculated as 1594 kW, 19.71 % and 40.53 %, respectively. This power output puts another 28.16 % power on top of the existing power output of the system.

When all cycles are considered from the point of thermodynamic relations, the results show that the GT-KAL cycle is the best performance in terms of energetic and exergetic efficiencies such as 19.71% and 40.53%, respectively. In addition to this, it is concluded that the highest net power production, which is found to be 1594 kW, can also be ensured by GT-KAL cycle from the available exhaust gas.

#### References

- [1] Tozlu A., Abusoglu A. and Ozahi E., Thermoeconomic analysis and optimization of a Recompression supercritical CO2 cycle using waste heat of Gaziantep Municipal Solid Waste Power Plant, Energy,143, 168-180, 2018.
- [2] Y.A, S. Bae, M. Kim, S. Cho, S. Baik, J. Lee, and J. Cha, Review of supercritical CO2 power cycle technology and current status of research and development, Nuclear Engineering and Technology, 47, 647-661, 2015.
- [3] Akbari A.D.and Mahmoudi S.M.S., Thermoeconomic analysis & optimization of the combined supercritical CO2 (carbon dioxide) recompression Brayton/organic Rankine cycle, Energy, 78, 501-512, 2014.
- [4] Tozlu A., Abusoglu A. and Ozahi E., Thermodynamic and thermoeconomic analyses of an organic Rankine cycle adapted gas turbine cycle using S-CO2, Journal of the Faculty of Engineering and Architecture of Gazi University, 33/3, 917-928, 2018.
- [5] U. Drescher, and D. Brüggemann, Fluid selection for the organic Rankine cycle (ORC) in biomass power and heat plants, Applied Thermal Engineering, 27, 223–228, 2007.



The International Conference of Materials and Engineering Technology

UWE Bristol

- [7] Ozahi E., Tozlu A. and Abusoglu A., Thermoeconomic multi-objective optimization of an organic Rankine cycle (ORC) adapted to an existing solid waste power plant, Energy Conversion and Management, 168, 308-319, 2018.
- [8] X. Zhang, M. He and Y. Zhang, A review of research on the Kalina cycle, Renewable and Sustainable Energy Reviews 16 (2016) 5309–5318.
- [9] E. Thorin, Power cycles with ammonia-water mixtures as working fluid: Analysis of different applications and the influence of thermophysical properties, Stockholm, Sweden: Royal Institute of Technology (2000).



The International Conference

aterials and Engineering Technology

## Esin SARIOĞLU<sup>1</sup> and Gülbin FİDAN<sup>2\*</sup>

<sup>1</sup> Gaziantep Üniversitesi, Güzel Sanatlar Fakültesi, Moda ve Tekstil Tasarımı Bölümü, Gaziantep/TÜRKİYE.
<sup>2</sup> Gaziantep Üniversitesi, Naci Topçuoğlu Meslek Yüksekokulu, Makine ve Metal Teknolojileri Bölümü, Gaziantep/ TÜRKİYE.

## ÖZET

Tekstil endüstrisinde farklı liflerin özelliklerinden aynı anda faydalanabilmek amacıyla belirli oranlarda karışım yapılması yaygın olarak kullanılan bir yöntemdir. Teknolojik gelişmeler ile birlikte sentetik ve rejenere lif ailesinden birçok farklı lif üretimi mümkün hale gelmiştir. Bu çalışma kapsamında, yeni nesil rejenere selülozik lif grubundan Viloft<sup>®</sup>, ProModal<sup>®</sup> ve Bambu lifleri farklı karışım oranlarında (%67/33, %50/50, %33/67) pamuk lifi ile harmanlanarak aynı üretim koşullarında Ne 30/1 ring iplik numuneleri üretilmiştir. Ayrıca, %100 Viloft<sup>®</sup>, ProModal<sup>®</sup>, Bambu ve Pamuk ring iplik numuneleri üretilmiştir. Üretilen numunelerin, ring iplik makinesinden sonra ve bobinleme işlemi uygulandıktan sonra, kalite kontrol testleri uygulanarak mukavemet, uzama, düzgünsüzlük, iplik hataları ve tüylülük özellikleri belirlenmiştir. Test sonuçları istatistiksel olarak analiz edilerek, karışım oranı, rejenere selülozik lif tipi ve bobinleme işleminin mukavemet, uzama, tüylülük ve düzgünsüzlük özellikleri üzerinde anlamlı bir etkiye sahip olup olmadıkları belirlenmiştir. Test sonuçlarına göre rejenere selülozik lif tipinin muavemet, düzgünsüzlük, iplik hataları ve tüylülük özellikleri bir tekiye sahip olup olmadıkları belirlenmiştir. Test sonuçlarına göre rejenere selülozik lif tipinin muavemet, düzgünsüzlük, iplik hataları ve tüylülük özellikleri belirlenmiştir.

Anahtar Kelimeler: rejerenere lif, ring iplik, Viloft, ProModal, bobinleme işlemi.

## 1. GİRİŞ

Tekstil endüstrisindeki gelişmeler ve artan müşteri talepleri farklı tekstil ürünlerinin geliştirilmesini şart hale getirmiştir. Bu ürünlerden beklenen özellikler sadece ürünün yüksek dayanıma sahip olması veya iyi konfor özelliklerini bünyesinde barındırması değil aynı zamanda moda beklentilerine de cevap verebilecek nitelikte olmasıdır. Bu gibi farklı özellikleri bir arada sağlayan ürünlerin geliştirilmesi, farklı özelliklerdeki liflerin bir arada kullanılması sayesinde mümkün olmaktadır. Bu şekilde iplik ve kumaş üretimi uzun yıllardır yaygın biçimde yapılmaktadır. Rejenere selülozik lifler, yeni iplik tipleri için lif karışımlarında yaygın olarak kullanılmaktadır.



Bu çalışma kapsamında, Viloft®, ProModal®, bambu rejenere lif tipleri pamuk lifi ile farklı oranlarında (%100, %67/33, %50/50 ve %33/67) karıştırılarak sonuçların iplik mukavemeti, uzama değerleri, düzgünsüzlük, hata endeksi ve tüylülük özellikleri üzerinde etkileri istatistiksel olarak (%95 güven aralığında) incelenmiştir.

## 2. MATERYAL VE METOT

#### 2.1. Materyal

Çalışma kapsamında kullanılan rejenere selülozik lif tiplerinin özellikleri Tablo 1'de gösterilmiştir. Bununla birlikte, 30 mm lif uzunluğuna ve 4,5 mikroner lif inceliğine pamuk lif kullanılmıştır. Rejenere selülozik lif tipi, karışım oranı ve bobin prosesinin iplik özellikleri üzerindeki etkilerini belirleyebilmek amacıyla rejenere selülozik lif tipleri olarak kullanılan Viloft<sup>®</sup>, ProModal<sup>®</sup> ve bambu lifleri farklı karışım oranlarında (%67/33, %50/50 ve %33/67) pamuk lifi ile harmanlanmıştır. Ayrıca, %100 Viloft<sup>®</sup>, ProModal<sup>®</sup>, bambu ve pamuk iplikleri aynı üretim parametrelerinde ring iplik eğirme sisteminde üretilmiştir. Ring eğirme sisteminde 10000 rpm hızda, 797 tur/m büküme sahip Ne 30/1 iplik numuneleri kops olarak elde edilmiştir. Daha sonra ise üretilen ipliklere işletme koşullarında bobin prosesi uygulanmıştır.



Hammadde	Doğrusal yoğunluk, dtex	Lif uzunluğu, mm
Viloft®	1,9	38
ProModal®	1,3	38
Bambu	1,5	38

## 2.2. Metot

Bobin prosesi öncesi ve sonrası iplik numunelerine mukavemet, uzama, düzgünsüzlük, hata endeksi ve tüylülük özellikleri ilgili standartlar uygulanarak belirlenmiştir. İplik testleri öncesinde, numuneler 24 saat bekletildikten sonra standart laboratuvar koşullarında ( $20 \pm 2 \circ C$  ve % 65  $\pm 4$ ) kondisyonlanmıştır [11]. İpliklerin tüylülük, düzgünsüzlük ve hata endeksi özellikleri, 2,5 dakika boyunca 400 m/dak test hızında Uster<sup>®</sup> Tester 4 kullanılarak belirlenmiştir [12]. Hata endeksi kalın yer (+%50/km), ince yer (-%50/km) ve neps (+200/km) değerlerinin kümülatif olarak toplanmasıyla elde edilmiştir. Mukavemet ve uzama özellikleri ise Uster<sup>®</sup> Tensorapid cihazı kullanılarak 5 m/dak test hızında ve 500 mm ölçüm uzunluğunda belirlenmiştir [13].

Rejenere selülozik lif tipi, karışım oranı ve bobin prosesinin iplik mukavemet, uzama, düzgünsüzlük, hata endeksi ve tüylülük özellikleri üzerindeki etkilerini belirleyebilmek amacıyla SPSS 22 paket programı kullanılarak %95 güven aralığında varyans analizi (ANOVA) yapılmıştır.

## **3. TARTIŞMA VE BULGULAR**

İplik numunelerine ait mukavemet ve uzama sütun grafiği sırasıyla Şekil 1 ve Şekil 2'de gösterilmiştir.



Şekil 1. İplik numuneleri mukavemet (cN/tex) sütun grafiği



Materials and Engineering Technology

Şekil 2. İplik numuneleri uzama (%) sütun grafiği

Yapılan çalışma sonucunda rejenere selülozik lif tipleri kendi içlerinde değerlendirildiklerinde mukavemet yönüyle en iyi değerin ProModal<sup>®</sup> lifi ile üretilen ipliklerde olduğu görülmüştür. Ardından bambu, pamuk ve Viloft<sup>®</sup> lifleri ile üretilen iplikler sırayla gelmektedir. Pamuk/Viloft<sup>®</sup> karışımlı ipliklerde genel olarak pamuk karışım oranının artmasıyla birlikte mukavemetin arttığı görülmektedir. Bununla birlikte, pamuk/bambu karışımlı ipliklerin mukavemet değerleri incelendiğinde ise %100 pamuk ipliğinden %50/50 pamuk/bambu ipliğine kadar mukavemet değerleri değerinin azaldığı bambu oranının artmasıyla birlikte arttığı gözlemlenmiştir. %100 rejenere selülozik liflerden üretilen iplik mukavemetleri incelendiğinde ise en yüksekten en düşüğe doğru ProModal<sup>®</sup>>bambu> Viloft<sup>®</sup> liflerinden elde edilmiştir. Bununla birlikte kops mukavemet test değerlerinin bobin değerlerine göre bir miktar yüksek olduğu görülmektedir.

Şekil 2'deki uzama sütun grafiği incelendiğinde ise bambu lifi ile üretilen iplikler uzama yönünden en yüksek sonuçları verirken Viloft<sup>®</sup>, ProModal<sup>®</sup> liflerinden üretilen ipliklerin uzama değerlerinin daha düşük olduğu görülmektedir. En düşük uzama değerleri ise pamuk lifinden üretilen ipliklerde tespit edilmiştir. Pamuk/ ProModal<sup>®</sup> ve Pamuk/ Viloft<sup>®</sup> karışım ipliklerde ProModal<sup>®</sup> ve Viloft<sup>®</sup> oranı arttıkça uzama değerlerinin arttığı tespit edilmiştir. Rejenere selülozik liflerden %100 olarak üretilen ipliklerin uzama değerlerinde ise sırasıyla bambu> ProModal<sup>®</sup>> Viloft<sup>®</sup> sonucuna ulaşılmıştır. Bobin prosesinin etkisi incelendiğinde ise kops uzama değerlerinin %100 Viloft<sup>®</sup> ve %100 bambu iplikleri hariç bobin değerlerine göre daha düşük kopma uzamasına sahip oldukları belirlenmiştir.

İplik numunelerine ait düzgünsüzlük sütun grafiği Şekil 3'te verilmiştir.



aterials and Engineering Technology

Şekil 3. İplik numuneleri düzgünsüzlük (CVm%) sütun grafiği

Şekil 3 incelendiğinde, en yüksek düzgünsüzlük değerinin %100 Viloft<sup>®</sup> ipliğine ait olduğu görülmektedir. Farklı karışım oranlarında üretilen pamuk/Viloft<sup>®</sup> ipliklerin düzgünsüzlük değerlerinin Viloft<sup>®</sup> lif oranının artmasıyla birlikte arttığı belirlenmiştir. Ancak bu trend ProModal<sup>®</sup> ve bambu rejenere selülozik lif karışımlı ipliklerde görülmemektedir. Pamuk/bambu karışımlı ipliklerin düzgünsüzlük değerlerinin %50/50 karışım oranında kadar düştüğü ancak bambu karışım oranı >%50'nin üzerine çıktığında ise düzgünsüzlükte artış olduğu belirlenmiştir. Pamuk/ProModal<sup>®</sup> lif karışımlı ipliklerde ise ProModal<sup>®</sup> oranının >%67'nin üzerinde olduğunda iplik düzgünsüzlüğünün arttığı belirlenmiştir. Rejenere selülozik liflerinin %100 olarak üretildiği ipliklerin düzgünsüzlük değerlerinin sırasıyla Viloft<sup>®</sup>>bambu>ProModal<sup>®</sup> olduğu görülmektedir. Bobin prosesinin düzgünsüzlük üzerindeki etkisi incelendiğinde ise kops düzgünsüzlük değerlerinin %67/33, %50/50 ve %33/67 karışım oranlarında genel olarak daha düşük olduğu belirlenmiştir.

Kalın yer, ince yer ve neps iplik hatalarının kümülatif toplanmasıyla elde edilen hata endeksi değerleri Şekil 4'te sütun grafiği olarak gösterilmiştir. Şekil 4 incelendiğinde en yüksek IPI değeri %100 Viloft<sup>®</sup> ipliğinde elde edilmiştir. Pamuk/bambu lif karışımlı ipliklerden en düşük IPI değeri %50/50 oranında elde edilmiştir. Karışımda ProModal<sup>®</sup> ve bambu lif oranının artması ile IPI değerlerinin daha iyi sonuçlar verdiği görülmektedir. Test sonuçları kops ve bobin olarak ayrı ayrı değerlendirildiğinde; IPI değerleri arasında önemli bir fark görülmemektedir.



Şekil 4. İplik numuneleri hata endeksi (IPI) sütun grafiği

İplik numunelerine ait tüylülük sütun grafiği Şekil 5'te gösterilmiştir.



Şekil 5. İplik numuneleri tüylülük (Uster H) sütun grafiği

Şekil 5 incelendiğinde, kops tüylülük değerlerinin genel olarak bobin tüylülük değerlerinden daha düşük olduğu görülmektedir. Diğer bir deyişle bobin prosesi ipliğin tüylülük özelliğini olumsuz etkilemektedir. Rejenere selülozik lif karışımlı pamuk/bambu karışımlı ipliklerin tüylülük değerlerinin bambu lif oranının artmasıyla azaldığı, pamuk/ Viloft<sup>®</sup> karışımlı ipliklerde ise Viloft<sup>®</sup> oranının artmasıyla birlikte tüylülük değerlerinin arttığı gözlemlenmiştir. Pamuk/ ProModal<sup>®</sup> oranı >%50 olduğunda ise arttığı belirlenmiştir.

İstatistiksel analiz neticesinde oluşturulan varyans analiz (ANOVA) sonucu Tablo 2'de verilmiştir.

Parametre/ Özellik	Rejenere Selülozik Lif Tipi (A)	Karışım Oranı (B)	Bobinleme İşlemi (C)	A*B	A*C	B*C	A*B*C
Mukavemet (cN/tex)	0,000*	0,001*	0,000*	0,000*	0,996	0,419	0,508
Uzama (%)	0,103	0,000*	0,230	0,000*	0,634	0,921	0,982
Düzgünsüzlük (CVm%)	0,000*	0,000*	0,509	0,000*	0,009*	0,221	0,747
Hata Endeksi (IPI)	0,000*	0,000*	0,926	0,000*	0,361	0,152	0,963
Tüylülük (Uster H)	0,000*	0,000*	0,000*	0,000*	0,000*	0,012*	0,001*

Tablo 2. İplik özelliklerine ait varyans (ANOVA) analiz sonuçları

Yapılan çalışmanın daha iyi yorumlanabilmesi ve lif tipi, karışım oranı ve bobinleme işleminin mukavemet, uzama, düzgünsüzlük, hata endeksi (IPI) ve tüylülük özellikleri üzerinde anlamlı bir etkiye sahip olup olmadıklarının belirlenmesi açısından yapılan varyans (ANOVA) analizi sonuçlarına göre,

- Rejenere selülozik lif tipi ve karışım oranı mukavemet, uzama, düzgünsüzlük, hata endeksi (IPI) ve tüylülük üzerinde anlamlı bir etkiye sahip olduğu görülmektedir.
- Rejenere selülozik lif tipi ve bobin prosesi düzgünsüzlük ve tüylülük üzerinde anlamlı bir etkiye sahipken; mukavemet, uzama, hata (IPI) üzerine anlamlı bir etki göstermemektedir.
- Karışım oranı ve bobin prosesi sadece tüylülük üzerinde anlamlı bir etkiye sahip olduğu görülmektedir.
- Rejenere selülozik lif tipi, karışım oranı ve bobin prosesi birlikte değerlendirildiğinde sadece tüylülük değeri üzerinde anlamlı bir etkiye sahip olduğu görülmektedir. Bu sonuç aynı zamanda kops ve bobin testleri birlikte değerlendirildiğinde en önemli ve anlamlı farkın tüylülük değeri üzerinde olduğunu göstermektedir. Bu durum bobin prosesinden kaynaklanan ve ipliğin kopstan bobine ek bir aktarma işlemi görmesi nedeniyle ipliğin daha tüylü bir yapı kazanmasından ileri gelmektedir.
- Rejenere selülozik lif tipi ve karışım oranı etkileşimlerinin (A\*B) analiz edilen bütün iplik özellikleri üzerinde anlamlı bir etkisinin olduğu belirlenmiştir. A\*C değişkenlerin etkileşimlerinin ise düzgünsüzlük ve tüylülük özellikleri, B\*C ve A\*B\*C değişkenlerin etkileşimlerinin ise sadece tüylülük özelliği üzerinde anlamlı bir etkisinin olduğu görülmektedir.

#### 4. SONUÇ

Tekstil endüstrisinde farklı özelliklere sahip liflerin özelliklerinden faydalanabilmek amacıyla farklı oranlarda karıştırılması yaygın olarak kullanılan bir yöntemdir. Bu çalışma kapsamında ise pamuk lifi ile farklı özelliklere sahip rejenere selülozik lifleri kullanılarak farklı karışım oranlarında ring iplikler elde edilmiştir. Bu ipliklerin bobin prosesi öncesi ve sonrası iplik özellikleri tespit edilerek sonuçlar istatiksel olarak irdelenmiştir. Analiz sonuçları ise aşağıda maddeler halince özetlenmiştir;

The International Conference of Materials and Engineering Technology

- Pamuk/rejenere selülozik lif karışımlı ipliklerin mukavemet özellikleri incelendiğinde, ProModal<sup>®</sup> ve bambu liflerinin karışım oranlarının artmasıyla birlikte mukavemet özelliklerinde yükselme görülmektedir. Bu durumun aksine Viloft<sup>®</sup> oranının artmasıyla birlikte mukavemet değerlerinin düştüğü tespit edilmiştir. Kopslara ait mukavemet değerlerinin ise bobinden daha yüksek olduğu belirlenmiştir. İpliklerin uzama özellikleri incelendiğinde ise bambu lifinden elde edilen ipliklerin en yüksek değer sahip olduğu görülmektedir. Rejenere selülozik lif tipi, karışım oranı ve bobin prosesinin mukavemet özellikleri üzerinde istatistiksel olarak anlamlı etkilerinin olduğu belirlenmiştir.
- Rejenere selülozik lif tipi, karışım oranının ipliklerin düzgünsüzlük, hata endeksi ve tüylülük özellikleri üzerinde istatistiksel olarak anlamlı etkilerinin olduğu tespit edilmiştir. Ancak bobin prosesinin düzgünsüzlük ve hata endeksi özelliklerine herhangi bir etkisinin olmadığı belirlenmiştir.

#### REFERANSLAR

[1]. Demiryürek, O., Uysaltürk, D. (2014). Statistical Analyses and Properties of Viloft/Polyester and Viloft/Cotton Blended Ring-Spun Yarns, Fibres&Textiles in Eastern Europe, 22, 1(103), 22-27.

[2]. Kilic, M., Okur, A. (2011). The Properties Of Cotton-Tencel And Cotton-Promodal Blended Yarns Spun in Different Spinning Systems, Textile Research Journal, 81(2), 156-172.

[3]. Demiryürek, O., Uysaltürk, D. (2016). Viloft/Polyester Karışımlı Örme Kumaşların Patlama Mukavemeti ve Boncuklanma Özelliklerinin Araştırılması. Tekstil ve Mühendis, 23(102).

[4]. Atasağun, H. G., Öner, E., Okur, A., Beden, A. R. (2015). A Comprehensive Study on The General Performance Properties of Viloft-Blended Knitted Fabrics, The Journal of The Textile Institute, 106(5), 523-535.

[5]. Chattopadhyay, R., Tyagi, G.K., Goyal, A. (2013). Studies of The Hybrid Effect in Mechanical Properties of Tencel Blended Ring-, Rotor-, And Air-Jet Spun Yarns, The Journal of the Textile Institute, 104(3), 339-349.

[6]. North, M. (2011). Engineered Viscose Fibres Delivering Enhanced Wearer Comfort and Fabric Performance, Lenzinger Berichte, 89, 37-42.

[7]. Karthikeyan, G., Nalakilli, G., Shanmugasundaram, O. L., Prakash, C. (2017). Moisture Management Properties of Bamboo Viscose/Tencel Single Jersey Knitted Fabrics, Journal of Natural Fibers, 14(1), 143-152.

[8]. Mahish, S. S., Patra, A. K., Thakur, R. (2012). Functional Properties of Bamboo/Polyester Blended Knitted Apparel Fabrics, Indian Journal of Fiber&Textile Research, 37, 231-237.



[9]. Majumdar, A., Mukhopadhyay, S., Yadav, R. (2010). Thermal Properties of Knitted fabrics Made From Cotton and Regenerated Bamboo Cellulosic Fibres, International Journal of Thermal Sciences, 49(10), 2042-2048.

[10]. Sekerden, F. (2011). Investigation on The Unevenness, Tenacity and Elongation Properties of Bamboo/Cotton Blended Yarns, Fibres&Textiles in Eastern Europe, 19(3), 26-29.

[11]. BS EN ISO 139:2005+A1:2011- Textiles- Standard atmospheres for conditioning and testing.[12]. ISO 16549:2004- Textiles - Unevenness of textile strands - Capacitance method test method.

[13]. BS EN ISO 2062:2009-Textiles-Yarns from packages- Determination of single-end breaking force and elongation at break using constant rate of extension (CRE) tester.



The International Conference of Materials and Engineering Technology

#### **KADİR MERT DÖLEKER<sup>\*1</sup>**

<sup>1</sup> Bartın Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, Bartın, TÜRKİYE.

#### Özet

Termal bariyer kaplamalar (TBCs), yüksek sıcaklık koşullarında metalik altlık malzemeleri termal ve korozif koruma sağlamaktadır. TBC sistemleri, iş parçalarının sıcak bölüm komponentlerinde oksidasyon ve sıcak korozyon hasarlara maruz kalmaktadır. Özellikle sıcak korozyon, oksidasyona nazaran daha erken yıpratıcı hasarlara neden olmaktadır. Bu çalışmada, NiCoCrAlY tozları, yüksek hız oksi yakıt (HVOF) tekniği kullanılarak 316L paslanmaz çelik altlık malzeme üzerine püskürtülmüştür. Kalsiya ile stabilize zirkonya (ZrO<sub>2</sub>-5% CaO, CSZ), bağ kaplaması üretilmiş altlıklar üzerine üst kaplama malzemesi olarak atmosferik plazma sprey tekniği (APS) ile biriktirilmiştir. Üretilen TBC sistemi, 900 ° C' de % 50 V<sub>2</sub>O<sub>5</sub>-50 Na<sub>2</sub>SO<sub>4</sub> karışımı ile 25 saatlik çevrimli sıcak korozyon testlerine tabi tutulmuştur. Sıcak korozyon testlerinin sonunda TBC sistemlerinde korozyon etkisi ve faz analizi stereo mikroskop, taramalı elektron mikroskobu (SEM), SEM - Enerji Dağılımlı X-Ray (EDX), SEM Haritalama ve XRD analizi kullanılarak incelenmiştir.

Anahtar Kelimeler: Sıcak korozyon, Termal bariyer kaplama (TBC), CaO-ZrO<sub>2</sub>, V<sub>2</sub>O<sub>5</sub>, Ergimiş tuz

#### 1. Giriş

Yüksek sıcaklık altında çalışan metalik malzemeler hızlı şekilde oksidasyona ve korozyona uğramaktadırlar. Yüksek sıcaklık etkisini ve bu koşullarda meydana gelecek korozyona karşı önlem amacıyla termal bariyer kaplamalar (TBC) kullanılmaktadır[1].

Bir termal bariyer kaplama sistemi üç farklı tabakadan meydana gelmektedir. İlk tabaka altlık malzemedir. Altlık malzeme olarak kullanım koşullarına göre farklı grup alaşımlar seçilebilir. Yaygın olarak gaz türbinlerinde TBC sistemleri kullanıldıklarından dolayı, burada hem mekanik hem de üstün korozif özellikleri açısından nikel esaslı süper alaşımlar özellikle tercih edilmektedir. Bunun yanında yüksek sıcaklık korozif ortamında çalışan paslanmaz çelik malzeme grupları da tercih edilmektedir. İkinci tabaka metalik bağ kaplamadan oluşmaktadır. Bağ kaplama TBC sisteminde büyük önem arz etmektedir. Çünkü bu tabaka atlıktan önce oksitlenmekte ve penetre olan korozyon ürünlerine karşı direnç göstermelidir. Bunun yanında üst kaplama ve altlık arasındaki adezyon ve termal genleşme uyumsuzluğunu da yine bu tabaka minimize etmektedir. Bu kapsamda en yaygın kullanılan bağ kaplama malzemesi MCrAIY (M= Ni, Co veya ikili kombinasyonları) tipi tozlardır. Seçici olarak alüminyumun oksitlenmesi ile alümina oluşumu sağlamasından ötürü özellikle tercih edilmektedir. Üçüncü tabaka ise üst seramik kaplama tabakasıdır. Burada çok yaygın şekilde (itriya, magnezya,

kalsiya vb. oksitlerle) stabilize edilmiş zirkonya malzeme grubu tercih edilmektedir. Çünkü bu malzeme gruplarının kırılma toklukları yüksek, ısıl iletkenlikleri düşük ve termal genleşme katsayı değerleri diğer seramiklere nazaran oldukça yüksektir [2,3].

The International Conference of Materials and Engineering Technology

Sıcak korozyona karşı alınan en önemli tedbirlerden biride termal bariyer kaplama kullanımıdır. Çünkü sıcak korozyon metalik malzemeler hızlı şekilde etkileşime girerek metalik malzemede ciddi kütle kaybına sebep olmaktadır. Sıcak korozyonda genellikle Na, K, S, CI ve V gibi yakıt kirlilikleri veya çalışma ortamından kaynaklı bulunabilen bu elementlerin bileşiklerinin etkileşimi ile meydana gelmektedir. Sıcak korozyon tip 1 ve tip 2 olarak iki şekilde olarak incelenmektedir. Tip 1 sıcak korozyonu yüksek sıcaklık korozyonu olarak adlandırılırken yaklaşık 850-900 °C ve üzerindeki sıcaklıklarda meydana gelmektedir. Tip 2 sıcak korozyonu ise düşük sıcaklık korozyonu olarak adlandırılmakta ve yaklaşık 600-750 °C civarlarında meydana gelmektedir [4,5]. Na<sub>2</sub>SO<sub>4</sub> ve V<sub>2</sub>O<sub>5</sub> gibi bileşiklerin reaksiyonu neticesinde NaVO<sub>3</sub> fazı oluşarak daha düşük ergime sıcaklıklı (yaklaşık 610 °C) bir bileşik meydana gelmektedir. Meydana gelen bu bileşik ergiyerek mevcut oksitlerle reaksiyona girerek malzemenin korozyonuna sebep olmaktadır [6]. Literatürde yapılan çalışmalarda NaVO<sub>3</sub>ileitriya ile stabilize zirkonya (YSZ) ve Gd<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub>reaksiyona girerek YVO<sub>4</sub>ve GdVO<sub>4</sub> gibi korozyon ürünleri meydana getirmiş ve bunun neticesinde kaplamalarda hasarlar meydana gelmiştir [7–9].

Bu çalışmada kalsiyum oksit ile kısmi stabilize edilmiş zirkonyanın (CSZ) sıcak korozyon davranışı 900 °C sıcaklıkta 5' er saatlik çevrimlerle toplam 25 saat sodyum sülfat ve vanadyum pentaoksit tuzları kaplama yüzeyine tatbik edilerek incelenmiştir. Elde edilen bulgular doğrultusunda üçüncü çevrimden sonra mevcut kalsiyum oksitlerin reaksiyona girdiği ve zirkonyum-vanadyum esaslı oksitlerin korozyon ürünü olarak meydana geldiği tespit edilmiştir.

#### 2. Deneysel Çalışma

Üretilen TBC sistemlerinde altlık malzeme olarak ticari olarak temin edilmiş 25 mm çapında ve 5 mm kalınlığında silindirik 316L paslanmaz çelik malzeme dörde bölünerek tek parçası kullanılmıştır. Altlık malzeme yaklaşık 40 µm çaplı alümina bilyelerle, 2,5 barlık bir basınçla kumlama işlemine tabi tutularak yüzeydeki mevcut kirlilikler ve oksitlerin uzaklaştırılması amaçlanmış bunun yanında metalik bağ kaplama işlemi öncesi numune altlık yüzey alanı pürüzlendirilerek artırılmıştır. Kumlama işlemi tamamlanmış numuneler üzerine 5-45 µm ebatlarında Amdry 365-1 marka ticari NiCoCrAlY tozları, HVOF tekniği ile püskürtülmüştür. Termal bariyer kaplama olarak ise Metco 201NS marka ZrO<sub>2</sub>-%5CaO (CSZ) tozları ise Metco MCN marka kontrol üniteli atmosferik plasma sprey (APS) tekniği kullanılarak biriktirilmiştir. Kaplama üretim parametreleri Tablo 1' de verilmiştir.

Tablo 1. Bağ ve üst kaplama üretim parametreleri								
Konlomo	Yanma		Toz taşıyıcı	Toz be	Toz besleme		Kaplama	
каріаніа	gaz	ları	gaz	ora	nı	rametreleri ne Kaplam mesafes k 250 mm Ar H <sub>2</sub> Ka sıncı basıncı me 80 15 psi psi	safesi	
NiCoCrAlY	O2,	CH <sub>4</sub>	$N_2$	12,5 l/dk		250 mm		
Wandana	Ciio	Alvum	Toz besleme	Gaz akış	Ar	$H_2$	Kaplama	
каріаніа	Guç	AKIIII	oranı	oranı	basıncı	basıncı	mesafesi	
C87	40	650	20	4,25	80	15	120	
COL	kW	А	g/dk	l/dk	psi	psi	mm	



Üretilen numuneler sıcak korozyon testlerine tabi tutulmuştur. Sıcak korozyon tuzu olarak %99 üzerinde saflıkta ağırlıkça %50 Na<sub>2</sub>SO<sub>4</sub> ve %50 V<sub>2</sub>O<sub>5</sub>, yaklaşık olarak 2 saat mekanik olarak karıştırılmıştır. Hazırlanan karışım numunenin kenarlarından yaklaşık 2-3 mm uzaklıkta olacak şekilde üst yüzeyine 10 mg/cm<sup>2</sup>lik birimle serilmiştir. Sıcak korozyon testleri Protherm PLF130/20 marka elektrik fırında 900 °C' de 5 saatlik çevrimlerle toplamda 5 çevrim yani 25 saat olarak gerçekleştirilmiştir. Sürenin 5 çevrim olması altlıkta meydana gelecek ileri oksidasyon veya üst kaplamada %10 un üzerinde görsel bir çatlak oluşması baz alınarak belirlenmiştir. Testleri sonrası üst kaplamada ufak çatlamalar oluşsa da testin bu sürede sonlandırılma sebebi altlık malzemenin fazla oksitlenmesinden kaynaklıdır.

The International Conference

aterials and Engineering Technology

Sıcak korozyon testleri sonrası numunelerin üst yüzey görüntüleri Nikon Shuttle Pix P-400R marka stereo mikroskop cihazı ile alınmıştır. Sıcak korozyon testi öncesi ve her çevrim sonrası numunelerin faz analizleri için Rigaku Dmax 2200 PC marka cihaz yardımı ile XRD (X-ışını kırınımı) paternleri alınmıştır. Ayrıca her çevrim sonrası numune üst yüzeylerinden ve son çevrim sonrası da hem üst hem de enine kesit yüzeyinden Tescan, MAIA3 XMU marka taramalı elektron mikroskobu (SEM) ile görüntüleri alınmıştır. Ayrıca yine her çevrim sonrası üst yüzeylerden enerji dağılımlı spektrum (EDS) ve enine kesitlerden de elementel haritalama görüntüleri SEM cihazında alınarak malzemeler korozyon testi öncesi ve sonrası karakterizasyon işlemleri gerçekleştirilmiştir.

#### 3. Bulgular ve Tartışma

Şekil 1' de sıcak korozyon testi öncesi ve her çevrim sonrası üst yüzey makro görüntüleri bulunmaktadır. Şekilde görüldüğü üzere numunelerde korozyon tuzları etkisiyle sararma meydana gelmiştir. Ayrıca numunelerin alt yüzeylerinde kırılma ve ayrılmaların meydana geldiği görülmektedir. Korozyon tuzları iç kesimlere serilerek testler yapılmış olsa da bu ayrılmalar kenar etkisinden kaynaklanmaktadır [10,11]. İlerleyen süreçte malzemede renk değişimlerinin yanı sıra yan yüzeylerinde altlık malzemenin fazla oksitlenmesinden ötürü şekilsiz büyümelerin meydana geldiği görülmektedir. Buralarda reaksiyonlar neticesinde açığa sıkan SO<sub>2</sub> ve SO<sub>3</sub> gibi gazların da etkisinin olabileceği düşünülmektedir.



Şekil 1. Numunelerin a) sıcak korozyon testi öncesi, b)5 c)10, d)15, e)20 ve f)25 saat sıcak korozyon çevrimleri sonrası 20x büyütmede üst yüzey makroskobik stereo mikroskop görüntüleri



Şekil 2' de üretilen TBC sistemine ait enine kesit SEM ve elementel haritalama görüntüsü bulunmaktadır. Bağ kaplamanın yaklaşık 80-100 µm kalınlığa sahip olduğu görülürken, üst kaplamanın ise 250 µm dolaylarında kalınlığa sahiptir. Bağ kaplamada üretim prosesi kaynaklı dahili oksitlerin meydana geldiği görülürken, üst kaplamada da yine proses kaynaklı süreksiz açıklıklar ve porozitelerin meydana geldiği görülmektedir. Elementel haritalama görüntüsünde bağ kaplama ve üst kaplama elementlerinin homojen olarak yapı içerisinde dağıldığı anlaşılırken, oksijen izleri takip edildiğinde bağ kaplamada dahili oksit oluşumlarının baskın olarak alüminadan oluştuğu anlaşılımaktadır.

he International Confer

sterials and Engineering Technology



Şekil 2. Üretilmiş TBC sistemine ait a) enine kesit SEM, b) büyütülmüş bağ/üst kaplama ara yüzey SEM ve c) ara yüzeyden alınan elementel haritalama görüntüsü

Şekil 3' te üretilen TBC sisteminin üst yüzeyinden alınan SEM görüntüsü ve EDS analiz sonuçları verilmiştir. Alınan birinci spektrum bölgesinde ergimenin tam meydana geldiği ve Zr, Ca ve O elementlerinden meydana geldiği görülürken, ikinci spektrum bölgesinde aynı elementlere rastlanmış ve bu bölgeler tam ergimenin olmadığı yerler olarak tanımlanabilir.



Şekil 3. TBC sisteminin üst yüzey bölgesinden alınan SEM görüntüsü ve EDS analizleri

Sıcak korozyon testlerinin ardından alınan XRD analizleri her çevrim için ayrı ayrı Şekil 4' te üst üste verilmiştir. İşlem görmemiş numune tamamıyla kübik (c)-ZrO<sub>2</sub> fazından meydana geldiği anlaşılmaktadır. Bu faz kübik florit yapılıdır ve normalde oda koşullarında monoklinik yapı da olan zirkonyanın bu formda olmasını az miktarda CaO ilavesi sağlamıştır[12]. Sıcak korozyon çevrimleri sonrası ise c-ZrO<sub>2</sub> fazlarının m-ZrO<sub>2</sub> fazına dönüştüğü veya sodyum vanadat (NaVO<sub>3</sub>) tuzu ile girdiği reaksiyon neticesinde yine monoklinik yapılı V<sub>0,02/0,01</sub>Zr<sub>0,98/0,99</sub>O<sub>7</sub> fazlarının oluştuğu anlaşılmaktadır. Burada oluşan fazların ayrımını yapmak oldukça zordur, o nedenle XRD analiz sonucunda 3 farklı fazında oluşmuş olabileceği pik sembollerinde gösterilmiştir. Artan çevrim sayısı ile Ca içerikli ürünlerin azaldığı Zr ve V içerikli piklerin arttığı görülmektedir. Bu durum zamanla yüzeyde bulunan mevcut CaO içeriğinin tükendiğini göstermektedir. Elde edilen XRD analiz sonuçlarından, CaO' nun NaVO<sub>3</sub> ile reaksiyonu sonrası oluşabilecek fazın CaV<sub>3</sub>O<sub>7</sub> olduğu anlaşılmaktadır. ZrO<sub>2</sub> ve V<sub>2</sub>O<sub>5</sub> in reaksiyonu neticesinde ise ZrV<sub>2</sub>O<sub>7</sub> fazlarının oluşmuş olabileceği tespit edilmiştir.



Şekil 4.Sıcak korozyon testi öncesi ve sonrası alınan XRD paternleri

Şekil 5' te 2 çevrim sonrası yani 10 saatlik sıcak korozyon çevrimi sonrası üst yüzeyden alınan SEM görüntü ve EDS analiz sonuçları verilmiştir. Şekilden görüldüğü gibi koyu renkli yapı içerisinde dağılım gösteren yassı çubuksu yapıların korozyon ürünü olduğu anlaşılmaktadır. Açık gri renkli bölgelerin V<sub>0,02/0,01</sub>Zr<sub>0,98/0,99</sub>O<sub>7</sub> veya m-ZrO<sub>2</sub> fazlarının olacağını anlaşılırken, siyah renkli yerler ise poroziteleri temsil etmektedir. Daha yüksek büyütmede alınan görüntüde yapının katmanlı olarak büyüme gösterdiği anlaşılmaktadır. Alınan EDS analizine göre ise katmanlı büyüyen bu korozyon ürün yapısının CaO-NaVO<sub>3</sub> etkileşimi ile meydana gelmiş bir faz olduğu anlaşılmaktadır. XRD analiz sonuçlarına göre bu fazın CaV<sub>3</sub>O<sub>7</sub> fazı olabileceği tahmin edilmektedir. Üçüncü spektrumun olduğu bölgede ergimiş bir yapının olduğu görülmektedir. Bu fazında ZrV<sub>2</sub>O<sub>7</sub> fazı olması muhtemeldir.



Şekil 5. 10 saatlik sıcak korozyon çevrim sonrası a) 500x SEM görüntüsü, b) 2kx SEM görüntüsü ve c) 2kx büyütmeli SEM görüntüsünden alınan EDS sonuçları

Üçüncü çevrim sonunda alınan SEM ve EDS analiz sonuçları Şekil 6' da verilmiştir. Çubuksu yassı oluşan tabakalarda ciddi bir azalmanın olduğu görülürken, ayrıca oluşan bu yapıların incelme dağılımlarında seyrelme olduğu anlaşılmaktadır. EDS ve XRD analizlerinin sonuçları doğrultusunda birinci spektrum yine V<sub>0,02/0,01</sub>Zr<sub>0,98/0,99</sub>O<sub>7</sub> veya m-ZrO<sub>2</sub> fazını temsil ettiği, ikinci spektrumun ergimiş formlu CaV<sub>3</sub>O<sub>7</sub> ve üçüncü spektrumun ise CaV<sub>3</sub>O<sub>7</sub> korozyon ürünü olduğu düşünülmektedir.



Şekil 6. 15 saatlik sıcak korozyon çevrim sonrası a) 500x SEM görüntüsü, b) 2kx SEM görüntüsü ve c) 2kx büyütmeli SEM görüntüsünden alınan EDS sonuçları



The International Conference

aterials and Engineering Technology



Şekil 7. 20 saatlik sıcak korozyon çevrim sonrası a) 500x SEM görüntüsü, b) 2kx SEM görüntüsü ve c) 2kx büyütmeli SEM görüntüsünden alınan EDS sonuçları

Son beş saatlik sıcak korozyon çevrimine ait SEM ve EDS analiz resimleri Şekil 8' de verilmiştir. Elde edilen bulgulara göre üst bölgelerde yine Ca içerikli herhangi bir fazın oluşmadığı üretim sonrası biriken partiküller arası boşluklarda ergimiş formlu yapıların sızmış olduğu anlaşılmaktadır. Koyu siyah bölgelerde poroziteleri temsil etmektedir. Elde edilen spektrum sonuçları ve XRD analizleri neticesinde dördüncü çevrime benzer fazların oluştuğu yani az miktarda NaVO<sub>3</sub>' ün yanında  $V_{0,02/0,01}Zr_{0,98/0,99}O_7$  veya m-ZrO<sub>2</sub> fazları ve ZrV<sub>2</sub>O<sub>7</sub> fazlarının kaplama üst yüzeyinde oluştuğu anlaşılmaktadır.



Şekil 8. 25 saatlik sıcak korozyon çevrim sonrası a) 500x SEM görüntüsü, b) 2kx SEM görüntüsü ve c) 2kx büyütmeli SEM görüntüsünden alınan EDS sonuçları

Son sıcak korozyon çevrim sonrası alınan enine kesit SEM görüntüsü ve elementel dağılım haritalaması Şekil 9' da verilmiştir. Şekil' den görüldüğü gibi kaplamada ciddi bir ayrılmanın olmadığı fakat özellikle yüzeye yakın bölgelerde ayrılmaların meydana geldiği anlaşılmaktadır. Ayrıca kaplamanın üretim sonrası poroziteli olduğu tespit edilmişti fakat sızan korozyon tuzlarının buralarda reaksiyon vererek bu boşlukları doldurduğu anlaşılmaktadır. Na ve V izlerinin üst kaplama içerisinde tamamen dağıldığı görülürken, Ca' nın nerdeyse üst kaplama boyunca hiç kalmadığı anlaşılmaktadır. Bağ kaplama yapısında ise ciddi şekilde oksitlenmelerin meydana geldiği görülmektedir. Bağ ve üst kaplama ara yüzeyinde meydana gelen termal olarak büyüyen oksit (TGO) tabakasının ise alümina yerine daha çok Cr<sub>2</sub>O<sub>3</sub> ve karışık oksitlerden meydana geldiği anlaşılmaktadır. Alüminyumun bağ kaplama içerisinde dahili olarak özellikle splatlar arası oksitlendiği anlaşılırken TGO tabakasında az miktarda bulunduğu alınan elementel haritalama görüntülerinden anlaşılmaktadır. TGO tabakası TBC sistemlerinde hasar da baskın rol oynayan sebeplerden biridir [13–16]. Fakat elde edilen sonuçlara göre TGO kaynaklı bir çatlama veya ayrılma tespit edilememiştir.



Şekil 9. 25 saatlik sıcak korozyon çevrim sonrası a) 500x SEM görüntüsü, b) 2kx SEM görüntüsü ve c) 2kx büyütmeli SEM görüntüsünden alınan EDS sonuçları

Elde edilen bulgular XRD, SEM ve EDS sonuçlarına göre kalsiyumun her çevrim sonrası azaldığı ve son iki çevrim de ise yapı içerisinde nerdeyse hiç kalmadığı görülmüştür. Başlangıçtaki çevrimde CaO' nun, NaVO<sub>3</sub> ile reaksiyonu sonucu CaV<sub>3</sub>O<sub>7</sub> korozyon ürününü üretmiştir. Zamanla bu yapının kaplamadan uzaklaşması bu ürünün uçucu olabileceğini veya çevrimler esnasında yeniden reaksiyonlar ile uçucu bir faza dönüşmüş olabileceği, elde edilen XRD ve SEM analizlerinden anlaşılmaktadır. Tükenen CaO ile beraber stabil olan zirkonya kaplamanın stabilitesi bozularak monoklinik yapılı zirkonyaya dönüştüğü, bunun yanında zikonya ile NaVO<sub>3</sub> tuzunun reaksiyonu neticelerinde ise yavaş büyüyen ZrV<sub>2</sub>O<sub>7</sub> fazının [17] ve yine monoklinik formda az V ihtiva eden V<sub>0,02/0,01</sub>Zr<sub>0,98/0,99</sub>O<sub>7</sub> fazlarının meydana geldiği tespit edilmiştir. Literatürde itriya ile stabilize edilmiş zirkonyanın bir çok çalışma da sıcak korozyon davranışı incelenmitir. Çalışmalarda stabilizatör itriyanın NaVO<sub>3</sub> ile reaksiyonu sonucu tetragonal olan zirkonyanın monoklinik zirkonyaya dönüşmesi neticesinde kaplamada meydana gelen hacimsel değişiklerin hasara sebep olduğu belirtilmiştir [18–20]. Mevcut çalışmamızda ise kısmi stabilize edilmiş zirkonya üst kaplamanın stabilizasyonu bozulması sonrası kaplamada ciddi bir hasar etkisine sebebiyet vermemiştir.

### 4. Sonuçlar

NiCoCrAlY bağ kaplama ve CSZ üst kaplama başarılı şekilde sırasıyla 316L paslanmaz çelik malzeme üzerine biriktirilmiştir. Üretilen numuneler çevrimli sıcak korozyon testleri tabi tutulmuş ve elde edilen sonuçlar aşağıdaki sıralanmıştır.

- 1. Sıcak korozyon testleri esnasında altlık numune ciddi şekilde oksidasyona maruz kalmış ve beşinci çevrimde test sonlandırılmıştır. Elde edilen görüntülerde üst kaplama da ciddi bir hasarın meydana gelmediği anlaşılmıştır.
- Sıcak korozyon testlerinin ilk çevrimlerinde korozyon ürünü olarak tabakalı olarak büyüyen CaV<sub>2</sub>O<sub>7</sub> fazları meydana gelmiş, son çevrimlere doğru ise bu fazların ve kalsiyum içerikli herhangi bir fazın izine rastlanmamıştır. Kübik formda olan CSZ yapısının monoklinik bir faza dönüştüğü tespit edilmiştir.



The International Conference of Materials and Engineering Technology

#### 5. Kaynaklar

- K.M. Doleker, Y. Ozgurluk, H. Ahlatci, A.C. Karaoglanli, Evaluation of oxidation and thermal cyclic behavior of YSZ, Gd2Zr2O7 and YSZ/Gd2Zr2O7 TBCs, Surf. Coatings Technol. 371 (2019) 262–275. doi:10.1016/j.surfcoat.2018.11.055.
- [2] N.P. Padture, Thermal Barrier Coatings for Gas-Turbine Engine Applications, Science (80-.). 296 (2002) 280–284. doi:10.1126/science.1068609.
- [3] A.C. Karaoglanli, K.M. Doleker, Y. Ozgurluk, State of the art thermal barrier coating (TBC) materials and tbc failure mechanisms, in: A. Öchsne, H. Altenbach (Eds.), Adv. Struct. Mater., Springer, Singapore, 2017: pp. 441–452. doi:10.1007/978-981-10-1602-8\_34.
- [4] D.J. Wortman, R.E. Fryxell, K.L. Luthra, P.A. Bergman, Mechanism of low temperature hot corrosion: Burner rig studies, Thin Solid Films. 64 (1979) 281–288. doi:10.1016/0040-6090(79)90521-2.
- [5] N. Eliaz, G. Shemesh, R.M. Latanision, Hot corrosion in gas turbine components, Eng. Fail. Anal. 9 (2002) 31–43. doi:10.1016/S1350-6307(00)00035-2.
- [6] Y. Hui, S. Zhao, J. Xu, B. Zou, Y. Wang, X. Cai, L. Zhu, X. Cao, High-temperature corrosion behavior of zirconia ceramic in molten Na2SO4+NaVO3 salt mixture, Ceram. Int. 42 (2016) 341–350. doi:10.1016/j.ceramint.2015.08.116.
- [7] M.H. Habibi, L. Wang, S.M. Guo, Evolution of hot corrosion resistance of YSZ, Gd2Zr2O7, and Gd2Zr2O7+YSZ composite thermal barrier coatings in Na2SO4+V2O5 at 1050°C, J. Eur. Ceram. Soc. 32 (2012) 1635–1642. doi:10.1016/j.jeurceramsoc.2012.01.006.
- [8] Y. Ozgurluk, K.M. Doleker, A.C. Karaoglanli, Hot corrosion behavior of YSZ, Gd2Zr2O7and YSZ/Gd2Zr2O7thermal barrier coatings exposed to molten sulfate and vanadate salt, Appl. Surf. Sci. 438 (2018) 96–113. doi:10.1016/j.apsusc.2017.09.047.
- [9] Y. Ozgurluk, K.M. Doleker, A.C. Karaoglanli, Investigation of the effect of V 2 O 5 and Na 2 SO 4 melted salts on thermal barrier coatings under cyclic conditions, Anti-Corrosion Methods Mater. 66 (2019) 644–650. doi:10.1108/acmm-12-2018-2042.
- [10] A. Keyvani, M. Saremi, M. Heydarzadeh Sohi, Microstructural stability of zirconia-alumina composite coatings during hot corrosion test at 1050 °c, J. Alloys Compd. 506 (2010) 103–108. doi:10.1016/j.jallcom.2010.06.110.
- [11] P. Wang, S. Deng, Y. He, C. Liu, J. Zhang, Oxidation and hot corrosion behavior of Al 2 O 3 /YSZ coatings prepared by cathode plasma electrolytic deposition, Corros. Sci. 109 (2016) 13– 21. doi:10.1016/j.corsci.2016.03.017.
- [12] W.L. Worrel, Solid Electrolytes, in: S. Geller (Ed.), Solid Electrolytes, Springer-Verlag, New York, 2006: pp. 143–166.
- [13] H.E. Evans, Oxidation failure of TBC systems: An assessment of mechanisms, Surf. Coatings Technol. 206 (2011) 1512–1521. doi:10.1016/j.surfcoat.2011.05.053.
- [14] J. Toscano, D. Naumenko, A. Gil, L. Singheiser, W.J. Quadakkers, Parameters affecting TGO growth rate and the lifetime of TBC systems with MCrAIY-bondcoats, Mater. Corros. 59

(2008) 501-507. doi:10.1002/maco.200804134.

[15] K.M. Doleker, Y. Ozgurluk, A.C. Karaoglanli, Isothermal oxidation and thermal cyclic behaviors of YSZ and double-layered YSZ/La2Zr2O7 thermal barrier coatings (TBCs), Surf. Coatings Technol. 351 (2018) 78–88. doi:10.1016/J.SURFCOAT.2018.07.069.

The International Conference

of Materials and Engineering Technology

- [16] K.M. Doleker, H. Ahlatci, A.C. Karaoglanli, Investigation of Isothermal Oxidation Behavior of Thermal Barrier Coatings (TBCs) Consisting of YSZ and Multilayered YSZ/Gd2Zr2O7 Ceramic Layers, Oxid. Met. 88 (2017) 109–119. doi:10.1007/s11085-016-9690-4.
- [17] Z.G. Liu, J.H. Ouyang, Y. Zhou, X.L. Xia, Hot corrosion behavior of V2O5-coated Gd2Zr2O7 ceramic in air at 700-850 °C, J. Eur. Ceram. Soc. 29 (2009) 2423–2427. doi:10.1016/j.jeurceramsoc.2009.01.001.
- [18] S.Y. Park, J.H. Kim, M.C. Kim, H.S. Song, C.G. Park, Microscopic observation of degradation behavior in yttria and ceria stabilized zirconia thermal barrier coatings under hot corrosion, Surf. Coatings Technol. 190 (2005) 357–365. doi:10.1016/j.surfcoat.2004.04.065.
- [19] A. Afrasiabi, M. Saremi, A. Kobayashi, A comparative study on hot corrosion resistance of three types of thermal barrier coatings: YSZ, YSZ + Al2O3 and YSZ/Al2O3, Mater. Sci. Eng. A. 478 (2008) 264–269. doi:10.1016/j.msea.2007.06.001.
- [20] R. Ahmadi-Pidani, R. Shoja-Razavi, R. Mozafarinia, H. Jamali, Evaluation of hot corrosion behavior of plasma sprayed ceria and yttria stabilized zirconia thermal barrier coatings in the presence of Na 2SO 4V 2O 5 molten salt, Ceram. Int. 38 (2012) 6613–6620. doi:10.1016/j.ceramint.2012.05.047.

# ÇEVRİMLİ VE İZOTERMAL SICAK KOROZYONUN NİKEL ESASLI SÜPER ALAŞIM MALZEMEYE ETKİSİ

The International Conference

aterials and Engineering Technology

# KADİR MERT DÖLEKER \*1, ABDULLAH CAHİT KARAOĞLANLI<sup>2</sup>

<sup>1</sup> Bartın Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, Bartın, TÜRKİYE.
 <sup>2</sup> Bartın Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, Bartın, TÜRKİYE.

#### Özet

Nikel esaslı süper alaşımlar gaz türbinleri, nükleer reaktörler, jeneratörler gibi geniş bir kullanım alanına sahiptir. Mekanik dayanım ve korozyon direncinin önemli olduğu alanlarda özellikle tercih edilirler. Sıcak korozyon, yüksek sıcaklık uygulamalarında oldukça yıpratıcı bir hasar türüdür. Sıcak korozyonda, erimiş tuzlar reaktif bileşenlerle kolayca reaksiyona girer ve iş parçalarında erken hasara neden olur. Bu çalışmada, nikel bazlı süper alaşım olarak Inconel 718, sıcak korozyon testlerine tabi tutulmuştur. % 50 V<sub>2</sub>O<sub>5</sub>-50% Na<sub>2</sub>SO<sub>4</sub> karışımları korozyon bileşeni olarak kullanılmıştır. 900 ° C' de, 18 saat süreyle çevrimli ve izotermal sıcak korozyon testleri uygulanmıştır. Inconel 718' de oluşan korozyon ürünleri ve mikroyapısal değişiklikler, sıcak korozyon testlerinden sonra taramalı elektron mikroskobu (SEM), SEM-Enerji Dağılımlı X-Işını (EDX), SEM-haritalaması ve X-Işını Kırınımı (XRD) analizleri ile karakterize edilmiştir.

Anahtar Kelimeler: Sıcak korozyon, Inconel 718, Na<sub>2</sub>SO<sub>4</sub>, V<sub>2</sub>O<sub>5</sub>, Ergimiş tuz

#### 1. Giriş

Süper alaşım malzemeler yüksek sıcaklıklarda mekanik, korozyon, oksidasyon ve sürünme gibi zorlamalara karşı üstün direnç gösteren alaşım gruplarıdır. Yaygın olarak demir, nikel ve kobalt esaslı üç tip süper alaşım grubu kullanılmaktadır. Belirtilen element miktarının fazlalığı o alaşım grubuna adını vermektedir. Demir esaslı alaşımlar eski dönemlerde tercih edilen nispeten nikel ve kobalt esaslılara göre daha zayıf özelliklere sahiptirler. Kobalt esaslılar ise demir esaslılara nazaran daha dikkat çekici özelliklere sahiptirler. İçerik olarak önemli miktarda kobaltın yanı sıra Cr ve W içerirken, az miktarda Mo, Nb, Ta ve Ti gibi elementlerde içermektedirler. Demir esaslılara kıyasla kısmen yüksek sıcaklık dirençleri daha üstündür. Nikel esaslı süper alaşımlar ise en çok tercih edilen gruptur [1,2]. Özellikle gaz türbin ve motor bileşenlerinde tercih edilen bu malzeme grubu hem çökelme sertleşmesi hem de katı eriyik sertleşmesi ile mukavemet özellikleri geliştirilmiş süper alaşımlardır. Nikel esaslı süper alaşımlardır. Nikel esaslı süper alaşımları gibi gibi fazlar içermektedirler [3].

Inconel 718 en çok tercih edilen nikel esaslı süper alaşım malzemelerden biridir. Ağırlıkça Ni, Fe, Cr içerirken bir miktar Nb, Mo, Ti ve az miktarda birkaç element daha içermektedir. Çökelme sertleşmesi ile Ni<sub>3</sub>Nb fazı çökeltilerek yapının mukavemet kazanması sağlanabilmektedir. Yüksek oksidasyon,



korozyon ve yorulma dayanımına sahip bir malzemedir. Yaygın olarak pervanelerde, türbin kanatçıklarında, roket motorlarında, egzoz valf, yakıt tankları, nükleer reaktörlerde ve sıcak iş kalıplarında tercih edilmektedir [4]. Çalışma koşullarına bağlı olarak bu malzeme grupları oksidasyon ve sıcak korozyona maruz kalmaktadırlar. Özellikle sıcak korozyon hasarları, oksidasyona nazaran daha tahribat verici hasarlar ve malzeme kayıplarına sebep olmaktadır. Örneğin uçak motorlarının yakıt tankları sülfür içerikli yakıtlardan kaynaklı sülfidasyona maruz kalmaktadır [5].

The International Conference of Materials and Engineering Technology

Yakıtlarda bulunan empüriteler ergimiş tuz formunda malzeme yüzeyinde oluşan oksitlerden kolaylıkla penetre olarak malzemenin korozyonuna sebep olmaktadır. Sıcaklığa bağlı olarak tip 1 ve tip 2 olarak ikiye ayrılan sıcak korozyonda, tip 1 için 850 °C ve üzeri sıcaklıklarda meydana gelirken, tip 2 sıcak korozyon ise 650-750 °C' ler de meydana gelmektedir. Yaygın olarak nispeten düşük ergime sıcaklıklı olan sodyum sülfat ve vanadat karışımlarının malzemeye yüzeyinden penetre olmasıyla sıcak korozyon meydana gelmektedir. Tip 1 sıcak korozyonu daha yüksek sıcaklıklarda meydana gelmesinden ötürü daha hasar verici olmaktadır. Bu tip korozyonda başlangıçta yavaş bir korozyon oranı görülürken, daha sonra hızlanarak devam etmektedir. Bu durum başlangıçta meydana gelen koruyucu oksit oluşumunun direnci kaynaklıdır, fakat devam eden süreçte koruyucu oksit tabakası çözünerek hasar içeriye doğru ilerlemektedir [6]. Sodyum sülfat ve vanadatın reaksiyonu ile meydana gelen sodyum vanadat düşük ergime sıcaklıklı asidik eritkenlik gösteren bir tuz türü olduğundan oluşan oksitlerle reaksiyona girerek korozyon ürünleri meydana getirmektedir [7]. Bu çalışmada Inconel 718, izotermal ve çevrimli olarak sodyum sülfat ve vanadat içeren sıcak korozyon testlerine tabi tutulmuştur. Testler sonunda malzemede meydana gelen korozyon etkisi, derinliği ve oluşan korozyon ürünleri incelenmiştir.

#### 2. Deneysel Çalışma

Ticari olarak temin edilen Inconel 718 malzeme, 5mm kalınlıklı, bir inç çaplı numune dörde enine kesit görüntülerini rahat incelemek amacıyla dörde bölünmüştür. Yaklaşık %99 saflık değerlerine sahip V<sub>2</sub>O<sub>5</sub> ve Na<sub>2</sub>SO<sub>4</sub> bileşikleri ağırlıkça yarı yarıya oranda mekanik olarak 1 saat karıştırıldıktan sonra malzemenin bir yüzeyinin tamamına yaklaşık 10 mg/cm<sup>2</sup> lik ağırlıkta serilmiştir. Sıcak korozyon testleri elektrik firmında (Protherm PLF130/20) 900 °C' de 6 saatlik çevrimlerle toplamda 3 çevrim yani 18 saat olarak ve sadece 18 saat izotermal olarak gerçekleştirilmiştir.

Sıcak korozyon testleri sonrası numunelerin üst yüzey görüntüleri stereo mikroskop (Nikon Shuttle Pix P-400R) ile incelenmiştir. Sıcak korozyon testinden önce ve her korozyon çevrimi sonrası numunelerin faz analizleri için XRD (X-ışını kırınımı) paternleri (Rigaku Dmax 2200 PC) alınmıştır. Ayrıca her çevrim sonrası numune üst yüzeylerinden ve son çevrim sonrası da hem üst hem de enine kesit yüzeyinden taramalı elektron mikroskobu (SEM) (Tescan, MAIA3 XMU) ile görüntüleri alınmıştır. Her çevrim sonrası üst yüzeylerden enerji dağılımlı spektrum (EDS) ve enine kesitlerden de elementel haritalama görüntüleri alınarak korozyon etkisinin karakterizyon incelemeleri yapılmıştır.



## 3. Bulgular ve Tartışma

Inconel 718 süper alaşım malzemenin her bir sıcak korozyon testi sonrası stereo mikroskopla 20x büyütmede alınan makro görüntüleri şekil 1' de verilmiştir. Alınan görüntülerde numunelerde ciddi bir pullanma vb. hasarın olmadığı anlaşılmaktadır. Çevrimli oksidasyon testlerinde çevrim sayısı arttıkça numune yan yüzeylerinde meydana gelen oksit büyümeleri dikkat çekmektedir. Bunun yanında numune yüzeyinde sarı renkli korozyon tuzlarına ait izlerin olduğu görülmektedir.



Şekil 1. Inconel 718 malzemeye ait a) sıcak korozyon testi öncesi, b) izotermal 18 saat sonrası, c) 6 saat sonrası (1. çevrim), d) 12 saat sonrası (2. çevrim) ve e) 18 saat sonrası (3. çevrim) makro görüntüleri

Şekil 2' de kullanılan süper alaşım Inconel 718 malzemeye ait dağlamasız SEM görüntüsü ve EDS ile alan elementel tarama analiz sonucu verilmiştir. Analiz sonucuna göre malzemenin ağırlıklı olarak Ni, Fe ve Cr' dan meydana geldiği bununda yine bir miktar Nb olduğu görülürken diğer elementlerin oranlarının çok az olduğu görünmektedir.



Şekil 2. Inconel 718 malzemeye ait SEM görüntüsü ve EDS analizi

Sıcak korozyon testleri öncesi ve sonrası alınan XRD analiz verileri Şekil 3' de verilmiştir. XRD analizine göre Inconel' in γ fazından meydana geldiği yani Ni, Fe ve Cr içeriği zengin bir katı çözelti fazına sahip olduğu anlaşılmaktadır. Altı saatlik ilk sıcak korozyon çevrim sonrası ise matriks fazın piklerinde azalmalar görülürken Ni<sub>3</sub>V<sub>2</sub>O<sub>8</sub>, (Fe, Cr)VO<sub>4</sub>, NiV<sub>2</sub>Nb<sub>2</sub>O<sub>10</sub>, gibi korozyon ürünlerinin yanı sıra Cr<sub>2</sub>O<sub>3</sub> ve spinel (Fe, Ni)Cr<sub>2</sub>O<sub>4</sub> fazlarının meydana geldiği tespit edilmiştir. İlerleyen süreçte bu fazların yanı sıra Ni<sub>3</sub>Nb intermetalik fazınında meydana geldiği ve 18 saatlik izotermal sıcak korozyon testi sonunda benzer fazların meydana geldiği tespit edilmiştir.



Şekil 3. Sıcak korozyon testleri öncesi ve sonrası XRD paternleri



aterials and Engineering Technology



Şekil 4. 6 saatlik tek çevrimli sıcak korozyon sonrası SEM görüntüsü ve EDS analizleri

İkinci sıcak korozyon çevrimi sonunda yüzeyden alınan SEM ve EDS analizleri şekil 5' te verilmiştir. Yapının baskın olarak kısa çubuksu forma sahip Ni<sub>3</sub>V<sub>2</sub>O8 veya NiV<sub>2</sub>Nb<sub>2</sub>O<sub>10</sub> fazları olabileceği düşünülmektedir. İkinci spektrumda ise ergimiş formlu bir yapının olduğu düşünülmektedir. NaSO<sub>4</sub> ve V<sub>2</sub>O<sub>5</sub> in reaskiyonu sonrası oluşan NaVO<sub>3</sub> ve Na<sub>2</sub>O nun NiO ile reaksiyonu neticesi de oluşabilecek Ni<sub>3</sub>V<sub>2</sub>O<sub>8</sub> fazı olduğu düşünülmektedir. Üçüncü spektrum ise Nb miktarının yüksek olması Ni<sub>3</sub>Nb



intermetaliğinin yanı sıra NiV<sub>2</sub>Nb<sub>2</sub>O<sub>10</sub>, (Fe, Cr)VO<sub>4</sub> gibi korozyon ürünlerinin de meydana gelmiş olabileceğini göstermektedir.



Şekil 5. 12 saatlik 2 çevrimli sıcak korozyon sonrası SEM görüntüsü ve EDS analizleri

18 saatlik son sıcak korozyon çevrimi sonrası alınan SEM ve EDS analizleri Şekil 6' da verilmiştir. İkinci çevrimden farklı olarak ergimiş formlu yapının (üçüncü spektrum) malzeme yüzeyinde hâkim olduğu bir önceki çevrimde elde edilen çubuksu yapıların (ikinci spektrum) üstlerini örttüğü görülmektedir. Ayrıca nikelce zengin adacıklardan (birinci spektrum) meydana bir fazın oluştuğu görülmektedir. Bu yapının reaksiyon vermemiş nikelce zengin  $\gamma$  fazını temsil ettiği düşünülmektedir. Koyu siyah bölgelerde (dördüncü spektrum) ise çukurcuk oluştuğu yani korozyon etkisiyle sızan korozyon tuzlarından kaynaklı olduğu düşünülmektedir. EDS analizlerine göre vanadyum içeriğinin yüksek olması bu bölgede NaVO<sub>3</sub> ve Ni<sub>3</sub>V<sub>2</sub>O<sub>8</sub> gibi fazların olduğu düşünülmektedir.



Şekil 6. 18 saatlik 3 çevrimli sıcak korozyon sonrası SEM görüntüsü ve EDS analizleri

Son çevrime ait numunenin enine kesit SEM görüntüsü ve elementel haritalama görüntüleri şekil 7' de verilmiştir. Korozyon etkisinin bütünlüğünü koruduğu yapıda çatlak veya ayrılma meydana getirmediği anlaşılmaktadır. Yaklaşık 250 µm derinliğe kadar korozyon etkisinin tesir ettiği anlaşılmaktadır. Alınan elementel dağılım haritalamaları Ni, Fe, Cr ca zengin korozyon ürünlerinin olduğunu teyit etmektedir. Na ve V izlerinin çakışması NaVO<sub>3</sub> formunun ergiyerek yapı içerisine sızdığı ve Ni, Fe, Cr ile reaksiyon verdiğini göstermektedir. Yapılan XRD ve EDS incelemelerinde de muhtemel oluşmuş olabilecek fazlardan yukarda bahsedilmiştir. Mikroyapıya dikkat edildiğinde korozyon tabakası orta kısımlarında adacıklar görünmektedir. Elementel haritalamaya göre bu adacıkların büyük kısmı ağırlıkça Nb içeriklidir ve herhangi bir korozif etkiye maruz kalmadığı anlaşılmaktadır. Ayrıca Nb içeriklilerden daha küçük parçacıklı ağırlıkça Ni oranı daha yüksek adacıklarda bulunmaktadır. Yine bu yapılarda da herhangi bir reaksiyon olmadığı takip edilen Na ve V izlerinden anlaşılmaktadır. Na ve V izleri en alt tabakalara yani ana metale doğru azaldığı dikkat çekmektedir. Burada Fe ve Cr ca zengin bir dağılımın olduğu yani FeCr<sub>2</sub>O<sub>4</sub> spinel oksit fazının oluşmuş olabileceği düşünülmektedir. Korozyon tabakasının bittiği kısımda ise Ni izlerinin baskın olarak sınırladığı ve reaksiyon vermediği düşünülmektedir.



Şekil 7. 18 saatlik 3 çevrimli sıcak korozyon sonrası enine kesit SEM görüntüsü ve elementel haritalama analizi

Şekil 8' de izotermal olarak 18 saat sıcak korozyon testine tabi tutulmuş Inconel 718 malzemenin üst yüzeyinden alınmış SEM görüntüsü ve EDS analiz sonuçları verilmiştir. Birinci spektrumun sahip olduğu fazın daha baskın olarak yapıda dağıldığı görülürken, ikinci ve üçüncü spektrumlar ise daha çok reaksiyon vermemiş, iri adacıklar şeklinde yapıda dağılım gösteren ürünler olduğu görülmektedir. Birinci spektrum Inconel 718' e ait elementel dağılıma sahiptir fakat önemli miktarda V içermesi, vanadyum içerikli korozyon ürünleri ve oksitler içerdiğini göstermektedir. Adacık şeklinde bulunan 2 ve 3. spektrumların ise Ni ve Nb ca oldukça zengin olması bu yapıların intermetalik Ni<sub>3</sub>Nb bileşikleri olduğunu göstermektedir. Bu durum Ni<sub>3</sub>Nb' un korozyona uğramadığını da göstermektedir. Benzer durum çevrimli 18 saatlik sıcak korozyon testi sonrası alınan enine kesit görüntülerinde de tespit edilmiştir.



Şekil 8. 18 saatlik izotermal sıcak korozyon sonrası SEM görüntüsü ve EDS analizleri

İzotermal 18 saatlik sıcak korozyon testi sonunda alınan enine kesit SEM görüntüsü Şekil 9' da verilmiştir. Mikroyapı incelendiğinde orta kesimlerde plakalı parçacıklar gömülü vaziyette durmaktadırlar. Bu parçacıklar Ni<sub>3</sub>Nb fazlarını temsil ettiği düşünülmektedir. Yapının diğer dağılımları çevrimli sıcak korozyona benzer şekildedir yani Na ve V içerikli tuzun sızarak yapıda korozyon ürünleri meydana getirdiği düşünülmektedir. Ana malzemeye doğru açık gri renkli bölgelerinde spinel yapılı FeCr<sub>2</sub>O<sub>4</sub> fazı olduğu düşünülmektedir. Korozyonun tesir ettiği derinlik ise yaklaşık 140 μm olarak ölçülmüştür.



Şekil 9. 18 saatlik izotermal sıcak korozyon sonrası enine kesit SEM görüntüsü ve EDS analizi

Yapılan korozyon testleri sonrası çevrimli sıcak korozyonun iki kata yakın daha fazla ilerlemiş olduğu anlaşılmaktadır. Bu durum çevrimler sonrası tekrar korozyon tuzlarının ilavesi ve çevrimler neticesinde meydana gelmiş olabilecek termal gerilmelerin etkisi ile oluşabilecek kılcal açıklıklardan korozif tuzların sızmasından kaynaklı olduğu düşünülmektedir. 900 °C Inconel 718' in oksidasyona uğraması için oldukça yüksek bir sıcaklıktır. Bu nedenle başlangıçta malzemenin oksitlendiği sonrasında ise korozyon tuzları ile aşağıdaki gibi reaksiyonlar vermiş olabileceği düşünülmektedir.

 $\begin{aligned} &2Na_2SO_4 + V_2O_5 = 2NaVO_3 + Na_2O + SO_3(g) \\ &Fe_2O_3 + 2NaVO_3 = 2FeVO_4 + Na_2O \\ &Cr_2O_3 + 2NaVO_3 = 2CrVO_4 + Na_2O \\ &3NiO + 2NaVO_3 = Ni_3V_2O_8 + Na_2O \\ &Ni + Nb_2O_5 + 2NaVO_3 = NiV_2Nb_2O_{10} + Na_2O \end{aligned}$ 

Oluşan FeVO<sub>4</sub> ve CrVO<sub>4</sub> fazlarının da test sıcaklığından daha düşük sıcaklıkta ergidiği belirtilirken, Na<sub>2</sub>O' nun ise metal oksitlerin çözünürlüğünü düşürdüğü belirtilmiştir [8]. Ni<sub>3</sub>V<sub>2</sub>O<sub>8</sub>' in ise daha yüksek ergime sıcaklığına sahip olduğu ve bu nedenle nikelin diğer elementlere nazaran korozyonu daha yavaşlatıcı etki yaptığı belirtilmişir [9]. Bu çalışmada Ni<sub>3</sub>Nb ve nikelce zengin oluşumların korozyon testleri sonrası tespit edilmesi bu bulguyu desteklemektedir. Ayrıca Na<sub>2</sub>O' nun Cr<sub>2</sub>O<sub>3</sub> ile reaksiyon vererek Na<sub>2</sub>CrO<sub>4</sub>, Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, Na<sub>2</sub>CrO<sub>7</sub> gibi fazlarda meydana getirdiği literatürde belirtilmiştir [10]. Benzer bulgu çalışmamızda tespit edilememiştir. Fakat alınan EDS analizlerinde Na elementi ile Cr elementinin beraber pik verdiği analizlerde eser miktarda olma ihtimali söz konusudur. Inconel 718' e daha düşük sıcaklıkta yapılan vanadat içerikli sıcak korozyon testlerinde korozyon ürünlerinden ziyade oksitlere daha çok rastlanmıştır. Artan sıcaklık korozyon tuzunun daha akışkan ve reaktif olmasını sağlamasından dolayı bu çalışmada oksitlerden ziyade korozyon ürünleri daha çok tespit edilmiştir. Kamal ve arkadaşları [11] yapmış oldukları çalışmada Inconel 718, Nimonic 75 ve Incoloy 800H süper alaşımlara, Na<sub>2</sub>SO<sub>4</sub>–%60V<sub>2</sub>O<sub>5</sub> içerikli korozyon testlerini 900 °C


# 4. Sonuçlar

Nikel esaslı bir süper alaşım malzeme olan Inconel 718' e sodyum sülfat ve vanadat karışımlarıyla 900 °C' de çevrimli ve izotermal sıcak korozyon testleri başarılı şekilde uygulanmıştır. Sıcak korozyon testleri sonrası yapılan karakterizasyon testleri ve incelemelere bağlı olarak elde edilen sonuçlar aşağıda sıralanmıştır.

- 1. Çevrimli sıcak korozyon testi, izotermal sıcak korozyon testine nazaran malzemenin daha fazla korozyona uğramasına sebep olmuştur.
- 2. Korozyon testleri sonrası oluşan her iki deney sonunda benzer korozyon ürünleri tespit edilmiştir.
- 3. Meydana gelen korozyon ürünlerinin koruyucu oksitlerle reaksiyonu neticesinde meydana geldiği görülürken, XRD ve EDS analizlerine göre Ni<sub>3</sub>V<sub>2</sub>O<sub>8</sub> fazının nispeten daha baskın olduğu tespit edilmiştir.
- 4. Korozyon sonrası bazı bölgelerde nikelce zengin ve Ni<sub>3</sub>Nb gibi fazların korozyondan etkilenmeden kaldığı görülmüştür. Korozyon etkisinin azaldığı yani ana metalin yakın bölgelerde FeCr<sub>2</sub>O<sub>4</sub> fazlarının meydana gelerek korozyonun ilerlemesini yavaşlattığı görülmüştür.

# 5. Kaynaklar

- [1] E. Akca, A. Gursel, A Review on Superalloys and IN718 Nickel-Based INCONEL Superalloy, Period. Eng. Nat. Sci. 3 (2015) 15–27.
- [2] W. (Walter) Betteridge, J. Heslop, The nimonic alloys, and other nickel-base high-temperature alloys., Edward Arnold, London, 1974.
- [3] A. Thomas, M. El-Wahabi, J.M. Cabrera, J.M. Prado, High temperature deformation of Inconel 718, J. Mater. Process. Technol. 177 (2006) 469–472. doi:10.1016/j.jmatprotec.2006.04.072.
- [4] G.A. El-Awadi, S. Abdel-Samad, E.S. Elshazly, Hot corrosion behavior of Ni based Inconel 617 and Inconel 738 superalloys, Appl. Surf. Sci. 378 (2016) 224–230. doi:10.1016/j.apsusc.2016.03.181.
- [5] M. Naghiyan Fesharaki, R. Shoja-Razavi, H.A. Mansouri, H. Jamali, Evaluation of the hot corrosion behavior of Inconel 625 coatings on the Inconel 738 substrate by laser and TIG cladding techniques, Opt. Laser Technol. 111 (2019) 744–753. doi:10.1016/j.optlastec.2018.09.011.



- [6] G.Y. Lai, Hot Corrosion in Gas Turbines, in: G.Y. Lai (Ed.), High-Temperature Corros. Mater. Appl., ASM International, 2007: pp. 249–528.
- [7] T.S. Sidhu, S. Prakash, R.D. Agrawal, Hot corrosion and performance of nickel-based coatings, Curr. Sci. 90 (2006) 41.
- [8] O. Sotelo-Mazón, C. Cuevas-Arteaga, J. Porcayo-Calderón, R.M. Melgoza-Alemán, M.G. Valladares Cisneros, G. Izquierdo-Montalvo, L. Martínez Gómez, Electrochemical study of the corrosion performance of AISI-309 and AISI-310 exposed in NaVO3 at high temperature, Int. J. Electrochem. Sci. 10 (2015) 9112–9130.
- [9] O. Sotelo-Mazón, J. Porcayo-Calderon, C. Cuevas-Arteaga, J.J. Ramos-Hernandez, J.A. Ascencio-Gutierrez, L. Martinez-Gomez, EIS evaluation of Fe, Cr, and Ni in NaVOat 700°c, J. Spectrosc. 2014 (2014) 10. doi:10.1155/2014/949168.
- [10] G.S. Mahobia, N. Paulose, V. Singh, Hot corrosion behavior of superalloy IN718 at 550 and 650 C, J. Mater. Eng. Perform. 22 (2013) 2418–2435. doi:10.1007/s11665-013-0532-0.
- [11] S. Kamal, R. Jayaganthan, S. Prakash, High temperature cyclic oxidation and hot corrosion behaviours of superalloys at 900°C, Bull. Mater. Sci. 33 (2010) 299–306. doi:10.1007/s12034-010-0046-4.
- [12] F. Pettit, Hot corrosion of metals and alloys, Oxid. Met. 76 (2011) 1–21. doi:10.1007/s11085-011-9254-6.
- [13] S. Prakash, Hot corrosion of alloys and coatings, in: W. Gao, Z. Li (Eds.), Dev. High-Temperature Corros. Prot. Mater., Elsevier Ltd, 2008: pp. 164–191. doi:10.1533/9781845694258.1.164.

# COMPARATIVE ANALYSIS OF CLASSIFICATION TECHNIQUES FOR CRISIS MANAGEMENT SYSTEMS

The International Conference of Materials and Engineering Technology

# SAED ALQARALEH, MERVE IŞIK

Hasan Kalyoncu University, Engineering Faculty, Computer Engineering Department, Gaziantep, TURKEY.

#### Abstract

In recent years, social media analytics has become important research fields, which lead to improving many approaches such as crisis management systems. In addition, impressive attention has been given for mining the publically available huge amount of data to gain situational awareness, which may help in preventing or decrease the effect of some disaster through taking the correct responses. The main purpose of this study is to investigate the performance of some well-known machine learning Classifiers, i.e., K-Nearest Neighbor (KNN), Naïve Bayes (NB), Random Forest (RF), AdaBoost (AdaBoost), GradientBoosting (GBC), for enhancing the task of classifying the information available before or even during any crisis. This comparison is conducted to analyze the performance of the classification framework and provide recommendations related to improving crises management systems.

Several experiments have been conducted, and it has been observed that the performance of all the studied algorithms is somehow similar to each other. In addition, the accuracy is very much affected by the quality of the processed data. Overall, none of the studied algorithms has a stability in the performance, however, by building an ensemble system of the studied algorithms, the performance, robust and stability of the classifying process has been significantly improved.

**Keyword**: Crises Management Systems; K-Nearest Neighbors; Naïve Bayes; Random Forest; AdaBoost Classifier; GradientBoosting Classifier.

#### **1. Introduction**

Social media generally refers to the communication between users through online sharing platforms such as social networking sites. In recent years, social media channels have come to the point of being everywhere in our daily lives. Platforms such as Twitter, Facebook, and İnstagram are playing a crucial role in sharing information quickly. As a result, social media's data repository became a popular in terms of information mining, and have a valuable information for many fields such as marketing, politics, education and many others such as the crisis monitoring applications, where it is possible to use social media data to immediately detect a situation that requires an action from a recovery team (ex. during human-made or natural disasters). However, detecting such an event is not hard, while, manipulating and processing the amount of social media data, for such purpose, requires a huge team. Crisis Management System is a system that aims to provide disaster detection and prevention of disasters, timely, rapid and effective response to incidents that cause disasters and a safer and more developed environment for disaster-affected communities. In recent years, building such systems by taking advantages from the available data and the improvements in machine learning techniques have attracted the attention of many researchers.

In the following, due to space constraints, a few of the recent developments and studies related to the Crisis Management System have been summarized. In [1], the Twitcident, which is a Web-based system that automatically filters and analyzes tweets about events has been introduced. In general, the system consists of three parts: 1) the first part keeps a list of events in the Netherlands in real-time. It does this by parsing messages that are publicly available in real-time and informs the emergency services by obtaining information such as disaster type, location, start time. 2) the second part, responsible for creating a Twitter search query, using the information collected by the first part, about the city name and the most commonly used words of people (for example, "earthquake" when an earthquake occurs). The system then uses the Search API to get past tweets and the Streaming API to get a valid tweet stream. 3) The third component includes features analysis, such as the type of tweets, i.e., retweet, mentioning, answering, singleton and then this component can visualize these features. In [2], a new disasters management system that has offline and real-time services was proposed. The goal of the offline part is to extract geographic data, using some applications such as OpenStreetMap and GooglePlaces API. The real-time location service works with the Twitter crawler that responsible for collecting the related information. Then, a series of operations such as text cleaning and tokenizing are performed, where the Location tokens are determined and matched. Finally, all statistics related to this matched are saved into the system database.

The International Conference of Materials and Engineering Technology

In [3], a system that can detect new emergency situations from Twitter data has been implemented. The system has the following four components: 1) First, in this component, the first story detection (FSD) is performed using the Locality Sensitive Hashing algorithm [4], to group similar tweets, so that each tweet that has the same hash has to be compared to each other only. 2) Second, which is responsible for collecting the same tweets in the same bucket. 3) In the third component, which is the disaster (Catastrophe) detector that works on finding out if the candidate incident is an emergency situation and try to find the location of this case. Then, once a disaster event is identified, all the related information is transmitted to the fourth component, i.e., 4) Alert component, as soon as a disaster event, and its location, type, and representative keywords are identified, these keywords will be used to initiate a new data collection sessions from Twitter, both from the past and from a real-time stream. In [4], First Story Detection (FSD) system that processes each new incoming document in a fixed time and uses a fixed storage is introduced. This fixed processing time is accomplished by using LSH, which is used to divide each query point into parts called buckets. In addition, Locality Sensitive Hashing is used to limit the required space.

## 2. Machine Learning Approaches for Text Classification

In general, this work aims at classifying and identifying Social media data related to the crisis, and this process consists of the following stages:

1) **Data aggregation:** In this step, data is captured from Social media using Twitter Search API. This API allows to collect public tweets. The tweets contain user information, tweet date, location, favorite count, etc. The API also allows to filter tweets by keywords.

2) Data pre-processing: The "Pre-processing" stage regulates the data to ensure an efficient configuration of the classification system. In general, it has been proved that the preprocessing of the text data is an important and essential step and if we skip such step, the chance of badly affected by the noisy and inconsistent data is increased. The objective of this step is to clean by eliminating the noise and irrelevant part of tweets such as punctuation, special characters, numbers, and terms which don't carry much weight in context to the text. The main steps of Data pre-processing are A)

**Tokenization:** Tokenization is the step of splitting longer text strings into smaller pieces or tokens. Large text sets can be divided into sentences, words, and characters. Tokenization is also called text segmentation or lexical analysis. In this work, tokenization is used to separate the text data into words. **B) Stop-word Elimination:** Stop words are functional words specific to language that commonly used (pronouns, prepositions, conjunctions) such as 'the', 'of', 'and', 'to', etc. [5]. In this step, these stop words are removed. Hence, it has been proven that this step is very important for Information Retrieval (IR) and text mining [6, 7]. **C) Normalization:** Before further processing the text data, it must be normalized. Normalization generally consists of a series of related tasks to put all text on a flat playing field: converting all text to the same letter size (upper or lower), removing punctuation, converting numbers to word equivalents, and so on. In addition, stemming, and lemmatization, spelling and grammar correction can also be performed in this steps.

The International Conference of Materials and Engineering Technology

**3)** Feature extraction: In general, words of the text represent discrete, categorical features. Hence, we need to map the textual data into real-valued vectors. In this paper, this step is done using the Word Embedding, which is one of the most popular approaches and can represent the words in low dimensional vector space [4], while efficiently preserving the contextual similarity [7]. The Word2Vec [9] and GloVe [10] are the most popular embedding approaches.

4) Classification: In this paper, the performance of some well-known machine learning approaches, i.e., K-Nearest Neighbors (KNN), Naïve Bayes and Random Forest, AdaBoost Classifier, and GradientBoosting Classifier that can be used in classifying the information available before or even during any crisis have been investigated. In the following, we briefly summarized these approaches.

# I. K-Nearest Neighbor (KNN)

K-nearest neighbors [11], is a simple algorithm that stores all existing neighbors and classifies new data according to the used similarity criteria (ex, distance functions as Manhattan, Euclidean distance) [11]. KNN was used as a non-parametric technique for statistical estimation and pattern recognition tasks in the early 1970s. Hence, in our case, a tweet is assigned to the most common class among its' nearest neighbors [12].

## II. Naïve Bayes (NB)

Naive Bayes is a probabilistic classifier that based on Bayes theorem with naive independence assumption. Naive Bayesian model is easy to build, with no complicated iterative parameter estimation, which makes it particularly useful for very large datasets. Naive Bayes operates by assuming independence, i.e., the presence of some feature will not affect the other features [13]. In other words, Naive Bayes classifier relies on the probability model, and in many practical applications, the method of maximum likelihood is used in parameter estimation for "Naive Bayes" models [14].

## III. Random Forest (RF)

A random forest is an ensemble classifier based on a series of decision tree models [15]. The random forest consists of a large number of individual decision trees working as an ensemble. Each tree in the random forest predicts a class, and the class with the most votes becomes the prediction of our model. These trees that the Random Forest model consists of are relatively uncorrelated. In addition, Random forest applies the technique of bagging (bootstrap aggregating) to decision tree learners. Hence, Bootstrapping enables the random forest to work well on relatively small datasets. However, it is getting more complicated and its execution time increases when it is required to deal with a large number of samples.





# IV. AdaBoost Classifier (AdaBoost)

Similar to the RF, AdaBoost or Adaptive Boost is another ensemble classifier. In general, the AdaBoost classifier is an iterative ensemble method that boosts the performance of a weak classifier by using it within an ensemble structure. The classifiers in the ensemble are added one at a time so that each subsequent classifier is trained on data which have been "hard" for the previous ensemble members. In other words, AdaBoost trains the machine learning model by selecting the training set based on the estimation of the last training [16].

# V. GradientBoosting Classifier (GBC)

Gradient boosting is an ensemble technique for regression and classification problems. This classifier produces a prediction model by sequentially fitting the base learner to current "pseudo"-residuals by least squares at each iteration. As shown in [17], the pseudo-residuals are the gradient of the loss functional being minimized, with respect to the model values at each training data point evaluated at the current step. Determining the weaknesses of weak learnings can be considered as the major difference between the AdaBoost and the Gradient Boost Algorithm. The AdaBoost model identifies weaknesses using high-weight data points, while gradient boosting carries this by using gradients in the loss function [17].

#### **3. EXPERIMENTS**

In this section, the performance of the K-Nearest Neighbor (KNN), Naïve Bayes (NB), Random Forest (RF), AdaBoost Classifier (AdaBoost), GradientBoosting Classifier (GBC), and all possible ensemble systems consisted of three and five of the studied algorithms have been investigated. To ensure the robustness of the experiments, multiple databases have been used in this study and its details as shown in the following sub-section. It is important to note that related to the "K" value for the KNN, we have tested the values in the range [1, 25] and the Grid Search was used to find the best K value. The same process was done to detect the value of the "number of estimators" for the RF Classifier by testing the values {25, 50, 75,100, 150, and 200}. In addition, we have used the Word2Vec word embedding approach in our implementation.

# A. Datasets and Evaluation

In this work, the "socialmedia-disaster-tweets-relevent" dataset [18], which includes 10.860 tweets collected using multiple search keywords such as "ablaze", "quarantine", and "pandemonium", then hand classified as relevant or irrelevant, i.e., two classes: disaster and non-disastrous, where the non-disastrous could be a gossip, rumor, joke or a movie review or something else. It is worth mentioning that a very few tweets in this dataset were classified as "Can't Decide" and these ones are ignored. In addition, we have reconstructed four sub-datasets from this dataset, which contains 2.500, 5.000, 7.500 and 10.860 respectively.

Related to the evaluation, we have used the following main standard metrics that are well known used for evaluating classification systems: 1) F1 score, which is a weighted average of Precision and Recall, consider both false positives and false negatives into account. F1 can be calculated using Equation (1). 2) Accuracy, which is the ratio of the total number of correctly classified tweets divide by the total number of tweets and calculated using Equation (2).

F1 Score = 
$$\frac{2*\text{Recall}*\text{Precision}}{\text{Recall}+\text{Precision}}$$
 (1) Accuracy =  $\frac{TP+TN}{\text{Total}}$  (2)

# **B.** Experiments and Results Analysis

# **Experiment #1: The Effects of Pre-processing the Tweets on the Performance of the Studied Algorithms**

The International Conference

In this experiment, we have investigated the effect of preprocessing the tweets on the performance of each of the studied techniques. The main aims of this experiment are to find the degree that performance can be affected by either preprocessing the data or use it directly. In addition, it is expected that some algorithms can be more sensitive to the noise and unprocessed data than others. In other words, we will find the percentage of improvement, if existed, for each algorithm. It is worth mentioning that the fourth sub dataset, i.e., 10.860 tweets were used in this experiment. The result is shown in Table 1.

Algorithm	Accu	Improvement	
Aigonunn	Without pre-processingWith pre-processing		(%)
KNN	80.53	84.55	7.9
NB	60.31	60.98	0.5
RF	86.28	91.17	5.9
AdaBoost	80.69	83.96	7.9
GBC	82.50	91.06	8.2

**Table 1**: The accuracy of the studied algorithms with and without the pre-processing operation.

As shown in Table 1, it is clear that the preprocessing has improved the performance of almost all the algorithms, and Gradient Boosting Classifier achieved 8.2% as improvement, i.e., it can be considered as the most sensitive. In addition, although, the NB achieved the lowest performance (Accuracy), it has been found that it is the least affected by the noise as its performance with and without the preprocessing is almost same.

## Experiment 2: Robustness and Scalability of the KNN, NB, RF, AdaBoost and GBC

In this experiment, the robustness and scalability of the mentioned techniques were investigated using multiple different size datasets, where the first dataset consisted of 2.500 tweets, the second dataset consisted of 5.000 tweets, the third dataset consisted of 7.500 tweets, and the fourth dataset consisted of 10.860 tweets. Also, in addition to the accuracy, the F1 score, which as mentioned before considers both false positives and false negatives into account has been calculated. Figure 1(a) shows the results of this experiment using the first and second datasets, and Figure 1(b) shows the results using third and fourth datasets, and the results can be summarized as follows.

- 1) Using all the datasets, the RF, AdaBoost, and GBC achieved very good results (approximately the same performance), and for the fourth datasets, which contains a large number of tweets, the performance has been decreased a little bit, however, related to the number of processed tweets it can be accepted. In addition, KNN was able to achieve above 80% accuracy for all the datasets, however, it has been defeated by the RF, AdaBoost, and GBC algorithms.
- 2) Related to the NB, it has achieved the worst performance as compared to the other algorithms using all the four datasets.
- 3) Related to the Robustness and Scalability while increasing the number of processed tweets, all these classifiers showed that it has stability and can handle all the datasets efficiently.

Technology



**Figure 1**: (a) The accuracy and F1 score of the studied algorithms using the first and second datasets, and (b) the accuracy and F1 score of the studied algorithms using the third and fourth datasets.

## **Experiment 3: Performance of Ensemble Systems**

As mentioned before, it has been proven that ensemble learning can overcome the weakness of using the classifiers individually and it has the ability to make the system more accurate, robust and scalable. In this experiment, the performance of the possible ensemble systems that can be created using the KNN, NB, RF, AdaBoost, GBC have been investigated. In other words, we have checked the accuracy of all possible three and five classifiers ensemble systems. The majority voting, i.e., soft, was used to make the final decision on the class that each sample (tweet) belongs to. Based on Table 2, the followings can be derived:

- 1) The results (accuracy) of all the studied ensemble system are good and it is clear that such a system is more stable as compared to any single classifier.
- 2) Overall, the 10<sup>th</sup> ensemble system (RF, AdaBoost, and GBC) can be considered as the best one, as it has achieved the highest accuracy for the first three datasets, and almost achieved same as the 5<sup>th</sup> system for the fourth dataset. In addition, this system (10<sup>th</sup>) outperforms the last system (11<sup>th</sup>), which is consisted of five classifiers. Hence, increasing the number of classifiers in an ensemble may make the system more complicated and at the same time may not improve the performance.



Table 2: The accuracy	of the studied	Ensemble systems	using four	different size	datasets.
<b>Lubic 2</b> . The accuracy	of the studied	Linseniole systems	using rour		uuusets.

Ensemble	Used	Accuracy			
Learning	Algorithms	First	Second	Third	Fourth
#	Algorithms	Dataset	Dataset	Dataset	Dataset
	KNN				
1 <sup>st</sup>	NB	91.52	91.84	86.72	81.67
	RF				
	KNN				
2 <sup>nd</sup>	NB	91.36	91.28	79.36	66.77
	AdaBoost				
	KNN				
3 <sup>rd</sup>	NB	91.04	91.92	86.34	80.92
	GBC				
	KNN		94.56	93.54	89.21
4 <sup>th</sup>	RF	97.92			
	AdaBoost				
	KNN				
$5^{\text{th}}$	RF	96.64	96.24	94.13	90.64
	GBC				
	KNN		95.68	93.65	89.93
6 <sup>th</sup>	AdaBoost	98.40			
	GBC				
	NB	91.68	93.20	90.77	69.15
7 <sup>th</sup>	RF				
	AdaBoost				
8 <sup>th</sup>	NB				
	RF	97.28	95.44	92.69	88.15
	GBC				
9 <sup>th</sup>	NB				
	AdaBoost	95.20	93.76	90.77	75.75
	GBC				
10 <sup>th</sup>	RF				
	AdaBoost	98.56	96.72	95.52	90.00
	GBC				



	KNN				
	NB				
11 <sup>th</sup>	RF	97.28	96.70	93.76	89.10
	AdaBoost				
	GBC				

# **Conclusions and Future Works**

Nowadays, social media analytics is an important research field and has led to improve internet-based crisis management systems. In this work, the performance of the K-Nearest Neighbor (KNN), Naïve Bayes (NB), Random Forest (RF), AdaBoost Classifier (AdaBoost), GradientBoosting Classifier (GBC) for text (tweets) classifying has been investigated. In addition, the ensemble systems consisted of three and five of the studied algorithms have been studied as well. Results show that the preprocessing the text is a must step and has improved the performance of almost all the studied algorithms. In addition, the ensemble system constricted of the RF, AdaBoost, and GBC can be considered as the best for the process of classifying candidate tweets about disasters and crisis. Furthermore, it has been found that by using the ensemble learning techniques, the performance, robust and stability of the classifying process has been significantly improved. However, such an ensemble system can be considered as complicated and its execution time increases when it is required to deal with a very large number of samples.

As future work, this study can be expanded in different ways starting by taking advantages of the multiple available CNN models which can be integrated and considered as very good choices for improving crisis management systems. In addition, a more efficient and professional tweets pre-processing model is essential and can significantly improve the overall performance.

## References

- 1. Terpstra, Teun, et al. *Towards a realtime Twitter analysis during crises for operational crisis management*. Burnaby: Simon Fraser University, **2012**.
- 2. MIDDLETON, Stuart E.; MIDDLETON, Lee; MODAFFERI, Stefano. Real-time crisis mapping of natural disasters using social media. *IEEE Intelligent Systems*, **2013**, 29.2: 9-17.
- 3. GIRTELSCHMID, Sylva, et al. Near real-time detection of crisis situations. In: 2016 39th International Convention on Information and Communication Technology, Electronics and Microelectronics (MIPRO). IEEE, 2016. p. 247-252.
- 4. PETROVIĆ, Saša; OSBORNE, Miles; LAVRENKO, Victor. Streaming first story detection with application to twitter. In: *Human language technologies: The 2010 annual conference of the north american chapter of the association for computational linguistics*. Association for Computational Linguistics, **2010**. p. 181-189.
- 5. AÏT-SAHALIA, Yacine; XIU, Dacheng. Principal component analysis of high-frequency data. *Journal of the American Statistical Association*, **2019**, 114.525: 287-303.
- 6. ROGSTADIUS, Jakob, et al. CrisisTracker: Crowdsourced social media curation for disaster awareness. *IBM Journal of Research and Development*, **2013**, 57.5: 4: 1-4: 13.

7. SHARMA, Dharmendra; JAIN, Suresh. Evaluation of stemming and stop word techniques on text classification problem. *International Journal of Scientific Research in Computer Science and Engineering*, **2015**, 3.2: 1-4.

The International Conference of Materials and Engineering Technology

- 8. NAILI, Marwa; CHAIBI, Anja Habacha; GHEZALA, Henda Hajjami Ben. Comparative study of word embedding methods in topic segmentation. *Procedia computer science*, **2017**, 112: 340-349.
- 9. MIKOLOV, Tomas, et al. Efficient estimation of word representations in vector space. *arXiv* preprint arXiv:1301.3781, **2013**.
- PENNINGTON, Jeffrey; SOCHER, Richard; MANNING, Christopher. Glove: Global vectors for word representation. In: *Proceedings of the 2014 conference on empirical methods in natural language processing (EMNLP)*. 2014. p. 1532-1543.
- 11. NIKHATH, A. Kousar; SUBRAHMANYAM, K.; VASAVI, R. Building a K-Nearest Neighbor Classifier for Text Categorization. *International Journal of Computer Science and Information Technologies*, **2016**, 7.1: 254-256.
- 12. CUNNINGHAM, Padraig; DELANY, Sarah Jane. k-Nearest neighbour classifiers. *Multiple Classifier Systems*, 2007, 34.8: 1-17.
- 13. FRANK, Eibe; BOUCKAERT, Remco R. Naive bayes for text classification with unbalanced classes. In: *European Conference on Principles of Data Mining and Knowledge Discovery*. Springer, Berlin, Heidelberg, **2006**. p. 503-510.
- 14. DIETTERICH, Thomas G. Ensemble methods in machine learning. In: *International workshop* on multiple classifier systems. Springer, Berlin, Heidelberg, **2000**. p. 1-15.
- 15. ONAN, Aytuğ; KORUKOĞLU, Serdar; BULUT, Hasan. Ensemble of keyword extraction methods and classifiers in text classification. *Expert Systems with Applications*, **2016**, 57: 232-247.
- RODRIGUEZ, Juan José; KUNCHEVA, Ludmila I.; ALONSO, Carlos J. Rotation forest: A new classifier ensemble method. *IEEE transactions on pattern analysis and machine intelligence*, 2006, 28.10: 1619-1630.
- 17. FRIEDMAN, Jerome H. Stochastic gradient boosting. *Computational statistics & data analysis*, **2002**, 38.4: 367-378.
- 18. Figure Eight, Disasters on Social Media, https://www.figure-eight.com/data-for-everyone/, **2015**, last visit: 29/8/2019.

# EFFECT OF SECONDARY AGING of EN AC 43200 ALUMINUM ALLOY to MECHANICAL PROPERTIES

The International Conference of Materials and Engineering Technology

# BEDRI BAKSAN<sup>\*1</sup>, IBRAHIM CELIKYUREK<sup>1</sup>, YUSUF KILIC<sup>2</sup>

<sup>1</sup> Eskisehir Osmangazi University, Faculty of Engineering and Architecture, Department of Metallurgical and Materials, Eskişehir, TURKEY.

<sup>2</sup> Delta Dökum San.A.Ş., İstanbul, TURKEY.

#### Abstract

In this study EN-AC 43200 Aluminum alloy was subjected to secondary or interrupted aging following a T6 heat treatment. The 43200 alloy is a used widely in automotive industry for lowering weights of vehicles by substituting with ferrous alloys. This study covers the substitution of an automotive company's part. 4320 Al alloy was melted under Argon atmosphere with an induction furnace, and cast into graphite molds. The samples were homogenized at 500 oC for 96 hours. Solutionizing treatment also done at 500 °C for 14 hours, following water quenching, T6 treatment was done at 250 °C for 2 hours ended with a water quench. Secondary aging trials were done at 100, 150 and 200 °C for 2, 4, 6 and 8 hours for each temperature range respectively. Tensile tests and microhardness tests were applied to for cast, T6 condition, and secondary aged samples. The samples were polished and observed for microstructure under optical microscope. Maximum strength value of 370 MPa, and hardness 113 Hv was obtained from samples aged secondarily at 150 °C for 6 hours.

Keyword: Aluminum alloys, Optical microscopy, Tensile Strength, Hardness, Aging

## **1. Introduction**

For today's automotive manufacturers the substitution of aluminum alloys with high density alloys in engineering applications are major design concerns due to high fuel price [1]. Aluminum and magnesium alloys are the first materials for reducing the weight of automobiles, especially the battery operated cars [2] main structural parts need to have high specific strength. Among these age hardened aluminum alloys are main candidates for reducing the vehicles' weight just as in aerospace vehicles [3]. However, the final cost of structural element is another point to be take care of because of huge mass production rates [4]. The 2XXX and 7XXX series age hardenable alloys more expensive than the EN AC 43200 Aluminum alloy. Therefore the EN AC 43200 alloy said to be compatible with 2XXX or 7XXX series aluminum alloys for its relatively high mechanical strength respect to its cost [5]. The studies about lowering the weight of vehicles goes back to '80s [6, 7]. There are some studies for reducing the weight of military or logistics vehicles by substitution of iron based alloys with aluminum based alloys were realized [8]. There are numerous studies about secondary aging procedure for aluminum alloys. One of the studies was about AlSi10Mg alloy's wear resistance [9], the overview of aging heat treatment of AlSi10Mg alloy was held by Vatansever et.al [10]. There are some studies about the effects of secondary aging and interrupted aging to aluminum alloys indicating

that the secondary aging heat treatment have increasing the mechanical properties [11-15]. The goal of this study is to achieve superior properties from a cheaper aluminum alloy to substitute and compete with iron based alloys as well as 2xxx, and 7xxx series aluminum alloys by secondary aging heat treatment following a T6 treatment. This study was a demand from an automotive manufacturer for reducing vehicle weight by substituting cast iron component with an aluminum alloy.

The International Conference

of Materials and Engineering Technology

#### 2. Materials and Methods

The EN-AC 43200, AlSi10Mg alloy locally obtained in 7 Kg ingots. The ingots cut into small pieces approximately 10-15 mm in cubic forms, then melted in custom made induction furnace under argon atmosphere, and cast into graphite molds to obtain cylindrical ingots in 10mm diameter. All the cylindrical samples were homogenized at 500 °C for 96 hours, and cooled in furnace to overcome the unfavorable effects of fast cooling in graphite mold. Solutionizing heat treatment also done at 500 °C for 14 hours and ended with water quenching. Aging heat treatment was done at 250 °C for 2 hours the samples were quenched in water. Secondary heat treatment procedures were selected as at 100, 150 200 °C for 2, 4, 6 and 8 hours for each temperature range respectively. For every sequence of heat treatment tensile test and micrograph samples were prepared. From micrograph samples microhardness tests also done. The tensile tests were done with Shimadzu AG-IS 250 universal testing instrument with 5 mm/min cross head speed. The samples were ground and polished with Struers polishing instrument, the micrographs were taken with Olympus PMG-3 microscope by Kameram software after etching with Keller etchant. The microhardness tests were done with Future Tech FV-800 hardness tester under 300g load for 10 seconds.

#### 3. Results and Discussion

The tensile testing results of samples are given in Figure 1. As seen from figure the tensile strength of cast sample was found 218 MPa, by the T6 heat treatment the strength was obtained as 280MPa. The secondary aging heat treatment at 100, 150 200 °C for 2, 4, 6 and 8 hours gives us different results. If the tensile testing results inspected, the more stable condition can be said that around 150 °C, at the other temperatures the results are variable. By T6 heat treatment the increase in strength was found to be about 30 %, by adding a secondary heat treatment about 30% over T6, and about 70% over cast condition can be obtained. However the maximum strength obtained at 200 °C for 2 hrs, but the stability and lower temperature concerns, it would be desired to expose the samples at 150 °C for 6 hrs to get maximum strength value of 365 MPa. The specific strength of T6 condition samples are around 85 kN•m/kg, if secondary aging treatment is done this specific strength reaches approximately 120 kN•m/kg value, whereas spheroidal cast iron's specific strength is around 77 kN•m/kg. From these results the heat treated aluminum alloys can be strong candidates for substitution with iron based alloys or cast iron. The wall thickness can be reduced, and the lightweight structures can be manufactured by an about 60% decrease in weight respect to iron based structures.

The hardness values also taken for each sample, the same tendency was observed for the tensile tests (Figure.2). The as-cast sample's hardness value was found as 77 Hv, by a T6 heat treatment the hardness value was found as 101 Hv. Maximum hardness value of 114 Hv was obtained from the secondarily aged samples of 150  $^{\circ}$ C for 6 hours.



Figure 1. Tensile strength of samples at different conditions



Figure 2. Hardness test results of the samples at different conditions

The microstructure analysis revealed that the precipitates of MgSi are responsible for the increase of strength. The  $\Box$ -Al phase mostly seen phase in as-cast samples, by the homogenization heat treatment the  $\Box$ -Al phase islands are rounded. At the grain boundaries eutectic Al-Si structure was observed. With T6 and and additional aging heat treatment Mg<sub>2</sub>Si phase precipitated on  $\Box$ -Al phase. The microstructures of as-cast, T6 condition and secondarily aged samples were given in Figure.3. The cast AlSi10Mg alloy has a microstructure consisting of primary  $\alpha$ -Al dendrites and coarse eutectic mixture in the interdendrite area. Under T6 conditions it is observed that the Si was getting more homogenous and spheroidal in the microstructure, whereas as-cast condition Si is getting thinner eutectic structure. There are different explanations for the effects of secondary phases in casting alloys on the aging process, depending on the alloy compositions involved. It is stated by that the Mg-containing phases that may occur during solidification in the Al-Si-Mg system are Mg2Si and  $\pi$ -Fe phases [9]. In addition, it is stated that  $\beta$ -Fe and  $\pi$ -Fe ratios determine Mg and Fe content and

Technology



solidification rate of the alloy as well as Mg content dissolved in the solid melt. A356 and 357 materials with low iron content (0.1-1.14%) in Al7SiMg alloy, higher magnesium, up to higher temperatures Mg containing  $\pi$  (FeMg3Si6Al8) phase increases the stability of the second, Mg2Si increased the temperature of magnesium  $\beta$ -AlFeSi5 phase [9]. Since the solid solubility of iron in aluminum is very low (0.05%), the iron is generally present in the second phase form as Al-Fe or Al-Fe-Si intermetallic. It is stated that there are three main phases in sub-eutectic and eutectic alloys containing Fe, Mn, and Mg and these are  $\alpha$ -Al15 (FeMn) 3Si2 ( $\alpha$ -Fe),  $\beta$ -Al5FeSi ( $\beta$ -Fe),  $\pi$ Al8FeMg3Si6 ( $\pi$ -Fe) [16]. In our study the detailed study of precipitates was not held. All the assumptions based on previous studies.





**Figure 3.** Microstructures of as-cast (top), T6 condition (middle) 250 °C for 2 hours, secondarily aged (bottom) at 150 °C for 6 hours

Acknowledgments: This study is supported by TUBITAK-TEYDEB Project Number 116054.

# **References:**

- 1. Başer, T., *Alüminyum alaşımları ve otomotiv endüstrisinde kullanımı*. Mühendis ve Makina, 2013. **53**(635): p. 51-58.
- 2. Hofer, J., E. Wilhelm, and W. Schenler, *Optimal Lightweighting in Battery Electric Vehicles*. World Electric Vehicle Journal, 2012. **5**(3): p. 751-762.
- 3. Century, C.M., et al., *Materials Research Agenda for the Automobile and Aircraft Industries*. 1993: National Academies Press.
- 4. Roth, R., J. Clark, and A. Kelkar, *Automobile bodies: Can aluminum be an economical alternative to steel?* Jom, 2001. **53**(8): p. 28-32.
- 5. Abd El-Rehim, A.F. and M.A. Mahmoud, *Transient and steady state creep of age-hardenable Al-5 wt% Mg alloy during superimposed torsional oscillations*. Journal of Materials Science, 2013. **48**(6): p. 2659-2669.
- 6. Century, C.M., et al., *Materials Research Agenda for the Automobile and Aircraft Industries*. 1993: National Academies Press.
- 7. Vehicles, N.R.C.C.o.M.f.L.M. and N.R.C.N.M.A. Board, *Materials for Lightweight Military Combat Vehicles: Report.* 1982: National Academy Press.
- 8. Trucks, C.o.L.M.f.s.C.A., N.M.A. Board, and D.o.E.a.P. Sciences, *Use of LIGHTWEIGHT MATERIALS in 21st century ARMY TRUCKS*, ed. N.R.C.o.t.N. Academies. 2003, U.S.A: National Academies Press.
- 9. GÜL, F., *AlSi10Mg Döküm Alaşımlarının Bazı Mekanik Özellikleri Üzerine İkincil Yaşlandırma İşleminin Etkisi.* Süleyman Demirel Üniversitesi Fen Bilimleri Enstitüsü Dergisi, 2014. **18**(1).
- 10. Fahri VATANSEVER, A.T.E., Sedat KARABAY, *Alüminyum-Silisyum Alaşımlarının Mikroyapısal ve Mekanik Özelliklerinin T6 Isıl İşlemi ile İyileştirilmesi.* Dokuz Eylül Üniversitesi-Mühendislik Fakültesi Fen ve Mühendislik Dergisi, 2018. **20**(60).
- 11. Lumley, R., I. Polmear, and A.J. Morton, *Interrupted aging and secondary precipitation in aluminium alloys.* J Materials Science Technology, 2003. **19**(11): p. 1483-1490.
- 12. Hai, L., Z. Ziqiao, and W. Zhixiu, *Investigation of Secondary Ageing Characteristics of 7055 Aluminum Alloy—(II) Microstructures and Fractography [J]*. J Rare Metal Materials Engineering, 2005. 8.
- 13. Koch, G. and D. Kolijn, *The heat treatment of the commercial aluminum alloy* 7075. J Journal of Heat Treating, 1979. **1**(2): p. 3-14.



- Buha, J., R. Lumley, and A. Crosky, *Microstructural development and mechanical properties of interrupted aged Al-Mg-Si-Cu alloy*. J Metallurgical Materials Transactions A, 2006. 37(10): p. 3119-3130.
- 15. Lumley, R., I. Polmear, and A.J. Morton, *Development of mechanical properties during secondary aging in aluminium alloys.* J Materials Science Technology, 2005. **21**(9): p. 1025-1032.
- 16. Cao, X. and J. Campbell, *Morphology of \beta-Al5FeSi phase in Al-Si cast alloys*. Materials Transactions, 2006. **47**(5): p. 1303-1312.

# PERFORMANCE INVESTIGATION OF DISTRIBUTED ORTHOGONAL SPACE-TIME BLOCK CODING BASED ON RELAY SELECTION IN WIRELESS COOPERATIVE SYSTEMS

The International Conference

aterials and Engineering Technology

# MOHAMMED K.<sup>1</sup> SIDDEEQY.AMEEN<sup>2</sup>

<sup>1</sup>Yousif Dept.of Electronics Techniques Mosul TechnicalInstitute, Northern Technical University Mosul,IRAQ <sup>2</sup>Department of Management Information Systems Applied Science University BAHRAIN

#### Abstract

The idea of cooperative communication is founded as an encouraging technology to advance the performance of communication systems such as transmission rate and bit error rate via channels of transmission. The benefits of multi antenna system can be achieved using relay nodes. Distributed communication terminals can form a cooperative system, some terminals can play the role of relays or fixed terminals designed relay nodes can cooperate and form virtual multi input multi output systems. In this paper the main is to study the promising improvement of cooperating system by using distributed orthogonal space-time codes. By using these codes and suggesting a selection criterion, the performance of the system will improved without needing for increasing the signal to noise ratio. The benefits of cooperative systems could be attained using few relay nodes based on channel quality between the source and the relay nodes and relay nodes to destination node individually. In the suggested model, the broadcast time is shared into two phases by the system. The first one, is called broadcast time has been assigned for the source node to send its information, while the relay nodes on reception mode. Alternatively, the second one, is known as multiple-access time is assigned for relay nodes to deliver the information to the destination node. Simulation values shown an enhancement in the performance is attained with respect to the classical distributed orthogonal space-time codes in expressions of BER and transmission rate.

**Keywords** Cooperative Diversity, Decode and Forward Relaying, Distributed Space-Time Block Codes, Relay Selection& Wireless Cooperative systems.

#### Introduction

Different types of schemes in previous work have been studied and displayed that spatial diversity can be achieved at the system, link or physical layers to offer an effectual transmission. At the physical layer, MIMO system uses multiple antennas at both the transmitter and receiver to attain spatial diversity. Nevertheless, MIMO system needs multi-antennas terminals, which may be unrealistic in small devices because of the size, power of user's terminals and spacing between any two antennas should be more than a half wavelength to preserve all the channels uncorrelated. Thus the idea of cooperative communication has been recommended to offer spatial diversity with single antenna



In this paper a selection algorithm criterion is suggested for wireless cooperative networks. It is shown in performance evaluation that the network diversity gain can be increased if all the relay nodes are used. A network with multi relays which uses decode and forward protocol is adopted. The DOSTBC is then applied at the relay nodes for relaying the data. The selection criterion which used by the destination is to choose the best link out of all links, this aims to enhance the received SNR, and reduce the bit error rate.

The outline of the paper is as follows: Section II discusses cooperative relaying protocols. Section III states the principles of distributed space-time block coding technique. In Section IV, the system and channel model are presented. Then in Section V, the selection scheme is analyzed and presented. Section VI shows the simulation evaluation for the network. Finally, the conclusions are drawn in Section VII.

#### **Cooperative Relaying Protocols**

The relays are one of the main inventions, which improve the coverage of the system and increase the transmission rate as well. A main feature of the cooperative communication progression is the processing techniques of the information expected from the transmitter which be done by the relay nodes. These diverse processing schemes cause different cooperative communications protocols. Cooperative communications protocols can be commonly classified in to amplify and forward and the decode and forward. Amplify and forward is considered the simplest one in which the received signal at the relay node will be amplified and then sent to the destination node [9]. The destination node will receive multi copy of the same information from the relay and source nodes when there is direct link



between the source and the destination nodes as displayed in Fig.1. These versions of signals can be combined in various different methods. The best technique that enhances the SNR is known as the maximum ratio combining [10].



Figure. 1 Amplify and Forward model

The main benefits of this technique are the worthy diversity gain, improved performance than direct communication and high capacity when the relay nodes can be increased as much as possible [11]. But, the disadvantage for this protocol is the amplification of the noise at the relay terminal [12].

The decode and forward protocol, in this protocol the relay node try to decode the received signal to obtain the transmitted signal. Then, the recovered signal is encoded and resend to the destination node as presented in Fig.2. In this protocol the important thing is the noise at the relay node will be excluded by the decoding process. The disadvantage of this protocol when error occurred during the recovery process at the relay node because of the fading in the channel between the source and the relay link, this problem will give a bad performance at the destination node[12].



Figure. 2 Decode and forward protocol

distributed space time block coding

Diversity can be achieved at the source node using the idea of space-time block coding (STBC) with low-complexity at the maximum likelihood detector and good reachable diversity gain as well. STBC is commonly used for wireless networks, it's realized for the collocated antennas, it's easy to deploy at the base stations to enhance the performance of the downlink transmission. But, the implementation of STBC is unfeasible in the uplink transmission due to difficulty in hardware implementation.



The distributed space-time block code can be formerly designed for the distributed fashion of multi hop networks. Consider a wireless system consists of N+2 nodes, which shown in Fig.3 there is one source node and one receive node. All nodes in the system are assumed with single antenna used for transmitting and receiving process. The source-relays channels  $hSR_i$ , and the relays-destination channels  $hRD_i$  without direct link between the source and destination nodes, assuming that the channels are demonstrated as flat fading. The main objective is to transmit the information from the source to the destination with a good performance.



Figure. 3 Cooperative Network Using DSTBC

## **Proposed System Model**

In the system modelling, it is supposed that the model is a multi-hope network with single source and N relays. Also, the source and destination nodes in the system are supposed with single antenna whereas the relays are supposed to have two antennas. The channels are modelled as flat fading channels with additive white Gaussian noise (AWGN) and the channels also are supposed to be uncorrelated.

The model is illustrated in Fig. 4 adopting time sharing in which, the communication is divided between the source and destination into two phases with synchronous transmission between transmitting nodes for each phase.



Figure. 4 Cooperative communication system model

The first phase which called the broadcast phase and the second phase is named the multiple access phase. In the first phase, source node transmits x information symbols to the relays while in the second phase the relays will detect the transmitted symbols, before retransmit it to the destination.



the International Conference

 $y_{rn} = hSR_n x + n_n$  (1)  $n_n = [n_1, ..., n_M]$  defines the vector of the AWGN at the relays. The relay nodes decode and prepare  $\hat{x} = [\hat{x}_1, ..., \hat{x}_M]$  to deliver it to the destination node as shown below:

$$\widehat{\boldsymbol{x}} = \arg\min_{\overline{x} \in \boldsymbol{x}} [\boldsymbol{y}_{rn} - \boldsymbol{h} S R_n \overline{x}]$$
(2)

Where  $\bar{x}$  indicate the vector of all probable transmitted information expected by using the maximum likelihood detector which reduces the Euclidean distance from a signal constellation with the same probabilities.

The DOSTBC is constructed at phase II at the relay nodes, to leverage the code design of matrix C, it is can be determine hypothetically that OSTBC can be implemented in such a way that their columns only contain  $x_1, ..., x_M$  or their conjugates  $x_1^*, ..., x_M^*$  can be applied in distributed nature. The implementation of DOSTBC approach for the two transmit antennas system is done only at phase II which is subdivided in to two consecutive transmission periods. For the first period, the relay nodes transmit two symbols ( $x_1$  and  $x_2$ ) instantaneously from antenna 1 and antenna 2 individually of each relay where \* is the conjugate process as shown in Table I, the coding is done in both space and time domains.

**Table 1.** DOSTBC Sequence for Cooperative System

Transmission Period	Antenna 1	Antenna 2	
First Period	<i>x</i> <sub>1</sub>	<i>x</i> <sub>2</sub>	
Second Period	$-x_{2}^{*}$	$x_1^*$	

The coding matrix C shown below has the following properties:

$$\begin{bmatrix} x_1 & -x_2^* \\ x_2 & x_1^* \end{bmatrix}$$
(3)

The transmitted sequences from the two transmit antennas are orthogonal to each other, then the inner product of the rows of matrix C is equal to zero as shown below :

 $x_1 x_2^* - x_2^* x_1 = 0$ (4) The code matrix also has the following function:  $CC^{H} = \begin{bmatrix} |x_1|^2 + |x_2|^2 & 0\\ 0 & |x_1|^2 + |x_2|^2 \end{bmatrix}$  $= (|x_1|^2 + |x_2|^2) \mathbf{I}_2$ (5) where C<sup>H</sup> is Hermitian matrix of C and  $\mathbf{I}_2$  is 2x2 identity matrix.

The fading coefficients of the channels from the first and second transmit antenna to the destination node are assumed as  $hR_iD_1$  and  $hR_iD_2$  respectively.

Supposing that these coefficients are kept constant over the two periods of communication, which can be stated as shown below:

$$hR_iD_1(t) = hR_iD_1(t+T) = |hR_iD_1|e^{j\theta_1}$$

 $hR_iD_2(t) = hR_iD_2(t+T) = |hR_iD_2|e^{j\theta_2}$ 

where  $|hR_iD_j|$  and  $\theta_j$ , are the magnitude and phase for the channel coefficients from the transmitted antenna of the relay (*i*) to the destination node while T is the transmission period.

#### **Selection Scheme**

Here, the system model is adopting that the relays having the same coding matrix shown above and broadcasting it at the same time, according to that the destination node can estimate the channel state information for each link from the source to relay nodes and from the relay nodes to destination node. The receiver will decide to choice the finest relay that gives the maximum SNR to estimate the transmitted information by source as much as possible by computing the factor f for each relay as shown below which give an indication about channels quality:

$$f_{1} = |hSR_{1}| * |hR_{1}D_{1}| + |hSR_{1}| * |hR_{1}D_{2}|$$
$$f_{N} = |hSR_{N}| * |hR_{N}D_{1}| + |hSR_{N}| * |hR_{N}D_{2}|$$

(7)

(9)

 $\boldsymbol{f} = [f_1, \dots, f_N]$ 

Now the criterion is to find the maximum value of f which maximize the overall SNR at the receiver destination as shown below:

 $f_{max} = max[f_1, ..., f_N]$  (8) After selecting the max value of *f*, these values which iin dictate the best relay, the selecting received signal vector from this relay which can improve the performance of the system is expressed as :

$$r_k = H_k C + n_k$$

where k is the selected relay and  $H_k$  denotes the channel matrix between the selected relay antennas and the destination nodes and is defined as:

$$\boldsymbol{H}_{\boldsymbol{k}} = \begin{bmatrix} hR_{\boldsymbol{k}}D_{1} & hR_{\boldsymbol{k}}D_{2} \\ hR_{\boldsymbol{k}}D_{1} & hR_{\boldsymbol{k}}D_{2} \end{bmatrix}$$
(10)

Assuming *C* is the coding transmitted matrix by the relay and  $n_k$  is the AWGN supposed to be made up of iid random variables. The maximum likelihood decision variables for symbols  $x_1$ ,  $x_2$  are computed as:

$$\begin{bmatrix} \tilde{x}_1\\ \tilde{x}_2 \end{bmatrix} = \boldsymbol{H}\boldsymbol{H}^{\mathrm{H}} \begin{bmatrix} \hat{x}_1\\ \hat{x}_2 \end{bmatrix} + \begin{bmatrix} h_{RkD_1}^* & h_{RkD_2}\\ -h_{RkD_1}^* & h_{RkD_2}^* \end{bmatrix} \begin{bmatrix} n_1\\ n_2^* \end{bmatrix}$$
(11)

(6)

From above it can be see that two information symbols can be detected individually at the same time with low complexity decoding. The SNRs for  $S \rightarrow R_k$  and  $R_k \rightarrow D$  links are given by:

$$\gamma_{SRk} = \frac{p_S |hSR_K|^2}{\sigma^2} \tag{12}$$

$$\gamma_{RkD} = \sum_{i=1}^{M} \frac{p_{l} |nR_{K}D_{i}|}{\sigma^{2}}$$

(13)

The International Conference of Materials and Engineering

Technology

where  $p_s$  and  $p_l$  are the transmitted powers of the source and the relay,  $\sigma^2$  is the variance of the noise and M is the number of transmitted antenna in the relays.

The achievable rate of the transmission is restricted by the ability of the  $S \rightarrow R$  links. But, this can be enhanced upon if the destination is permit to decode the information based only on the signal received, this done by swap to the best relay that has been sent the best information as much as possible which leads to the so called relay selection scheme discussed above. The achievable rate in two phases is given by:

The achievable rate in Phase I at the relay is given by:

$$R1 = log2 (1 + \gamma_{SRk}) \ bps/Hz$$
(14)  
And the achievable rate at destination in Phase II is given by:  
$$R2 = log2(1 + \gamma_{RkD}) \ bps/Hz$$
(15)

VI. Simulation Evaluation

This part, the bit error rate of the suggested model is estimated using the simulation setup as shown in Tabel.2 and compared it with the normal DOSTBC. In this model, every channel state information is assumed to have the following properties.

Table 2. Simulation Setup			
Relay Mode	DF MODEL		
SNR Range (dB)	[5:2.5:50]		
No. of Bits / Slot	2		
No. of Frames	100		
No. of Blocks	5000		
No. of Iterations	2000		
No: Relays	3		
Type of Channel	Rayleigh		
p <sub>s</sub>	Normalized power		
$p_l$	$0.5p_{s}$		
Modulation	BPSK		
AWGN	iid random variables		

Table 2	Simulation	Setup
---------	------------	-------



The International Confer



Figure. 5 Performance comparison of the proposed model with classical DOSTBC for BPSK

Also, extra information may attained by the destination node when selection criterion is applied; these enhance the performance but with some effort drawbacks at the destination. The rate of the transmission is also enhanced as shown in Figs. 6 and 7 by the suggested system if compared with the classical DOSTBC, e.g., at Phase I for 8 bps/Hz there is about 3 dB enhancement for three to one relay selection and about 2.5 dB for the two to one relay selection with respect to conventional DOSTBC, and in Phase II for 8 bps/Hz there is about 2 dB enhancement for three to one relay selection and about 1.5 dB for the two to one relay selection with respect to conventional DOSTBC. This gives the model to raise the average rate.



Figure. 6 Transmission rate of the proposed Model at Relay in Phase I.

Technology

terials and Engineering



Figure. 7 The transmission rate of the suggested model at Destination in Phase II.

# **VII.** Conclusion

In this paper, a proposed relay selection algorithm is suggested for DOSTBC system. The proposed system is investigate for single relay selection out of two and three relays for the Rayleigh flat fading channel, this model try to excluding the power of the source and enhance the BER and bit rate of the system, also the relays can be saved for another user when the channels of transmission are modelled as static and qusai-static channel by sending a feedback to the worst relay(s) to stop transmission. Simulated performance shows that cooperative model may give good diversity gain with respect to single antenna systems, it is important to say that cooperative networks have the power to beat multi input multi output system in terms of BER and transmission rate.

# References

- 1. Sendonaris, A., Erkip, E. & Aazhang, B. (2003) "User Cooperation Diversity Part I : System Description", IEEE Trans. Commun, Vol. 51, No. 11, pp1927-1938.
- 2. "User Cooperation Diversity Part II : Implementation Aspects and Performance Analysis", IEEE Trans. Commun, Vol. 51, No. 11, pp1938-1948.
- **3.** Laneman, J.N., Tse, D.N.C. & Wornell, G.W. (2004) "Cooperative Diversity In Wireless Networks: Efficient Protocols and Outage Behavior", IEEE Trans. Inform. Theory, Vol. 50, No. 12, pp3062-3080.
- **4.** Hunter, T.E. & Nosratinia, A. (2006) "Diversity Through Coded Cooperation", IEEE Trans. Wireless Commun., Vol. 5, No. 2, pp283-289.
- 5. Alamouti, S.M. (1998) "A Simple Transmit Diversity Technique for Wireless Communications", IEEE J. Sel. Areas in Commun., Vol. 16, No. 8, pp.1451–1458.



The International Conference of Materials and Engineering Technology

- 7. Sadek, A.K., Weifeng, Su. & Liu, K.J.R. (2007) "Multi-node Cooperative Communications in i. Wireless Networks", IEEE Transactions on Signal Processing, Vol. 55, No. 1, pp. 341–355.
- 8. Yang, Le. & Yang, H. (2005) "Cooperator Switch Diversity in Cooperative Networks", Communications, Computers and signal Processing, PACRIM. 2005 IEEE Pacific Rim Conference, pp.308-311.
- **9.** Murphy, P., Sabharwal, A. & Aazhang, B. (2009) "On Building a Cooperative Communication System: Testbed Implementation and First Results," EURASIP Journal on Wireless Communications and Networking (in press).
- **10.** Chin, Y., Jayalath , D. & Senadji, B. (2010) "Distributed Orthogonal Space-Time Block codes with Adaptive Diversity Gain," in Proc. 4th International Conference on Signal Processing and Communication Systems, pp. 1-6..
- **11.** Liu, P., Tao, Z., Lin, Z., Erkip, E. & Panwar, S. (2006) "Cooperative Wireless Communications: A Cross-Layer Approach", IEEE Wireless Communications, Vol.13, No.4, pp.84-92.
- **12.** Abrar , M., Gui1, X., Punchihewa, A., Khan , S. & Iqbal , M. (2010) "Cooperative Diversity versus Antenna Diversity in Wireless Communication Systems," In Proc. 4th International Conference on New Trends in Information Science and Service Science, pp. 260–263.

# FEASIBILITY STUDY OF AUTONOMOUS FREIGHT PLATOONING

he International Conference of Materials and Engineering Technology

# Peter John Kay \*1, Ceri Morris<sup>1</sup>, Karolina Pawluczak Rocha<sup>1</sup>

<sup>1</sup> University of the West of England, Faculty Environment and Technology, Department Engineering, Design and Mathematics, Bristol, UK.

#### Abstract

Climate change and air quality are growing issues globally, as such governments are implementing targets to reduce their dependency on fossil fuels. Road transportation is coming under greater scrutiny, as a major source of carbon dioxide emissions. Consequently, a range of technologies and strategies are being investigated to minimise the carbon dioxide emissions from road transportation.

Air drag from road freight accounts for up to 25% of the total fuel consumption. Therefore, reducing the drag on these vehicles can have a significant impact. Platooning is a method in which vehicles drive in each other's wake to minimise aerodynamic drag, similar to the peloton in cycling. With advances in autonomous driving systems of vehicles will be able to drive closer together to increase the fleet carbon dioxide emissions.

This paper presents a feasibility study for the development of autonomous platooning to reduce vehicle emissions. A validated CFD model was utilised to study the impact of vehicle train length on overall fleet carbon dioxide emissions reduction. The results show that the variation of drag force for each vehicle in the platoon is similar and independent of the number of vehicles in the platoon. The variation is analogous to a streamlined body that has the same aspect ratio.

Keyword: CFD, Platooning, Autonomous, Emissions

#### **1. Introduction**

Climate change and air quality are growing issues globally, as such governments are implementing targets to reduce their dependency on fossil fuels. Road transportation is coming under greater scrutiny, as a major source of carbon dioxide emissions. Consequently, a range of technologies and strategies are being investigated to minimise the carbon dioxide emissions from road transportation.

At high speeds most of the vehicle's tractive effort is used to overcome drag forces acting on the vehicle. Therefore, reductions in aerodynamic drag can improve the full efficiency and reduce the total carbon dioxide emissions. At the velocities pertinent to road freight travelling on motorways, form drag is the dominant drag force acting on the vehicle. Form drag can be reduced by modifying the geometry of the vehicle, for example trying to make the trailer more streamlined and adding fairings. However, the drag coefficient of a single vehicle can only be reduced so far without compromising its functionality. Consequently 'platooning' uses the phenomenon of drafting or slipstreaming to reduce the cumulative drag of the platoon. Platooning can be observed in nature, migrating birds and fishes swim in formations and shoals respectively to conserve energy.

With the advent of wireless technology and adaptive control systems it has been possible, in recent years, for vehicles to run in close proximity to each other safely. Several international projects [1,2] have investigated the suitability of platooning strategies. In 2009 the European Commission founded Safe Road Trains for the Environment (SARTRE). The project showed that for 3 vehicles

driving at 56 mph and within 6m there was a fuel saving between 7 and 15%. The first non-European project Partners for Advanced Transportation Technology was conducted in California. The results corroborated the SARTRE project with fuel savings of up to 8% observed.

The International Conference

The separation gap between the vehicles, intuitively, is inversely proportional to the change in fuel savings. A UK department for transport report states that once the gap exceeds 20 m then no benefit, compared to a single vehicle, is observed [3].

Numerous simulation studies have been conducted to examine platooning of multiple vehicles. However, the majority of studies are with a train of five or less vehicles [4,5]. This paper presents a study of long platoon trains (upto 9 vehicles). The platoon trains were simulated using an experimentally validated CFD model. The aim of this work is to understand the benefits of platoon length for long trains. This work will be used in a future study to understand how platoon length will impact on mobility on the road networks.

#### 2. Materials and Methods

In this study the general Class 2 semi-trailer truck was used as the bas vehicle. This class of has a maximum length of 16.5 m and a weight of 44 tons. Figure 1 shows a simplified geometry based on a DAF FX 95 truck and trailer. The model was simplified to a bulk representation of the truck main features, the driver cabin, trailer and tyres. The drawing also includes trailer skirt and roof wind deflector- aerodynamics features widely used in the semi-trucks across Europe.



Figure 1. Dimensions of simplified geometry based on a DAF FX 95 truck and trailer.

To ensure confidence in the CFD simulation it was first validated against experimental measurements. The validation experiments were carried out in a water flume within the university. The validation experiments were carried out in a water flume to increase the Reynolds number of the flow that the trucks experienced.

The velocity of the water in the flume at its maximum flowrate is around 0.4 m/s. An air flow of around 6.6 m/s has the same Reynolds number as a water flow of 0.4 m/s. This means that the highest effective air speed that the scaled models could be subjected to is 24 kph. Although these

Technology



Reynolds numbers are two orders of magnitudes lower than the Reynolds number of the full-scale vehicle. It is still closer that had the validation study taken place in the University wind tunnel.

Three trucks were cut from foam scaled at 35:1. The foam trucks were secured to the bottom of the flume and velocity measurements were made around the model trucks using a SonTek FlowTracker ADV Acoustic Doppler Velocimeter. Figure 2 shows a photo of one of the submerged trucks.



Figure 2. Photo of one of the submerged trucks in the water flume (truck scaled at 35:1)

The water flume was set to its maximum flowrate and the velocity at the front of each truck were measured. A replica of the conditions used in the water flume were recreated using CFD. Table 1 shows the velocity measured at the front of each truck for both the experiment and CFD simulation. The results show that there is good agreement between the experiment and the simulation. The total drag on the first vehicle in the water flume was calculated by assuming a drag coefficient of 0.6. The total drag compared well the value from the simulation. Consequently, the similarity between the results gives confidence in the validation of the CFD model.

<b>Table 1.</b> Summary of CFD validation study.						
Teat	Velocity [m/s]			Coefficient	Total Drag [N]	
Test	Vehicle 1	Vehicle 2	Vehicle 3	of drag [-]	Total Drag [N]	
Water	0.41	0.57	0.03	0.60*	0.29	
Experiment	0.41	0.57	0.05	0.00	0.29	
Water CFD	0.42	0.56	0.04	0.53	0.24	
Air CFD	6.61	6.81	0.82	0.59	0.08	

\* assumed drag coefficient from literature.

Table 1 also shows the results of the CFD simulation with the same Reynolds number using air as the fluid. The table shows that a similar drag coefficient was calculated for the first vehicle, increasing the confidence in the applicability of the CFD simulation.

For the full scale models the number of vehicles in the platoon was increased from three to nine. The separation distance between the vehicle was set to 6 m. This value was chosen as it is in the range of optimum fuel saving of 3-10 m [6]. Figure 3 shows the geometry of the three-vehicle platoon prior to meshing in the software.



Figure 3. Geometry of three vehicle platoon.

An initial mesh sensitivity study was performed. The mesh size used was 0.2 m with a body of influence of 0.1 m around the vehicle cabins, trailers and separation gaps. The standard k- $\epsilon$  model was used. The velocity was set to 27 m/s, which equates to a velocity of 97 kph. A symmetry plane was used along the axis of the vehicles to save computational effort.

# **3. Results and Discussion**

Figure 4 shows the variation of velocity magnitude from the CFD simulation for the three-vehicle platoon. The figure clearly shows that that wake behind the last vehicle decays well within a vehicle length. Therefore the mesh, which extends more than 5 vehicle lengths behind the last vehicle, has captured the characteristics of the flow.



Figure 4. Variation of velocity magnitude

Figure 5 shows the variation of vehicle drag with vehicle in platoon for differing length of platoons. Figure 6 shows the variation of fraction of vehicle drag compared to the first vehicle with vehicle in platoon for differing length of platoons. The results show that for each platoon there is a similar 'shape' of vehicle drag with vehicle number.

The figure shows that, intuitively, the drag on the first vehicle is the same (circa 1800 N) regardless of the length of the platoon. Thereafter the drag on the second vehicle drops to 60% compared with the first vehicle, circa 1100 N. Similar to the first vehicle the drag on the second vehicle is independent of the number vehicles in the platoon.

The drag on the last vehicle is also relatively independent of the number of vehicles in the platoon around 1100 N and 60% compared with the first vehicle. Interestingly the drag on the 'middle' vehicles, defined as the vehicle behind the first two vehicles and ahead of the last vehicle, is also



relatively independent. The drag of the middle vehicles are around 45% compared with the first vehicle, circa 800 N.



Figure 5. Variation of vehicle drag with vehicle in platoon for differing length of platoons.



**Figure 6.** Variation of fraction of vehicle drag compared to the first vehicle with vehicle in platoon for differing length of platoons.

Figure 7 shows the variation of reduction in drag of platoon with platoon size. The figure shows that the drag reduction from the platoon is directly proportional to the size of the platoon. This raises some interesting question about what the maximum size of a platoon could be. From an aerodynamic point of view the longer the platoon the better in terms of fleet carbon dioxide reduction. However, there will be a point in which the platoon size is impractically long and will

have a detrimental effect on traffic flow on the road networks. This will form the basis of a further study.



Figure 7. Variation of reduction in Drag of Platoon with platoon size

## 4. Conclusions

In this study a validated CFD simulation was used to determine the drag reduction of platoons of road freight of various lengths. Platoon lengths from three to nine vehicles, longer than other studies in the literature, were studied. The main conclusions are:

- The drag on the first vehicle in the platoon is independent of platoon length.
- The drag on the second and last vehicle is independent of platoon length and 60% of the lead vehicle.
- The 'middle' vehicles have experience 45% of the drag of the first vehicle.
- The cumulative drag reduction is directly proportional to the length of the platoon length.

# References

- 1. Applus+ IDIADA (2013). Report on Fuel Consumption. [online] SARTRE Safe Road Trains for the Environment. Available at: https://www.sp.se/sv/index/research/dependable\_systems/Documents/The%20SARTRE %20project.pdf [Accessed 2 Mar. 2019].
- 2. Shladover Sc.D, S. (2007). PATH at 20—History and Major Milestones. IEEE Transactions on Intelligent Transportation Systems. [online] Available at: https://ieeexplore-ieee-org.ezproxy.uwe.ac.uk/document/4358931 [Accessed 1 Mar. 2019].
- 3. Ricardo, TRL and TTR (2019). Heavy Vehicle Platoons on UK Roads. [online] Assets.publishing.service.gov.uk. Available at: https://assets.publishing.service.gov.uk/government/uploads/system/uploads/attachment\_ data/file/637361/truck-platooning-uk-feasibility-study.pdf [Accessed 15 Feb. 2019].

Technology



- 4. Michael Siemon, et al, An Integrated CFD and Truck Simulation for 4 Vehicle Platoons. WCX World Congress Experience. 2018-01-0797.
- 5. Arturo, Davila, et al, Environmental Benefits of Vehicle Platooning. Symposium on International Automotive Technology 2013. 2013-26-0142.
- 6. Browand, F., McArthur, J. and Radovich, C. (2004). Fuel Saving Achieved in the Field Test of Two Tandem Trucks. [online] Escholarship.org. Available at: https://escholarship.org/uc/item/29v570mm [Accessed 5 Mar. 2019].

# BEHAVIOUR OF LASER BEAM CHARACTERISTICS IN DIFFERENT FLUIDS

The International Conference of Materials and Engineering Technology

# NECİP FAZIL YILMAZ<sup>1</sup>, MUHAMMED PAKSOY<sup>\*2</sup>, HAKAN ÇANDAR<sup>3</sup>

<sup>1, \*2, 3</sup> Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, TURKEY.

#### Abstract

In the laser process, a high amount of heat is generated in the cutting zone which results as changes in the microstructure of the workpiece. This is one of the main drawbacks of the laser process. Nowadays, the laser beam is guided with water, named as water-guided jet or microjet in order to overcome this problem. There are few studies in the literature which mainly points out the advantages of this new hybrid process and on the effect of process parameters on both cutting quality and material removal rate. This is a novel study that presents the effect of different fluids on the behaviour of the laser beam experimentally.

In the experiments, a diode laser is mounted vertically on an adjustable table and it is radiated into glass tube filled by different fluids as pure water, brine solution, boron oil and hydraulic oil from 10 different heights (sending distance). Distributions of laser beams in the fluids are then monitored by optical lenses and they are modelled by using image processing methods by MATLAB. The results show that the laser beam is best guided into pure water among the other fluids and it shows almost linear characteristic. It is also found that there is a considerable effect of sending distance on the width of the laser beam.

Keyword: Laser Beam, Laser Machining, Waterjet Guided Laser, Microjet

#### 1. Introduction

In recent days, with the influence of developing technology, modern manufacturing methods are widely used in many industrial and technological fields. In order to be strong against to the global competition and to reach big markets, the production costs must be low; the manufacturing processes must be fast and have a high quality. Conventional manufacturing methods do not adequately cope with the goals to be achieved in this fast and technological race. Depending on the technological and scientific developments, modern and automation-based manufacturing methods have begun to play an important role in closing this deficiency. Among these new and modern manufacturing methods, laser-assisted manufacturing technology becomes one of the most preferred methods in the industrial area, especially in high economic and added value sectors such as the medical, automotive, aerospace and microelectronics [1-4].

Lasers provide advantages on precision cutting of even composite materials, hard-to-process ceramics and many various metals. Laser cutting does not cause any mechanical stress, rubbing and abrasion due to the fact that there is no direct contact between the tool and the workpiece as like the other non-traditional processes. In addition, it has an important place in the industry because of its easy removal of complex shapes, its rapidity and a high quality surface processing.



The International Conference of Materials and Engineering Technology

Another problem may arise from gases and liquids used in the laser processing. The workpiece can be directly affected from high energy absorption and it may be damaged because of sudden cooling-heating, melt formation and chemical reactions. As a result of these; various problems such as combustion, sintering, soldering, alloying may occur. Also, depending on the beam diameter of the laser used in micro machining processes, overheating can cause melting of the workpiece [8-9].

Another modern manufacturing method, abrasive water jet cutting (AWJ), is one of the metal cutting processes that are used extensively in various fields and applications, along with today's developing technology. Except from extreme hard materials, abrasive water jet cutting is the preferred method of cutting many materials at the desired thicknesses and shapes. The most remarkable feature of this cutting process is the "precision cold cutting" method, which does not leave a heat-affected cutting zone on the material. Despite these advantages, tapered surface formation and low transverse speeds especially cutting thick and hard materials is limiting this process [10-14].

In order to combine the remarkable advantages of laser process and abrasive waterjet process and to eliminate the drawbacks of these methods, waterjet guided laser technology is firstly developed by Bernold Richerzhagen. A laser microjet was designed and cooling effect of the water, laser cutting distance and pressure of the waterjet was investigated. Later, a hybrid laser and waterjet system designed and preventing thermal effect of the laser, removing of cutting particles from workpiece were researched. Another study was done to search focusing distance of waterjet guided laser. The reflection of the laser beam in the high pressured water wall provided longer cutting distances compared to normal laser processing [15-17]. Jyri et al. was studied on cutting thin sheet metal by using a waterjet guided laser. They performed cutting operations by controlling cutting distance, feed speeds and angle of incidence [18].

Ikeda at al. used a waterjet guided laser for cutting wafer. Wafer was cut by using a conventional laser and a waterjet guided laser. Cutting quality of these methods was compared. Waterjet guided laser cutting provided less structural changes, no drawing, clean cutting edges, narrow beam diameter and slightest mechanical load during wafer cutting processing [19]. Suvradip et al. studied on development of a waterjet assisted underwater laser cutting process produced less turbulence and gas bubbles compared to gas assisted underwater laser cutting process [20]. Regarding to previous studies, waterjet guided laser cutting operations were executed and generally comparison with conventional laser cutting were reported. This study aims to investigate the characteristic of the laser beam in the different fluids before designing a waterjet guided laser to contribute development of waterjet guided laser cutting technology.


## 2. Materials and Methods

In the experiments a diode laser having a wavelength of 450 nm and power of 2.8 W is mounted on an adjustable table as seen in Figure 1 and the laser beam is sent to a glass tube filled by different fluids such as pure water, brine solution 35%, boron oil and hydraulic oil respectively. The laser is radiated to fluid surface from 10 different distances from 12 cm to 3 cm respectively and the photos are taken for each condition. Then the pictures are processed by MATLAB Image Processing Toolbox and they are converted to black and white pictures to determine the beam thicknesses under each solution and for different sending distances. In this process, imadjust and histeq filters are also applied to eliminate noise in the pictures.



Figure 26. Experimental Setup

## 3. Results and Discussion

## 3.1 Effect of Laser Source Distance on Beam Characteristic in the Fluid

Figure 2 presents the characteristics of the laser beam in the pure water. It is seen that the laser beam is well guided in the water. Laser beam travels almost constant in the water for each sending distance. Only a slight difference is seen in the width of the beam between the water surface and end of the tube. But, there is a considerable deviation in the width of the laser beam in the water depending on the sending distance. When the laser is getting closer to the surface of the water, the laser beam in the water becomes thinner. Based on the assumption that the effect will increase with narrowing of the light beam, it can be inferred that the laser beam will be more effective when it is sending farther away from the water surface.

Technology



UWE Bristol

Figure 27. Characteristics of the laser beam in the pure water

Figure 3 shows the characteristics of the laser beam in the brine solution. Similar results are obtained as in the pure water. It is seen that there will be no significant difference by using either pure water or brine solution. When the laser source is getting closer to the water surface, the width of beam becomes larger.



Figure 28. Characteristics of the laser beam in the brine solution

Figure 4 indicates the characteristics of the laser beam in boron oil. Different from the pure water and brine solution, a nonlinear beam profile is obtained in boron oil. As the distance between the laser and liquid surface is getting closer, the beam becomes concave form from the convex form. In general, the width of laser beam increases and reaches to maximum value near the bottom of the tube from the top to the bottom. However, as it is seen in the 3mm distance result, an intimate approach of the laser on the liquid surface shows a positive effect on the laser beam and caused the profile to become thinner near the liquid base. Namely, as the laser beam getting closer to the liquid surface there is a tendency to decrease in the beam width near the bottom of the tube. It is also seen that the width of the beam is greater than the water and cannot focus on a point.

627



Figure 29. Characteristics of the laser beam in the boron oil

Figure 5 shows the characteristics of the laser beam in the hydraulic oil in different heights. The width of the laser beam is first greater at the entrance, it is getting smaller and it travels almost constant after approximately half of the tube. The width of the beam is increasing depending on decreasing distance. However, it cannot reach to the end of the tube independently from the sending distance.



Figure 30. Characteristics of the laser beam in the hydraulic oil

## 3.2 Comparison of the width of Laser Beam in Different Fluids

Thickness values of laser beam for each fluid obtained by using MATLAB image processing toolbox are given in Table 1 and demonstrated in Figure 6. It is clearly seen in the table that the width of the laser beam changes depending on the fluid types and sending distance. As a result of the measurements of the widths, the most thin laser beam is observed in the pure water. The laser beam is always linear and thinner in the pure water. Also, the width of the beam for pure water is generally increasing depending on decreasing laser source distance. It is also seen in this table that the results obtained for both boron oil and hydraulic oil are almost ten times greater than of pure water and brine solution. This difference may arise from the physical properties of these fluids.



Laser Source Distance (cm)	Pure Water (µm)	Brine Solution (µm)	Boron Oil (µm)	Hydraulic Oil (µm)
12	11,20	13,20	177,40	154,20
11	16,40	14,30	168,20	118,90
10	13,90	15,70	162,30	144,10
9	16,20	18,40	153,30	159,10
8	22,90	30,90	154,20	163,90
7	22,40	34,50	152,50	170,20
6	31,60	44,20	146,80	170,50
5	34,50	49,50	149,20	173,60
4	47,90	55,40	145,00	175,80
3	48,70	50,10	139,30	180,60

Table 3 Average	Diameter Measuremen	ts Depending on	Laser Source I	Distance to Fluid Su	rface
<b>I able J.</b> Average	Diameter Measuremen	its Depending on	Laser Source I	Distance to Fiuld Su	mace



Figure 31. Comparison of Average Laser Beam Diameters in the Fluids

## 4. Conclusions

The objective of this study is two folds; to investigate the ability of the laser beam to be guided in easily available cutting fluids and to find out the effect of sending distance in guiding the laser beam. The results show that, the laser beam can be best guided in the pure water between the liquids tested. Also, no significant difference is obtained between pure water and brine solution. Moving from this idea, it can be inferred that the tap water can be directly used for this purpose after filtering. It is also found that, hydraulic oil is not a good choice for using this purpose.

On the other hand, it is found that there is a significant effect of sending distance on the width of the laser beam. As the sending distance increases, the width of the beam decreases. This means that, the laser beam better focuses and it is getting more effectively. However, when the nozzle to



be designed for guiding purpose is considered, it will not be necessary to produce a very long nozzle, because there is no much difference in the width of the beam for pure water after 9cm sending distance.

It may be also concluded that some of the physical properties such as viscosity and density may also affect the characteristic of the laser beam in the liquid. The reason why the laser beam could not be travel along the hydraulic oil may be arise from this reaso

## References

1. Lamikiz, A., Lacalle, L. N. L. d., Sánchez, J. A., Pozo, D. d., Etayo, J. M., & López, J. M, CO<sub>2</sub> laser cutting of advanced high strength steels (AHSS). Applied Surface Science, 2005, 242(3), 362-368.

The International Conference of Materials and Engineering Technology

**2.** Rajaram, N., Sheikh-Ahmad, J., & Cheraghi, S. H., CO<sub>2</sub> laser cut quality of 4130 steel. International Journal of Machine Tools and Manufacture, 2003, 43(4), 351-358.

**3.** Wang, J., Abrasive waterjet machining of polymer matrix composites–cutting performance, erosive process and predictive models. The International Journal of Advanced Manufacturing Technology, 1999, 15(10), 757-768.

**4.** Dubey, A. K., & Yadava, V., Experimental study of Nd:YAG laser beam machining—An overview. Journal of Materials Processing Technology, 2008, 195(1), 15-26.

5. Chryssolouris, G., Anifantis, N., & Karagiannis, S., Laser assisted machining: an overview. Journal of manufacturing science and engineering, 1997, 119(4B), 766-769.

6. Chryssolouris, G., Bredt, J., Kordas, S., & Wilson, E. Theoretical aspects of a laser machine tool. Journal of engineering for industry, 1988, 110(1), 65-70.

7. Jackson, M., & O'Neill, W. Laser micro-drilling of tool steel using Nd: YAG lasers. Journal of Materials Processing Technology, 2003, 142(2), 517-525.

8. Yilbaş, B., & Sahin, A., Oxygen assisted laser cutting mechanism—a laminar boundary layer approach including the combustion process. Optics & Laser Technology, 1995, 27(3), 175-184.

9. Jarosz, K., Löschner, P., & Niesłony, P., Effect of cutting speed on surface quality and heataffected zone in laser cutting of 316L stainless steel. Procedia Engineering, 2016, 149, 155-162.

10. Guo, N., Louis, H., & Meier, G., Surface structure and kerf geometry in abrasive waterjet cutting: formation and optimization. Paper presented at the Seventh American Water Jet Conference Seattle, 1993, Washington.

11. Hashish, M., Optimization factors in abrasive-waterjet machining. Journal of engineering for industry, 1991, 113(1), 29-37.

12. Hashish, M., A model for abrasive-waterjet (AWJ) machining. Journal of Engineering Materials and Technology, 1989, 111(2), 154-162.

13. Wang, J., Kuriyagawa, T., & Huang, C., An experimental study to enhance the cutting performance in abrasive waterjet machining. Machining Science and Technology, 2003 7(2), 191-207.

14. Wang, J., & Wong, W., CO<sub>2</sub> laser cutting of metallic coated sheet steels. Journal of Materials Processing Technology, 1999, 95(1-3), 164-168.

15. Siores, E., Wong, W., Chen, L., & Wager, J., Enhancing abrasive waterjet cutting of ceramics by head oscillation techniques. CIRP Annals-Manufacturing Technology, 1996 45(1), 327-330.

16. Richerzhagen B., Chip singulation process with a water-jet guided laser, Solid State Technology, **2001**, 44(4), 25–28.



17. Tangwarodomnukun V., Wang J., Huang C.Z., Zhu H.T., An investigation of hybrid laser waterjet ablation of silicon substrate. Journal of Machine Tool Manufacturing, **2012**, 56, 39-49.

18. Porter J.A., Louhisalmi Y. A., Karjalainen J. A., Füger S., The International Journal of Advanced Manufacturing Technology; Heidelberg, **2007**, Vol. 33, 961-967.

19. Ikeda T., Ozawa K., Kutsuna M., Waterjet guided laser processing for wafer, Welding in World, **2003**, (47)3.

20. Suvradip M., Yuvraj K.M., Subhransu R., Shailesh K., Dinesh K. S., Ashish K., Development and parametric study of a water-jet assisted underwater laser cutting process, International Journal of Machine Tools and Manufacture, **2013**, (68) 48-55.



of Materials and Engineering Technology

## Fazh BULUT<sup>1</sup>, Kevser GÜNAL<sup>1</sup>, Esin SARIOĞLU<sup>2</sup>, Ahmet AĞAÇ<sup>1</sup>, Şeyda AÇIKGÖZ<sup>1</sup>\*

<sup>1</sup>Arıkan Mensucat San. ve Tic. A.Ş. Ar-Ge Merkezi, Kahramanmaraş. TÜRKİYE <sup>2</sup> Gaziantep Üniversitesi, Güzel Sanatlar Fakültesi, Moda ve Tekstil Tasarımı Bölümü, Gaziantep.TÜRKİYE

#### ÖZET

Günümüzde teknolojik gelişmeler ve artan müşteri talepleri doğrultusunda kullanım, görünüm ve performans özelliklerinin iyileştirilmesi ile su, kir itici, antibakteriyel, anti-stres, buruşmazlık, parlaklık, güç tutuşurluk vb. fonksiyonel özelliklere sahip tekstil ürünü elde edilmektedir. Genel olarak, kumaşlara fonksiyonellik kazandırılması fonksiyonellik özelliği kazandıracak malzemenin kimyasal (yaş) apre uygulaması ile kumaş yüzeyine aplikasyonu şeklinde gerçekleştirilmektedir. Bu uygulamalardan bir tanesi ise sivrisinek kovucu apre uygulamasıdır. Özellikle tropik bölgelerde birçok hastalık taşıması ve bulaştırması nedeniyle sivrisinekleri uzaklaştırmak için kumaşlara sivrisinek kovucu apre uygulaması yapılmaktadır. Bu çalışmada, %100 pamuk süprem ve farklı tasarım örme kumaşlara sivrisinek kovucu apre işlemi uygulanmıştır. Bu uygulanmanın performansını değerlendirmek için süprem kumaşı için sivrisinek kovucu bitim işlemi uygulanmış, uygulanmamış ve 10 tekrarlı yıkama sonrasında kumaşlar üzerinde analiz yapılmıştır. Sonuç olarak, apre işlemi uygulanmış kumaşın sivrisinek iticilik oranının çok yüksek olduğu belirlenmiştir.

Anahtar Kelimeler: sivrisinek kovucu apre, fonksiyonellik, örme kumaş, kumaş tasarımı.

#### ABSTRACT

Today, with aid of technological developments and increasing customer demands, usage, appearance and performance characteristics of textile products having functional properties such as water, dirt repellent, antibacterial, anti-stress, wrinkle, brightness, flame retardant are improved. Generally, the functionalization of the fabrics is carried out by chemical (wet) application of the material that will give the functionality to the surface of the fabric. One of these applications is mosquito repellent finishing application. Especially in the tropics, mosquito repellent finishing is applied to fabrics in order to repel mosquitoes that cause many disease transmissions. In this study, mosquito repellent finishing was applied to 100% cotton single jersey and different design knitted fabrics. Test was performed on fabrics with and without mosquito repellent finishing application and after 10 repeated cycles of washing for single jersey fabric in order to evaluate the performance of this application. As a result, it was determined that mosquito repellency was a very high ratio on the treated fabric.

Keywords: mosquito repellent, functionalization, knitted fabric, fabric design.



## 2. GİRİŞ

Sivrisinekler halk sağlığı sorunlarının önde gelen problemlerinden birisidir, çünkü birçok tür hastalık taşıyıcısı olarak sınıflandırılmaktadır. Küresel ısınma nedeniyle sivrisineklerin dağılımı tropikal bölgelerden kuzey enlemlerine kadar ilerleyerek viral enfeksiyonlar sivrisinekler aracılığıyla yayıldığı görülmektedir [1]. Küresel ısınmanın yanı sıra hızlı kentleşme ve diğer faktörler nedeniyle, sivrisinekler tarafından taşınan hastalıkların etkisi günden güne artmaktadır [2]. Günümüzde sivrisinek kovucu olarak satılan losyonlar ve spreyler çeşitli nedenlerden dolayı sınırlı kullanıma sahiptirler. Bu sınırlı kullanım neticesinde sivrisinek kovucu kumaşların geliştirilmesine ihtiyaç duyulmuştur [3]. Sivrisinek kovucuların tekstil materyaline uygulandığı birçok çalışma mevcuttur [1-8]. Bu çalışmada süprem ve farklı iplik kombinasyonları ile geliştirilen tasarım örme kumaşlara sivrisinek kovucu kimyasal aplike edilmiştir. Kumaş numunelerinin yıkama haslığı, tere karşı (asit ve alkali) renk haslığı ve suya karşı renk haslığı testleri analiz edilmiştir. Ayrıca üretilen kumaşlardan bir tanesi seçilerek biyolojik sivrisinek testi gerçekleştirilmiştir.

The International Conference

aterials and Engineering Technology

## 2. MATERYAL VE METOT

## 2.1. Materyal

Çalışma kapsamında kullanılan süprem örme kumaş ve farklı kombinasyonlarda üretilen kumaşlara ait fotoğraflar Şekil 'de verilmiştir. Bu kumaşlarda kullanılan iplik tipleri Tablo 1'de gösterilmiştir. Kumaş numuneleri üretildikten sonra sivrisinek kovucu kimyasal (GREENFIRST<sup>®</sup> PROFLY) emdirme yöntemiyle aplike edilmiştir.



**Şekil 1.** Çalışma kapsamında üretilen kumaş numuneleri (a) süprem (b) ve (c) üç iplik tasarım kumaşlar

Tablo 1. Kumaşlarda kullanılan iplik tipleri.							
Kumas	1.Kumaş	2.Kumaş	3.Kumaş				
Tini	(a)	<b>(b)</b>	(c)				
1101	Süprem	3 İplik Tasarım Kumaş	3 İplik Tasarım Kumaş				
	Ne 30/1	<b>İplik 1-</b> Ne 30/1 %100	<b>İplik 1-</b> Ne 20/1 %100 Pamuk				
Kullanılan	%100	Poliester Ring İplik	Ring İplik				
	Pamuk	<b>İplik 2-</b> Ne 30/1 %100 Pamuk	İplik 2- Ne 30/1 Ring Melanj				
Ipnk Tiplori	Kompakt	Kompakt İplik	İplik				
ripieri	İplik	<b>İplik 3-</b> Ne 10/1 %100 Pamuk	<b>İplik 3-</b> Ne 10/1 %100 Pamuk				
		O.E İplik	O.E İplik				



## 2.2. Metot

Örme kumaş numunelerine yıkama haslığı [9], asit ve alkali tere karşı renk haslığı [10] ve suya karşı renk haslığı [11] testleri uygulanmıştır. Sonuçların değerlendirilmesi kumaşla birlikte teste tabii tutulan refakat bezini oluşturan asetat, pamuk, naylon, poliester, akrilik ve yün kumaşlarının lekeleme (akma) derecelerinin gri skalada ("1"değeri en düşük haslığı, "5" değeri ise en yüksek haslığı göstermektedir) değerlendirilmesi şeklinde gerçekleştirilmiştir.

The International Conference of Materials and Engineering Technology

Sivrisinekleri kovmayı amaçlayan emdirme yöntemiyle edici bir kumaş işleminin etkinliğinin biyolojik deneyi süprem kumaş numunesine uygulanmıştır. Test prosedüründe sivrisinek kovucu uvgulanmamış, sivrisinek kovucu uvgulanmış kumaş numuneleri kullanılmıştır. Ayrıca sivrisinek kovucu uygulanmış kumaş numunesi 40 °C'de 10 defa yıkandıktan sonra teste tabii tutulmuştur. Toplam 3 tip kumaşa test uygulanmıştır. Test için her bir kumaştan 10cm\*10cm ebatlarında numuneler hazırlanmıştır. Denemeler 50cm\*50cm\*50cm kabin içeren kafeslerde gerçekleştirilmiştir. Testte 4-6 günlük olgun dişi Aedes Aegypti tür sivrisinekler kullanılmıştır. Sivrisinekler 12 saat boyunca ısırmaya istekli olacak şekilde aç bırakılmıştır. Testte sivrisineklerin ısırması için kanlı fare eti kullanılmıştır. Kanlı fare eti üzerine kumaş numuneleri kapatılarak numune üzerine konan sivrisinek sayıları 1 saat boyunca kayıt altına alınmıştır. Test iki replikasyon şeklinde gerçekleştirilerek kumaş üzerine konan sivrisinek sayılarının kümülatif toplamları belirlenmiştir. Sivrisinek iticilik özelliğini belirlemek amacıyla işlem uygulanmış ve uygulanmamış kumaş numune test sonuçları üzerinden Eşitlik (1) kullanılmıştır.

Sivrisinek iticilik (%) = 
$$\frac{AxB}{A}x100$$
 (1)

Burada,

A: İşlem uygulanmamış kumaş numunesine konan sivrisinek sayısı,

B: İşlem uygulanmış kumaş numunesine konan sivrisinek sayısı.

## **3. TARTIŞMA VE BULGULAR**

Çalışma kapsamında örme kumaş numunelerine uygulanan testlerin sonuçları Tablo 2'de gösterilmiştir. Tablo 2 incelendiğinde, bütün kumaşların yıkama haslıklarının 4-4/5 arasında değiştiği görülmektedir. Tere karşı (asit) renk haslığı sonuçlarına göre süprem kumaşın en kötü değerlere sahip olduğu görülmektedir. Bu durum alkali tere karşı renk haslığı sonuçlarında da görülmektedir. Bunun sebebi üç iplik tasarım kumaşlarda poliester iplik muhteviyatından kaynaklanabilir. Suya karşı renk haslığı sonuçlarında ise her üç kumaş tipinde de pamuk ve naylon kumaşların kirlenme değerleri 4 ve 3-4 arasında değiştiği görülmektedir.

Kumaş numuneleri arasından seçilen süprem kumaş numunesine uygulanan biyolojik sivrisinek test sonuçları Tablo 3'de verilmiştir. Tablo 3 incelendiğinde işlem uygulanmış kumaş numunesinin sivrisinek iticilik yüzdesinin %97,4 gibi çok yüksek oranda olduğu görülmektedir. 10 yıkama sonrasında ise sivrisinek iticilik yüzdesinin %52,6'ya düştüğü halde yüksek iticilik özelliği gösterdiği sonucuna ulaşılmıştır.



Table 2	Vuma	m11m11m	anina	ait	toot	0000110	10.00
I abiu 2.	Nullias	IIuIIIuIIC		an	ισδι	sonuç	Iall

The International Conference of Materials and Engineering Technology

UWE Bristol University

Tablo 3. Biyolojik sivrisinek analiz sonuçları								
Tekstile materyali ü	zerine konan sivr	isinek sayısı						
Replikasyon 1 Replikasyon 2 Toplam								
İşlem uygulanmamış kumaş numunesi	73	77	150					
İalam yayayılanmış laymaş nymyanaşi	3	5	8					
işlem uygulanmış kumaş numunesi	% sivrisinek itici	lik: %97,4						
10 yıkama sonrası işlem uygulanmış	38	33	71					
kumaş numunesi	% sivrisinek itici	lik: %52,6						



## 4. SONUÇ

Bu çalışmada, süprem kumaş ve üç iplik kullanılarak gerçekleştirilen tasarım kumaşlar üretilerek bu kumaşlara sivrisinek iticilik özelliğine sahip kimyasal aplike edilmiştir. Kumaş numunelerine haslık testleri uygulanarak sonuçlar analiz edilmiştir. Bununla birlikte bu kumaşlar arasından seçilen süprem kumaş numunesine biyolojik sivrisinek testi uygulanmıştır. Sonuçlar incelendiğinde tasarım kumaşların en iyi haslık değerlerine sahip olduğu görülmektedir. Sivrisinek iticilik özelliğinin yıkama öncesi ve 10 yıkama sonrası sırasıyla %97,4 ve %52,6 olarak değiştiği belirlenmiştir.

The International Conference of Materials and Engineering Technology

## REFERANSLAR

- 1. Anish Sharmila, M, Senthil Priya, P, Boobalan, S and Karthikeyan, LM, Development of mosquito repellent finished cotton fabric using eco friendly cymbopogancitros oil, International Journal of Science, Technology & Management. 2015, 4(2):96-101.
- 2. Raja, ASM, Kawlekar, S, Saxena, S, Arputharaj, A and Patil, PG, Mosquito protective textiles A review, International Journal of Mosquito Research. 2015, 2(4): 49-53.
- 3. Gupta, A and Singh, A, Development of mosquito repellent finished cotton fabric using eco friendly mint, International Journal of Home Science. 2017, 3(2): 155-157.
- 4. Gupta, A and Singh, A, Eco-friendly mosquito repellent finish for cotton fabric, International Journal of Current Research. 2017, 9(7): 53434-53435.
- 5. Sumithra, M and Vasugi Raja, N, Mosquito repellency finishes in blended denim fabrics, International Journal Of Pharmacy & Life Sciences. 2012, 3(4): 1614-1616.
- 6. Tseghai, GB, Mosquito repellent finish of cotton fabric by extracting castor oil, International Journal of Scientific & Engineering Research. 2016, 7(5):873-878.
- 7. Anuar, AA and Yusof, N, Methods of imparting mosquito repellent agents and the assessing mosquito repellency on textile, Fashion and Textiles. 2016, 3(12):1-14.
- 8. Yüce, İ, Sivrisineklerden koruyucu tekstil ürünlerinin incelenmesi, 6th International Symposium on Innovative Technologies in Engineering and Science 09-11 November 2018 (ISITES2018 Alanya Antalya Turkey).
- 9. TS EN ISO 105-C06- Tekstil- Renk haslığı deneyleri- Bölüm C06: Evsel yıkamaya ve ticari müesseselerde yıkamaya karşı renk haslığı.
- 10. TS EN ISO 105-E04- Tekstil Renk haslığı deneyleri Bölüm E04: Tere karşı renk haslığı.
- 11. TS EN ISO 105-E01- Tekstil Renk haslığı deneyleri Bölüm E01: Suya karşı renk haslığı.

# THE EFFECT OF DIFFERENT BED MATERIAL GRAIN SIZES ON THE EQUILIBRIUM SCOUR DEPTH AT THE LABYRINTH SIDE WEIR FLOW

of Materials and Engineering Technology

## MUSTAFA TUNÇ<sup>1</sup>, MUHAMMET EMİN EMİROĞLU<sup>2</sup>

<sup>1</sup> Firat University, Engineering Faculty, Civil Engineering Department, Elazig, TURKEY.
 <sup>2</sup> Firat University, Engineering Faculty, Civil Engineering Department, Elazig, TURKEY.

#### Abstract

With labyrinth weirs, the effective crest length can be increased and more water can be discharged than conventional weirs. Labyrinth weirs play a major role in flood control without damaging the hydraulic structures such as dam, check dam. For this reason, more studies are needed on the labyrinth side weirs in hydraulic engineering. The aim of this study; is to examine the values that the equilibrium scour depth will reach around the labyrinth side weir placed in an open channel. The minimization of the equilibrium scour depth is very important to ensure that the hydraulic structure in question is not destroyed. As it is known, the scour depths formed in the labyrinth side weir flow are less than the conventional side weirs. In this study, a series of experiments were performed by changing the dimensions of the bed material in the main channel. The experiments were conducted for different discharges. Accordingly, the equilibrium scour depth change around the labyrinth side weir at the end of 9 hours was investigated. As a result; It was determined that the equilibrium scour depth was increased with increasing flow intensity, and the equilibrium scour depth was decreased by increasing the median grain size of the bed material.

**Keywords:** Hydraulic structure, Open channel, Labyrinth side weir, Equilibrium scour depth, Bed material, Grain size, Flow intensity.

#### **1. Introduction**

Side weirs are used in many engineering applications. In the channels passed through the valley slopes, the excess flow that will occur due to the surface flow is also removed with the help of side weirs. It is thought that by using side weirs, it is possible to minimize water losses. The side weirs have different cross sections such as rectangular, triangular, trapezoidal and circular side weirs. Side weirs are generally built parallel to the main channel. Amount of the discharge from the weir is depend on the side weir type, channel cross section and the angle of placement of the weir. The hydraulics of the side weirs, which have been studied theoretically and experimentally since ancient times, still attracts the attention of researchers and many studies are carried out.

Scouring downstream of hydraulic structures is one of the major problem encountered in hydraulic engineering. By examining this phenomenon, scour depth should be minimized. Thus, it will be possible to increase the efficiency and life of hydraulic structures. However, sediment transport is observed in an alluvial river depending on the flow velocity [1]. Bed materials transported may cause scouring and deposit downstream of the hydraulic structure. This is undesirable since it will affect the weir flow and cause damage to the hydraulic structure.

Lateral water intake structures constitute the majority of water intake structures in our country. In these structures, the problem of moving sediment is manifested in a significant way due to the weak vegetation of the land and the slope of the rivers. In order to receive water containing less moving sediment from the lateral water intake structures, factors such as water intake structure type, location, crest height, guide rails, gravel passage, separation wall are of great importance.

The decrease in velocity and shear stresses due to lateral water intake creates a stagnation zone downstream of the side weir, causing the reverse current to occur. Due to changes in shear stress, a scour hole is formed between the main channel axis and the outer shore in the downstream region of the side weir.

Labyrinth side weirs are thought to be preferred more because their discharge capacity is higher than other side weirs [2-4]. Most of the studies in the literature regarding the discharge capacities of labyrinth side weirs have been performed for fixed-bottom channel situations.

There are a limited number of studies investigating the scouring around the labyrinth side weir in alluvial channels. Emiroglu et al. (2017) aimed to reduce the scour by preventing the vortex by using plates in the labyrinth side weir flow [5]. In Tunc and Emiroglu (2018a)'studies, the effect of labyrinth side weirs reflected on the bed topography for live bed scour was investigated experimentally [6]. In addition, the bed geometry around classical rectangular weirs has been investigated experimentally in the literature [7,8]. The relationship between bed morphology and discharge from the weir is determined in the literature [9].

The use of labyrinth side weirs is expected to increase due to the high discharge capacity. There are many hydraulic structures in our country which is rich in rivers. It should be ensured that excess water is safely removed from these hydraulic structures. In particular, it is thought that examining the scouring problem for labyrinth side weirs will contribute to the literature.

## 2. Experimental Method

In order to carry out this study, the present experimental setup as shown in Figure 1 was used. This test system consists of a main channel with labyrinth side weir and a collection channel where the water is discharged.



Technology

aterials and Engineering



In the experiments, the discharge of water entering the main channel was determined using an electromagnetic flow meter (Figure 2a). Discharges are measured in "L/s". Level measurements on the main channel and side weir were measured by electronic limnimeter ( $\pm 0.01$  mm accuracy) (Figure 2b). With this limnimeter the measurement can be taken in both x and y directions.



Figure 2. Testing machines used in experiments: a) Electromagnetic flowmeter, b) Digital limnimeter

The experiments were conducted using a triangular labyrinth side weir with weir opening length of L=25 cm, crest height of p=16 cm from the sediment bed and a apex angle  $\theta = 90^{\circ}$  in a straight channel with rectangular cross-section. A view of the labyrinth side weir flow is presented in Figure 3. The experiments were carried out under stable flow conditions, in the case of a free flow state of the live bed scour conditions ( $V_1/V_c>1$ ). Bricks are placed at certain points of the channel to ensure stable flow condition. In order to provide free flow and prevent any interference to the flow, the part that passes from the side weir to the collection channel is circular enough outward in size (Figure 3). In order to minimize the surface tensile effect, the minimum nappe height was taken as 3 cm [10-12].



Figure 3. Labyrinth side weir flow



Quartz sand with different grain sizes ( $d_{50}\approx 1.2 \text{ mm}$ ,  $d_{50}\approx 1.5 \text{ mm}$  and  $d_{50}\approx 1.8 \text{ mm}$ ) was placed in the main channel. The sediment bed, 20 cm high, was leveled on each side of the channel and made ready for testing (Figure 4a). Water was slowly introduced into the channel to prevent distortion of the flat sediment bed. After the certain discharge was set, the experiment was started. It is known that 9 hours is sufficient for reaching the equilibrium scouring depth [13]. After the experiment, the view of the sediment bed in the main channel is presented in Figure 4b.



Figure 4. Sediment bed: a) Before the start of the experiment, b) After the end of the experiment

After the end of the experiment, the maximum scour depth around the side weir area was measured with the digital limnimeter (Fig. 2b).

In this study, grain sizes and specific gravities of quartzitic sands were determined by experiments carried out in Hydraulic Laboratory of Firat University Civil Engineering Department. The specific gravity of the bed materials used is approximately  $\gamma_s=2.65$  g/cm<sup>3</sup>. Grain-size curves of the bed materials used in the experiments are given in Figure 5(a-c). According to Figure 5, uniform material is used.



**Figure 5.** Grain-size curves of quartzitic sediment: a) for  $d_{50}\approx 1.2$  mm, b) for  $d_{50}\approx 1.5$  mm, c) for  $d_{50}\approx 1.8$  mm

## 3. Experimental Results and Discussion

As a result of this study, the relationship between the flow intensity  $(V_1/V_c)$  and the equilibrium scour depth  $(d_{se})$  was observed in a rectangular cross-section straight channel for *L*=25 cm weir opening length and *p*=16 cm crest height at the triangular labyrinth side weir flow with different discharge values and live bed scour conditions. In the case of live bed scour condition  $(V_1/V_c>1)$ , it was observed that the sediment transport occurred at a very high level as the dunes were formed in a short time and the bed was constantly in motion (Figure 4b). Therefore, the amount of spilled sediment from the weir increased continuously with the increase of " $V_1/V_c$ " value. It was assumed that the equilibrium scour depth was reached when there was less than 1% change for the scour depth at a successive hour. The equilibrium scour depth generally occurred at the downstream end of the triangular labyrinth side weir (Figure 6).



Figure 6. Location of equilibrium scour depth

The critical flow velocities ( $V_c$ ) that will mobilize the bed material were calculated by Equation (1) [14,15].

$$\frac{V_c}{u_{*kr}} = 5.75 \log\left(5.53 \frac{y}{d_{50}}\right) \tag{1}$$

where  $u_{*c}$ =critical shear velocity (m/s), y=flow depth in the main channel (m),  $d_{50}$ =median grain size.

Shear velocities are determined with the help of Shields Diagram. For quartz sand at 20 °C, Equation (2) was obtained using the Shields Diagram [14,15].

$$u_{*c} = 0.0305 d_{50}^{0.5} - 0.0065 d_{50}^{-1} \quad ; \quad 1 \text{ mm} < d_{50} < 100 \text{ mm}$$
<sup>(2)</sup>

For  $d_{50}=1.2$  mm, equations (1) and (2) were used to obtain Equation (3) for critical velocities at various depths of the flow that could mobilize the bed material.

$$V_c = 0.1610 \times \log(4608 \times y)$$
(3)

For  $d_{50}=1.5$  mm, equations (1) and (2) were used to obtain Equation (4) for critical velocities at various depths of the flow that could mobilize the bed material.

$$V_c = 0.1899 \times \log(3687 \times y) \tag{4}$$

For  $d_{50}=1.8$  mm, equations (1) and (2) were used to obtain Equation (5) for critical velocities at various depths of the flow that could mobilize the bed material.

$$V_c = 0.2145 \times log(3072 \times y)$$
 (5)

The dimensions of weir, flow characteristics, grain sizes of bed materials, the experimental results from this study are presented in Table 1.

Conference of Materials and Engineering Technology

Table 1. Flow conditions and test results											
Exp. No	<i>p</i> (cm)	L (cm)	<i>Q</i> <sub>1</sub> (L/s)	y (cm)	<i>B</i> (cm)	<i>d</i> <sub>50</sub> (mm)	V <sub>1</sub> (m/s)	V <sub>c</sub> (m/s)	$V_1/V_c$ (-)	d <sub>se</sub> (cm)	dse∕p (-)
1	16	25	65	21	50	1.2	0.62	0.48	1.29	5.1	0.32
2	16	25	75	20	50	1.2	0.75	0.48	1.56	5.6	0.35
3	16	25	80	19	50	1.2	0.84	0.47	1.79	6.4	0.40
4	16	25	90	19	50	1.2	0.95	0.47	2.02	9.2	0.58
5	16	25	110	19	50	1.2	1.16	0.47	2.47	11.5	0.72
6	16	25	65	21	50	1.5	0.62	0.55	1.13	4.7	0.29
7	16	25	75	20	50	1.5	0.75	0.54	1.39	5.3	0.33
8	16	25	80	19	50	1.5	0.84	0.54	1.56	6.0	0.38
9	16	25	90	19	50	1.5	0.95	0.54	1.76	8.5	0.53
10	16	25	110	19	50	1.5	1.16	0.54	2.15	10.2	0.64
11	16	25	65	21	50	1.8	0.62	0.60	1.03	4.2	0.26
12	16	25	75	20	50	1.8	0.75	0.60	1.25	4.5	0.28
13	16	25	80	19	50	1.8	0.84	0.59	1.42	5.3	0.33
14	16	25	90	19	50	1.8	0.95	0.59	1.61	7.4	0.46
15	16	25	110	19	50	1.8	1.16	0.59	1.97	9.2	0.58

In this section, the variation of dimensionless equilibrium scour depth  $(d_{se}/p)$  with flow intensity  $(V_1/V_c)$  under different flow conditions is examined and given in Figure 7(a-c). In Figure 7(a-c), it is seen that is occurred scour for all  $V_1/V_c$  values, constant side weir opening length (L=25 cm) and constant crest height (p=16 cm). As the median grain diameter  $(d_{50})$  increased, it was determined that flow intensity and equilibrium scour depth decreased. It has been determined that with the increase of flow intensity  $(V_1/V_c)$  by about 90-92%, the dimensionless equilibrium scour depth  $(d_{se}/p)$  increases by about 120-125%. Moreover, it was determined that the median grain diameter  $d_{50}$  increased by 25%, the equilibrium scour depth  $d_{se}/p$  decreased by about 5-11%. Similarly, it was determined that the median grain diameter  $d_{50}$  increased by about 8-20%.



UWE Bristol

فليفة

ET?





Figure 7. Variation of  $d_{se}/p$  with  $V_1/V_c$ : a)  $d_{50}=1.2$  mm, b)  $d_{50}=1.5$  mm, c)  $d_{50}=1.8$  mm

646

## 4. Conclusions

UWE Stime

The conclusions of the present experimental study are summarized below:

In this study, equilibrium scour depths were measured for labyrinth side weir flow in an open • channel where bed material of different grain sizes was placed.

MET

The International Conference of Materials and Engineering Technology

- The experiments were performed for different discharges. •
- As the effective crest lengths are high, it is thought that the labyrinth weirs will distribute the • water to a larger area than the conventional weirs and take place a smaller scour depth at the downstream.
- Since the experiments were carried out for high flow intensities  $(V_1/V_c>1)$ , scour and dunes • were observed in all experiments.
- In case of live bed scour conditions  $(V_1/V_c>1)$ , it was observed that the scours and dunes were • formed in a short time.
- It has been determined that the flow intensity  $(V_1/V_c)$  decreases by increasing the median grain • diameter  $(d_{50})$  of the bed material.
- It has been determined that the equilibrium scour depth  $(d_{se})$  decreases by increasing the • median grain diameter  $(d_{50})$  of the bed material.
- It has been determined that with the increase in flow intensity  $(V_1/V_c)$ , the dimensionless • equilibrium scour depth  $(d_{se}/p)$  increases.
- The amount of bed material spilled from the labyrinth side weir increased with increasing flow • intensity  $(V_1/V_c)$ .
- The location of the equilibrium scour depth is generally observed as the downstream end of • the triangular labyrinth side weir.
- It is thought that examining the scouring problem for labyrinth side weirs will contribute to • the literature and the related engineers.
- It is recommended that this study be conducted for larger sediment median grain diameters, • different side weir types and sizes.

## References

- Tunc, M., and Emiroğlu, M. E. Experimental Investigation of Sediment Transport for Side 1. Weir Flow. Journal of Water Resources, 2019, 4(1), 1-11.
- Emiroglu, M. E., and Kaya, N. Discharge coefficient for trapezoidal labyrinth side weir 2. in subcritical flow. Water resources management, 2011, 25(3), 1037-1058.
- Borghei, S. M., Nekooie, M. A., Sadeghian, H., and Ghazizadeh, M. R. J. Triangular 3. labyrinth side weirs with one and two cycles. Proceedings of the ICE-Water Management, **2012**, 166(1), 27-42.
- Emin Emiroglu, M., Cihan Aydin, M., and Kaya, N. Discharge Characteristics of a 4. Trapezoidal Labyrinth Side Weir with One and Two Cycles in Subcritical Flow. Journal of Irrigation and Drainage Engineering, 2014, 140(5).
- 5. Emiroglu, M. E., Gogus, M., Tunc, M., and Islamoglu, K. Effects of Antivortex Structures Installed on Trapezoidal Labyrinth Side Weirs on Discharge Capacity and Scouring. Journal of Irrigation and Drainage Engineering, **2017**, 143(6), 04017006.
- 6. Tunç, M., and Emiroğlu, M. E. Investigation of Live-Bed Scour at Labyrinth Side Weirs. Firat University Turkish Journal of Science & Technology, 2018a, 13(1), 129-136.
- Rosier, B., Boillat, J. L., and Schleiss, A. J. Influence of lateral water withdrawal on bed 7. form geometry in a channel. Journal of Hydraulic Engineering, 2011, 137(12), 1668-1675.



- 8. Alluvial Channel. Irrigation and Drainage, 2013, 62(4), 488-500.
- 9. Paris, E., Solari, L. and Bechi, G. Applicability of the De Marchi hypothesis for side weir flow in the case of movable beds. Journal of Hydraulic Engineering, 2012, 138(7), 653-656.
- 10. Novak P., and Cabelka J. Models in Hydraulic Engineering, Pitman Publishing Limited, London. 1981.
- 11. Tunç, M., Emiroğlu, M.E., Doğan, Y., and Kaya, N. The Effect of Anti-vortex Plates on Scouring at Labyrinth Side Weirs. 8 th International Advanced Technologies Symposium, **2017**,3826-3833.
- 12. Tunc, M., and Emiroğlu, M.E. Relationship between sediment transport and scour geometry for high flow velocities at the labyrinth side weir. 5th International Symposium on dam Safety, 2018b,462-470.
- 13. Dey, S., and Raikar, R. V. Scour below a high vertical drop. Journal of Hydraulic Engineering, 2007, 133(5), 564-568.
- 14. Melville, B. W., and Chiew, Y. M. Time scale for local scour at bridge piers, Journal of Hydraulic Engineering, ASCE, 1999, 125(1), 59-65.
- 15. Tunc, M. An investigation of the hydrodynamics of flow at the labyrinth side weirs in the movable bed rivers, Firat University, Graduate School of Science. 2014. (in Turkish).



## A NUMERICAL APPROACH TO ESTIMATE THE TENSILE STRENGTH OF STRUCTURAL LIGHTWEIGHT CONCRETE

## ESRA TUĞRUL TUNÇ<sup>1</sup>, KÜRŞAT ESAT ALYAMAÇ<sup>2</sup>, ZÜLFÜ ÇINAR ULUCAN<sup>3</sup>

<sup>1</sup>Firat University, Engineering Faculty, Civil Engineering Department, Elazig, TURKEY.

<sup>2</sup> Firat University, Engineering Faculty, Civil Engineering Department, Elazig, TURKEY.

<sup>3</sup> Firat University, Engineering Faculty, Civil Engineering Department, Elazig, TURKEY.

#### Abstract

Since lightweight concrete has many advantages over traditional concrete, it is of great importance for the building industry to investigate more and examine its mechanical properties. It is considered that it will be very useful to use lightweight concretes in special concrete class if they have sufficient strength properties. Structural lightweight concrete can be produced from different lightweight aggregates. Expanded clay aggregate, which is one of them, is thought to have an important place in the production of structural light concrete with its high reserve and easy availability. At the same time, structural lightweight concrete can improve the properties of structures such as earthquake resistance and thermal insulation. The aim of this study; a series of concrete specimens produced with different mixing ratios containing lightweight expanded clay aggregate is to develop a numerical model using the tensile strength test results in the literature. For this purpose, the studies on structural lightweight concrete have been investigated in detail in the literature. With this numerical model, it is hoped that the tensile strength can be easily calculated depending on the content of cement, aggregate, powder/filler, silica fume, water, lightweight expanded clay aggregate and super plasticizer. In this context, the parameters affecting tensile strength were determined. The effects of these parameters on tensile strength were interpreted with related graphics. In the numerical model developed in this study, tensile strength affected by the related parameters is included as output. As a result; with the numerical model developed using nonlinear statistical analysis, it is planned to develop a practical equation with high precision that can be easily used by application engineers.

Keywords: Structural lightweight concrete, Tensile strength, Lightweight expanded clay aggregate, Numerical model.

#### **1. Introduction**

Concrete, which is a good structural bearing, has disadvantages such as high unit weight and high thermal conductivity coefficient. By reducing the unit weight of conventional concrete, it is possible to reduce the structures by reducing the self-weight of the reinforced concrete factors. Lightweight concrete is generally classified according to both unit weight and strength. Lightweight concretes vary according to different standards. According to ASTM C330-69, concretes with unit weights not exceeding 1840 kg/m<sup>3</sup> and having a cylinder compressive strength of 28 days of more than 17 MPa are classified as lightweight concrete [1]. In TS 2511, the dry unit weight of less than 1900 kg/m<sup>3</sup> and BS16 class concrete is defined as lightweight concrete [2]. In

DIN 1045, it is stated that the unit weight of lightweight concrete varies between 300-2000 kg/m<sup>3</sup> [3].

Lightweight aggregates that form lightweight concrete can be obtained as natural or artificial [4]. Substances such as pumice (pumice), volcanic tuff, perlite, vermiculite are naturally obtained light aggregates. The materials such as fly ash, blast furnace slag, expanded clay, expanded perlite are produced artificially and used as lightweight aggregate [5,6]. One of the most durable aggregates among artificial lightweight aggregates is expanded clay aggregate [7]. The expanded clay aggregate is thought to have an important place in the production of structural lightweight concrete, due to the appropriate raw material and technology, which enables the production of materials of desired grain size and properties. Expanded clays according to their commercial production and technical designations all over the world; It is also known as Liapor, lightweight expanded clay aggregate (LECA) and keramzite [8]. When the previous studies in the literature are examined; Lo et al. (2008) examined the effect of LECA aggregates' water absorption capacity on the gap distribution at aggregate-cement paste interface [9]. Bartolini et al. (2010) examined the sound insulation performance of LECA with epoxy and concluded that LECA shows high level sound insulation performance [10]. Self-compacting concrete specimens were prepared using LECA and lightweight concretes with compressive strength in the range of 37.4 MPa-60.8 MPa were obtained in Bogas et al. (2012) [11]. Costa et al. (2012) examined the shrinkage properties of lightweight concrete produced with LECA [12]. Uglyanitsa et al. (2014) investigated the presence and corrosion conditions of reinforced concrete produced with LECA [13]. Yang et al. (2014), examined the mix-design of lightweight concretes containing LECA and developed an empirical formula to calculate values such as compressive strength, dry unit weight [14]. Similarly; Tuğrul Tunc et al. (2018) examined the mix-design of lightweight concrete containing pumice aggregate and obtained a high accuracy formula to calculate the compressive strength [15]. Tunc et al. (2019) and Saglam et al. (2019) developed equations that can easily and reliably calculate the compressive strength of lightweight concretes containing different lightweight aggregates [16,17].

Due to Turkey have been exposed to major earthquakes have great financial and emotional damages, it was thought that the need to reduce the dead load of the building. This will be possible by using building materials produced with lightweight aggregates in buildings. The way to minimize the building load without reducing the strength characteristics of buildings is to produce lightweight concrete using lightweight aggregates. LECA, which is one of the lightweight aggregates, can be preferred in structural lightweight concrete production due to the abundant reserve in our country.

The aim of the present study is to develop a numerical model by using the tensile strength test results of structural lightweight concrete containing LECA from previous studies. For this purpose, a detailed literature review was researched about structural lightweight concrete and experimental data were collected to be used in nonlinear statistical analysis. With the developed equation, it is aimed to calculate the tensile strength of structural lightweight concrete with high accuracy. Thus, both the preliminary mixture design of concrete and the tensile strength of the lightweight concrete can be determined without testing. Today, due to the increasing need for structural light concrete, it is expected to provide labor, time saving and economic gain with the present study.

## 2. Materials and Statistical Method

#### a) Materials

According to ACI 213R-03; structural lightweight concrete, 28 days' minimum compressive strength 17 MPa, density between 1120-1960 kg/m<sup>3</sup> or defined as the concrete produced with lightnormal aggregate combination [18]. As a special type of concrete structural lightweight concrete, especially in recent years has been used for structural purposes. Nowadays, structural lightweight concrete is generally used to reduce the dead load of a building and the weight of the reinforced concrete building factors to be used. However, it plays an active role especially in increasing the usage areas and openings of tall buildings [19]. Structural lightweight concrete; It is mainly used in the construction of walls, panels and blocks, in roof floors, bridge openings, pre-built concrete units. For this purpose, it can be preferred to produce structural lightweight concrete is preferred in skyscrapers, bridges, piers, platforms and many other structures. The Hagia Sophia Museum in Istanbul and the Maya Pyramids in Mexico are some of the magnificent antique structures built using lightweight concrete to date (Figure 1).

The International Conference

aterials and Engineering Technology



Figure 1. a) Maya Pyramids, b) Hagia Sophia Museum [21]

Clay, one of the oldest raw materials, has never lost its importance until today. When natural clays are heated above 1000 °C, they form a gas filled porous structure due to the expansion of the gases in their structures. Thus, the expanded clay is formed [22]. Expanded clays are suddenly exposed to high temperatures in heated rotary furnaces. Clays heated between 1000-1300 °C according to the mineralogical structure of natural clay can produce volume increase of 1.5-6 times their initial volume [5]. However, their density varies between approximately 320 and 960 kg/m<sup>3</sup> [23]. Expanded clays are an artificial material and are not found in the expanded state in nature. Every clay found in nature does not expand. The raw materials used to produce expanded clay are early sintered clay, sandy clay, clayey schist and shale [5]. Expanded clays provide good heat and sound

insulation as well as lightened the structure. Owing to its robust sintered shell, it has the highest compressive strength among similar lightweight aggregates. Thus, according to the same compressive strength class concrete, the structure is lightened by approximately 25%-35%. Expanded clay aggregates have many advantages compared to other lightweight aggregates with this aspect.

1

#### b) Statistical Method

The statistical program used in this study is an advanced analytical software developed by StatSoft (StatSoft Inc., USA) [24]. The program can successfully provide services such as data analysis, data management and data visualization. Numerical modeling and regression analysis based on various estimates can be performed with this program. Different versions are available. The program includes analytical and research graphs in addition to standard 2D and 3D graphs. It allows the search for outliers and data analysis. Different estimation methods can be tried with this program (Figure 2). In this study, nonlinear estimation based numerical analysis was performed.



Figure 2. Different estimation methods developed with the software program

In this study, experimental data of previous studies investigating the tensile strength ( $f_t$ ) of structural lightweight concretes produced using LECA aggregates were used. Mixture amounts of materials such as cement, aggregate, LECA, powder, super plasticizer, silica fume and water given in the previous studies were converted into dimensionless parameters (Table 1). With using these data, regression analysis was performed using this program and a non-linear equation, Equation (1), was developed to calculate the tensile strength ( $f_t$ ) of structural lightweight concrete. This equation was obtained by performing many analyzes in the program. Because the formula that gives the correlation coefficient R<sup>2</sup> closest to 1.0 is tried to be determined. In the program, " $f_t/f_{tort}$ " values consistent with experimental results, the coefficients of the relevant parameters were determined by the program (Figure 3).

$$\frac{f_t}{f_{tort}} = -0.327 \times \frac{LECA}{TA} + 2.376 \times \frac{FA}{TA} + 2.609 \times \frac{CA}{TA} - 0.457 \times \frac{P}{C} + 1.737 \times \frac{SF}{C} - 3.384 \times \frac{W}{C} + 1.476 \times \frac{SA}{C}$$
(1)

where;  $f_t$ =tensile strength (MPa),  $f_{tort}$ =mean tensile strength (MPa), *LECA*=lightweight expanded clay aggregate content (kg/m<sup>3</sup>), *FA*=fine aggregate content (kg/m<sup>3</sup>), *CA*=coarse aggregate content (kg/m<sup>3</sup>), *TA*=total (fine + coarse) aggregate content (kg/m<sup>3</sup>), *P*=powder content (kg/m<sup>3</sup>), *C*=cement

content (kg/m<sup>3</sup>), *SF*=silica fume content (kg/m<sup>3</sup>), W=water content (kg/m<sup>3</sup>), *SA*=super plasticizer content (kg/m<sup>3</sup>).

ME

Conference of Materials and Engineering Technology

Continue	Dep. var: FTO Loss: (OBS-PRED)**2 Final loss: ,095658845 R=,96886 Variance explained: 93,870%									
N=18	A	K	L	в	с	D	G			
Estimate	-,326733	2,375702	2,608750	-,457256	1,736916	-3,38419	1,475786			

Figure 3. The coefficients of the relevant dimensionless parameters obtained by the numerical analysis

The numerical  $f_t/f_{tort}$  values obtained from Equation (1) are presented in Table 1.

UWE Bristol

**Table 1.** Mixture design, experimental  $f_t/f_{tort}$  values from the previous studies and numerical  $f_t/f_{tort}$  values from this study.

The previous studies	LECA/TA	FA/TA	CA/TA	P/C	SE/C	W/C	SA/C	$f_t/f_{tort}$	$f_t/f_{tort}$
The previous studies		111,111		170	5170	me	511,0	(experimental)	(numerical)
[25]	0.620	1.000	0.000	0.000	0.000	0.480	0.000	0.518	0.549
[20]	0.600	1.000	0.000	0.000	0.000	0.467	0.000	0.633	0.600
	0.294	1.000	0.000	0.500	0.000	0.350	0.018	0.991	0.893
	0.294	1.000	0.000	0.500	0.000	0.350	0.018	0.847	0.893
[26]	0.294	1.000	0.000	0.563	0.000	0.400	0.015	0.809	0.691
	0.294	1.000	0.000	0.500	0.000	0.350	0.018	0.821	0.893
	0.294	1.000	0.000	0.563	0.000	0.400	0.015	0.590	0.691
	0.538	1.000	0.000	0.000	0.111	0.444	0.020	0.985	0.920
	0.538	1.000	0.000	0.000	0.111	0.444	0.020	0.933	0.918
	0.538	1.000	0.000	0.000	0.111	0.444	0.020	0.824	0.918
	0.428	1.000	0.000	0.000	0.111	0.356	0.022	1.296	1.258
	0.295	0.688	0.312	0.000	0.111	0.356	0.022	1.348	1.375
[27]	0.381	0.707	0.293	0.000	0.111	0.356	0.0167	1.230	1.334
	0326	0.770	0.230	0.000	0.111	0.311	0.013	1.610	1.483
	0488	0.742	0.258	0.000	0.111	0.388	0.013	1.212	1.176
	0694	0.742	0.258	0.000	0.101	0.389	0.025	1.158	1.106
	0720	1.000	0.000	0.000	0.100	0.341	0.015	1.129	1.183
	0.327	0.759	0.241	0.000	0.111	0.311	0.015	1.431	1.487





## 3. Results and Discussion

In the previous section, it was stated that Equation (1) was developed for the calculation of  $f_t$ . The statistical program and method in which this equation is developed is explained below. In this section, the reliability of this equation will be emphasized. The mean and standard deviation values of the relevant dimensionless parameters from the software program are presented in Figure 4.

Continue	mean	st. dev.	minimum	maximum
LECATA	,442539	,151296	,294118	,720155
FATA	,911582	,129783	,687987	1,000000
CATA	,088418	,129783	0,000000	,312010
PC	,145833	,242536	0,000000	,562500
SFC	,066722	,054868	0,000000	,111110
WC	,385336	,051930	,311110	,480000
SAC	,015856	,006662	0,000000	,025250
FTFTORT	1,020272	,302964	,518380	1,609850

Figure 4. The program outputs: Mean and standard deviation values

Various graphs were drawn and comments were made to demonstrate the safe availability of Equation (1). In this context, the relevant experimental " $f_t/f_{tort}$ " values were compared with the numerical " $f_t/f_{tort}$ " values calculated from Equation (1) (Figure 5). There was a minimum deviation of 1% and a maximum of 15% between experimental and numerical results. When all the results were examined, the approximate average deviation value was 6.5%. Thus, it can be said that the experimental results of previous studies and the numerical results of the present study are compatible with each other.



Figure 5. Variation of *ftftort* with experiment number for the current study and previous studies



In Figure 6, the accuracy of Equation (1) is examined by regression analysis. Correlation coefficient was determined as  $R^2$ =0.94. Experimental results were plotted on x axis and numerical results were plotted on y axis. The data seem to be very close to 45° of perfect line. This indicates that the numerical results are quite compatible with the experimental results. Furthermore, this indicates the availability of Equation (1).



Figure 6. Comparison of experimental and numerical fuftort values for the current study and previous studies

Similarly, with the graph shown in Figure 7, the usability of Equation (1) was emphasized. Because Figure 7 shows the differences between experimental results and numerical results. The differences between -0.11 and +0.14 are observed. In other words, the residual values are very close to the zero axis.



Figure 7. Comparison of numerical and residual  $f_t f_{tort}$  values for the current study and previous studies



In addition, the effect of related dimensionless parameters on tensile strength was investigated in this study. It is known that concrete strength decreases as water/cement ratio (w/c) ratio increases [28]. As shown in Figure 8, as the "w/c" increases, both experimental and numerical " $f_t/f_{tort}$ " values decrease. In fact, the correlation coefficient for changing the numerical " $f_t/f_{tort}$ " values with "w/c" was higher, R<sup>2</sup>=0.92 (Figure 8b). This shows that the numerical results are consistent in this respect.



Figure 8. Variation of  $f_t f_{tort}$  with w/c: a) for the previous experimantal studies b) for the current numerical study

Since lightweight expanded clay aggregate (LECA) is a lightweight aggregate, it is a type of aggregate that reduces the strength when using it in concrete. When the results of this study are examined, it is seen that " $f_t/f_{tort}$ " decreases as "*LECA/TA*" increases (Figure 9). The correlation coefficient was higher (R<sup>2</sup>=0.92) for the variation of numerical " $f_t/f_{tort}$ " values with "*LECA/TA*" (Figure 9b). In this respect, it can be said that the numerical results are compatible.



**Figure 9.** Variation of  $f_t/f_{tort}$  with *LECA/TA*: a) for the previous experimantal studies b) for the current numerical study

## 4. Conclusions

The conclusions of the current numerical study are summarized below:

- In this study, it is thought that the tensile strength results of concrete can be calculated easily and accurately with the developed equation by nonlinear statistical analysis.
- A numerical model has been developed which can be useful for literature and application engineers.



- It is expected to provide labor, time savings and economic gains with this study.
- The numerical results from this study were found to be consistent with the previous experimental results.

#### Acknowledgements

The author acknowledges the support of the Scientific and Technological Research Council of Turkey (TUBITAK) PhD Scholarship [grant number 2211-A].

#### References

- 1. ASTM C330-69, Standard Specification for lightweight aggregates for structures concrete.
- 2. TS 2511. Mix design of structural lightweight concrete. T.S.E, Ankara, 1977. (in Turkish).
- **3.** Çetmeli, E. New German Reinforced Concrete Specification (DIN 1045, 1972), Uluğ Bookstore, Istanbul, **1974**. (in Turkish).
- **4.** Şimşek, O. Concrete and technology of concrete. Seçkin Publishing A.Ş., Ankara, **2007**, (in Turkish).
- **5.** Seyhan, İ. Expansion clays, eighth five-year development plan, mining specialization commission report industrial raw materials subcommittee building materials III, **2001** (in Turkish).
- 6. Gökçe, M. Searching usage of the expanded clay aggregate in light concrete. Master thesis, Gazi University, Institute of Science and Technology, Ankara, 2007. (in Turkish).
- 7. Lo T. Y., Tang W. C., and Cui H. Z. The effects of aggregate properties on lightweight concrete" Building and Environment, 2007, 42, 3025–3029.
- 8. Gündüz, L., Şapcı, N., Bekar, M., and Yorgun, S. Utilization of expanded clay as lightweight aggregate. Journal of Clay Science and Technology, Kibited, 2006, 1(2), pp. 115-121 (in Turkish).
- **9.** Lo, T. Y., Cui, H. Z., Tang, W. C., and Leung, W. M. The effect of aggregate absorption on pore area at interfacial zone of lightweight concrete. Construction and Building Materials, **2008**, Vol. 22, pp. 623 628.
- **10.** Bartolini, R., Filippozzi, S., Princi, E., Schenone, C., and Vicini, S. Acoustic and mechanical properties of expanded clay granulates consolidated by epoxy resin. Applied Clay Science, **2010**, Vol. 48, pp. 460 465.
- **11.** Bogas, J.A. and Gomes, A., and Pereira, M. F. C. Self-compacting lightweight concrete produced with expanded clay aggregate. Construction and Building Materials, **2012**, Vol. 35, pp. 1013 1022.
- 12. Costa, H., Julio, E., and Lourenço, J. New approach for shrinkage prediction of high-strength lightweight aggregate concrete. Construction and Building Materials, 2012, Vol. 35, pp. 84 91.
- **13.** Uglyanitsa, A. V., Gilyazidinova, N. V., Zhikharev, A. A., and Kargin, A. A. Study of reinforcement corrosion in expanded clay concrete. HBRC Journal. **2014**.
- Yang, K. H., Kim, G. H., and Choi, Y. H. An initial trial mixture proportioning procedure for structural lightweight aggregate concrete. Construction and Building Materials, 2014, Vol. 55, pp. 431 – 439.

15. Tunç, E. T., Alyamaç, K. E., Ragıp, İNCE and Ulucan, Z. Ç. Investigation of mechanical properties of high-performance lightweight concrete with pumice aggregate. Engineering Sciences, 2018, 13(4), 344-353.

MET

- 16. Tunc, E. T., Saglam, R.N., Ulucan, M., Demir, T., Ulucan, Z.Ç. and Alyamac, K.E. A Preliminary Mix Design For Structural Lightweight Concrete Produced With LECA. International Civil Engineering and Architecture Conference (ICEARC 2019) file:///C:/Users/esra23/Desktop/ICEARC'19/Bas%C4%B1lanlar/ICEARC2019\_Part\_A.pd f (2214 – 2222). **2019**.
- 17. Saglam, R.N., Tunc, E. T., Demir, T., Ulucan, M. and Alyamac, K.E. Structural Lightweight Concrete Produced With Perlite Aggregate - A Preliminary Mix Design. International Civil Engineering and Architecture Conference (ICEARC 2019) file:///C:/Users/esra23/Desktop/ICEARC'19/Bas%C4%B1lanlar/ICEARC2019\_Part\_A.pd f (2223 – 2228). **2019**.
- 18. ACI 213R-03. Guide for structural lightweight-aggregate concrete. ACI Manual of Concrete Practice, Part 1: Materials and General Properties of Concrete. American Concrete Institute, Farmington Hills, Michigan, 2003.
- 19. Kok, S. C., and Min-Hong, Z. Water Permeability and Chloride Penetrability of High-Strength Lightweight Aggregate Concrete, Cement and Concrete Research, 2002, No 32, pp. 639-645.
- 20. Sari, D., and Paşamehmetoğlu, A.G. The Effects of Gradation and Admixture on the Pumice Lightweight Aggregate Concrete. Cement and Concrete Research, 2005, No. 35(5), 936-942.
- **21.** Kaldı, C. Structural lightweight concrete design and its utilization in multi-story buildings. Master thesis, Ege University, Institute of Science and Technology, İzmir, 2011 (in Turkish).
- 22. Ariöz, O., Kilinc, K., Karasu, B., Kaya, G., Arslan, G., Tuncan, M., Tuncan A., Korkut, M., and Kıvrak, S. A preliminary on the properties of lightweight expanded clay aggregate, Journal of the Australian Ceramics Society, 2008, 44(1), 23-30.
- 23. Chandra, S. and Berntsson, L. Lightweight Aggregate Concrete. Noves Publications, USA, 2002, 1-430.
- 24. Statsoft, I. N. C. Statistica. Data analysis software system. Version, 8, 2001.
- 25. Alduaij, J., Alshaleh, K., Haque, M. N., and Ellaithy, K. Lightweight concrete in hot coastal areas. Cement and Concrete Composites, 1999, 21(5-6), 453-458.
- 26. Karamloo, M., Mazloom, M., and Payganeh, G. Effects of maximum aggregate size on fracture behaviors of self-compacting lightweight concrete. Construction and Building Materials, 2016, 123, 508-515.
- 27. Sajedi, F., and Shafigh, P. High-strength lightweight concrete using leca, silica fume, and limestone. Arabian Journal for Science and Engineering, 2012, 37(7), 1885-1893.
- 28. Tunc, E. T. Recycling of marble waste: A review based on strength of concrete containing marble waste. Journal of environmental management, 2019, 231, 86-97.

## EFFECT OF LOADING RATE ON FLEXURAL RESPONSE OF AERATED CONCRETE COMPOSITE BEAMS

of Materials and Engineering Technology

## DERYA BAKBAK<sup>1</sup>, AHMET EMIN KURTOGLU<sup>2</sup>

<sup>1</sup>The Grand National Assembly of Turkey (TBMM), Çankaya, Ankara, TURKEY <sup>2</sup>Department of Civil Engineering, Istanbul Rumeli University, Silivri, Istanbul, TURKEY

## Abstract

Aerated concrete is a type of lightweight concrete in which air-voids are entrapped in the matrix with the use of an aerating agent. Aerated concrete offers advantages such as ease and economy in design. This paper examines the influence of loading rate on the flexural behavior of autoclaved aerated concrete beams (lintels) reinforced with steel bars. Universal testing machine with a maximum capacity of 500 kN was used to conduct tests. Three variations of loading rates were applied, namely 0.2 mm, 1 mm, and 5 mm per minute to determine the maximum bearing capacity, displacement at failure, and failure modes. Results confirm that increasing loading rate yields slightly higher bearing capacity, yet, lower ductility and, the failure is observed to be slightly more brittle for higher loading rates.

Keyword: Lightweight concrete, Autoclaved aerated concrete, Composite beam, Flexural response, Loading rate

## **1. Introduction**

Autoclaved aerated concrete (AAC) is produced by the process of mixing cement or lime mortar and expansion agent used for the purpose of entrapping of air voids in the matrix. AAC is used in construction industry since mid-1920s for both structural and non-structural practice. AAC has several advantages such as low density (70-80% air voids), lower design cost, high thermal resistance and acoustic insulation property. Also, AAC is considered to be environmentally friendly material as it reduces 70% and 40% energy per material volume as compared to normal concrete and bricks, respectively [1-3]. The materials generally used in the production of AAC are (i) siliceous material (silica flour or finely ground siliceous sand), (ii) binding material (Portland cement with quick lime), (iii) aluminum powder and (iv) entrapping agent [4].

Production of AAC elements with reinforcement can offer an alternative for low-rise precast construction. 60% of new building constructions in Europe are built with different types of AAC elements [5]. In the housing industry in China, reinforced AAC materials for exterior walls are preferred to other materials [5].

Reinforced aerated concrete elements are manufactured by embedding the cage-formed steel bars spot-welded to the U-shaped links. The cages are treated with protective coating. Earlier studies on the mechanical properties of RAAC members are very limited. Matsumura (1984) investigated the Shear behavior of reinforced autoclaved lightweight cellular concrete members [6]. Aroni and

Cividini (1989) studied the shear strength of reinforced slabs made of autoclaved aerated concrete without shear reinforcement [7] Alengaram et al. (2010) studied on foamed aerated concrete reinforced beams with palm kernel shell as aggregate [8]. Taghipour et al. (2018) studied the seismic behavior of reinforced AAC vertical load-bearing wall panels under constant axial load and two-way cyclic lateral displacement excursions. [5]. Kurtoglu and Bakbak (2019) proposed models to predict the shear strength of reinforced aerated concrete slabs via support vector machines and artificial neural networks [9-10]

terials and Engineering Technology

In this study, the focus was placed upon the flexural response of RACB members tested with three variations of loading rates. The effect of loading rate on the load-deflection response and failure modes was examined.

## 2. Experimental Study

Reinforced aerated concrete beams (RACBs) were produced by YTONG Gaziantep firm, Gaziantep, Turkey. All production processes are made in accordance with TS EN 12602 standard [11]. Each beam specimen has a dimension of 1150 mm length, 250 mm depth and 200 mm width (see Figure 1a). Longitudinal reinforcements is embedded in RACBs. Concrete cover was 30 mm applied at each side.



(c) Reinforced RACB Figure 1. Schematic design of RACBs (dimensions in cm)


Figure 2. Test setup

In order to investigate the effect of loading rate on flexural response of RACBs, a universal testing machine with a capacity of 500 kN was used in displacement controlled mode. Two point bending test was applied to determine the ultimate load and displacement. Linear variable displacement transducer (LVDT) was used to measure the mid-span deflection of the beams. Samples with identical properties were tested at three variations loading rate, namely 0.2 mm, 1 mm and 5 mm per minute.

#### 3. Results and Discussion

Ultimate bearing capacities of RACBs are measured as 13.33 kN, 12.01 kN and 14.13 kN for 0.2 mm, 1 mm. and 5 mm. loading rates, respectively. The mid-span deflections where the ultimate loads were achieved are 19.18 mm, 11.87 mm and 7.68 mm for 0.2 mm, 1 mm. and 5 mm per minute loading rates, respectively.





As far as the ascending portions of load-displacement curves (Fig. 3) are concern, all specimens show similar patterns until the first cracking occurs and at 0.2 mm/min. and 1 mm/min. loading rates, peak load is achieved rather slower as compared to 5 mm/min. loading rate. At 0.2 mm/min. loading rate, ultimate load is achieved at later stages (i.e., high mid-span deflection) and a comparatively ductile behavior is observed. For higher loading rates (1 mm and 5 mm per minute), however, maximum load is achieved earlier and a sharp decrease in load is observed.

Crack propagations of specimens show that smoothness of crack lines decreased as the loading rate increased. Thus, brittle type of failure is observed for higher loading rates.



(c) **Figure 4.** Failure modes

Acknowledgments: Authors would like to thank YTONG Gaziantep firm and Dr. Ali Khaled HUSSEIN for their support and sponsorship of the tests.

ME

#### **References:**

- 1. Thongtha, A., Maneewan, S., Punlek, C., & Ungkoon, Y, Investigation of the compressive strength, time lags and decrement factors of AAC-lightweight concrete containing sugar sediment waste. Energy and Buildings, 2014, 84, (516-525).
- 2. Qu, X., & Zhao, X. Previous and present investigations on the components, microstructure and main properties of autoclaved aerated concrete-A review. Construction and Building Materials, 2017, 135, (505-516).
- 3. Bonakdar, A., Babbitt, F., & Mobasher, B, Physical and mechanical characterization of fiber-reinforced aerated concrete (FRAC). Cement and Concrete Composites, 2013, 38, (82-91).
- 4. Desai, S, Reinforced autoclaved aerated concrete roof slabs. In Innovations and Developments in Concrete Materials and Construction: Proceedings of the International Conference held at the University of Dundee, Scotland, UK on 9-11 September 2002 (523-532). Thomas Telford Publishing.
- 5. Taghipour, A., Canbay, E., Binici, B., Aldemir, A., Uzgan, U., & Eryurtlu, Z., Seismic behavior of reinforced autoclaved aerated concrete wall panels. ce/papers, 2018, 2(4), (259-265).
- 6. Matsumura, A., Shear strength and behavior of reinforced autoclaved lightweight cellular concrete members. Trans. Architect. Inst. Jpn, 1984, 343, (13-23).
- 7. Aroni, S., & Cividini, B., Shear strength of reinforced aerated concrete slabs. Materials and Structures, 1989, 22(6), (443-449).
- 8. Alengaram, U. J., Mahmud, H., Jumaat, M. Z., & Shirazi, S. M. Effect of aggregate size and proportion on strength properties of palm kernel shell concrete. International Journal of the Physical Sciences, 2010, 5(12), (1848-1856).
- 9. Kurtoğlu, A. E., & Bakbak, D. (2019). Modeling the Shear Strength of Reinforced Aerated Concrete Slabs via Support Vector Regression, International Journal of Engineering Technologies, **2019**, 5(1), (6-14).
- 10. Kurtoğlu, A. E., & Bakbak, D. (2019). Shear Resistance of Reinforced Aerated Concrete Slabs: Prediction via Artificial Neural Networks, Journal of Sustainable Construction Materials and Technologies, 2019, In press.
- 11. TS EN 12602, Önyapımlı donatılı gazbeton yapı elemanları, Türk Standartları Enstitüsü, **2011**, (in Turkish).

# r-GO /TiO2 NANOKOMPOZITININ ÜRETIMI, KARAKTERİZASYONU VE SERTLİK ÖZELLİKLERİNİN İNCELENMESİ

of Materials and Engineering Technology

#### DERYA TEKİN<sup>1</sup>, DERYA BİRHAN<sup>2</sup>, TANER TEKİN<sup>3</sup>

<sup>1</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, Erzurum, TÜRKİYE.
<sup>2</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, Erzurum, TÜRKİYE
<sup>3</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Kimya Mühendisliği, Erzurum, TÜRKİYE.

#### Özet

Yarı iletken fotokatalizler, farklı katalitik reaksiyonlarda çok önemlidirler. TiO<sub>2</sub>, WO<sub>3</sub>, CdS, ZnS, GaN, ZnO, In<sub>2</sub>O<sub>3</sub> ve BiVO<sub>4</sub> gibi farklı tiplerde fotokatalizörler mevcut olmaktadır. Titanyum oksit (TiO<sub>2</sub>), maliyet etkinliği, düşük toksisite, yüksek stabilite, mükemmel biyouyumluluk, yüksek kimyasallık ve benzersiz fotokatalitik özellikler gösterdiği için birçok uygulama alanında kullanılan bir yarı iletken fotokatalizör olmaktadır. Son yıllarda grafen esaslı nanokompozitler iki bileşenin avantajlı özelliklerini bir araya getiren yüksek katma değere sahip değerli malzemeleri üretmek için bir araya getirilmiştir. Grafen bal peteği şeklinde 2D kafes yapısına sahip sp<sup>2</sup> hibritlesmesi yapan, karbon atomları arasındaki mesafe 0.142 nm olan bir karbon tabakasıdır. İki boyutlu olarak indirgenmiş GO'nun (rGO) kenar ve bazal düzlemleri içerisinde var olan fonksiyonel grupların içerdiği az miktardaki oksijen miktarının varlığı büyük önem taşımaktadır. TiO<sub>2</sub> nanopartiküllerinin rGO ile hibritleşmesi, yapıya birçok avantaj sağlamaktadır. Bunun nedeni ise rGO elektron deliği çiftlerinin rekombinasyonunu azaltmakta ve yük transfer oranını artırmaktadır. Bu bildiri kapsamında r-GO/TiO2 nanokompozit katalizörü sentezlenmiştir. Sentezlenmiş olan nanokompozitin yapısal özelliklerini incelemek için SEM-EDS ve XRD analizleri kullanılmıştır. Sertlik özelliklerinin incelenmesi için Vickers sertlik cihazı kullanılmıştır. r-GO/ TiO<sub>2</sub> nanokompozitinin sentezinde, ilk asamada destek malzemesi Hummer metodu ile sentezlenmiş ve ikinci aşamada da sol-jel yardımı ile r-GO/TiO2 nanokompoziti sentezlenmiştir. Elde edilen r-GO/TiO<sub>2</sub> nanokompozitinin SEM görüntülerinde, TiO<sub>2</sub> nanopartiküllerinin ortala çapının ~ 15 nm olduğu ve çeşitli şekil ve boyutlara sahip olduğu gözlemlenmiştir. rGO tabakalarının tamamen TiO<sub>2</sub> nanopartikülleri ile kaplandığı tespit edilmiştir. XRD sonuçlarında, TiO<sub>2</sub>'nin sahip olduğu anataz kristal yapısının kırınım pikleri görülmektedir.

Sertlik ölçümü sonrası, sol-jel yöntemi ile sentezlenmiş r-GO/TiO<sub>2</sub> nanokompozitinin ortalama sertlik değeri 74,34 HV olarak ölçülmüştür. Yapının sahip olduğu karbon – karbon (C=C) kovalent bağı en güçlü bağlardan olmaktadır ve yapı içerisine katıldığında oldukça iyi mekanik özellikler gösterdiği görülmektedir.

Anahtar kelimeler: İndirgenmiş grafen oksit (r-GO), TiO2 nanopartikülü, Hardness Vickers



#### 1. Giriş

İyi bilinen fotokatalitik bir malzeme olan Titanyum oksit (TiO<sub>2</sub>), maliyet etkinliği, düşük toksisitesi ve yüksek kimyasal kararlılığı nedeniyle birçok alanda büyük ilgi görmektedir [1]. TiO2, yüksek oksitleme kapasitesi, orta bant aralığı, fotokorozyon ve kimyasal aşınmaya karşı toksik olmayan, ucuz ve yüksek kimyasal kararlılığasahip olması gibi üstün özellikleri nedeniyle, araştırılmış bir yarı iletken fotokatalisttir [2]. Bununla birlikte, TiO<sub>2</sub>'nin pratik uygulamaları, bazı sahip olduğu dezavantajlar nedeniyle büyük ölçüde engellenmiştir. Foton üreten elektron – holl çiftlerinin hızlı bir şekilde rekombinasyon olması, görünür ışık bölgesini yeterli derecede kullanamaması, UV bölgesine yakın absorbsiyon göstermesi gibi dezavantajlara sahip olmaktadır [1, 2]. Bu problemleri çözebilmek için metal iyon katma, karbon veya azot katkılama, metal yükleme gibi birçok yöntem kullanılmasıdır. Bu karbon materyaller arasında en iyi olanı iki boyutlu grafenin kullanılmasıdır [3]. Son zamanlarda grafen, TiO<sub>2</sub>'nin mekanik, fotokatalitik, termal ve elektrik iletkenlik özelliklerini artırmak için yaygın olarak kullanılan bir destek malzemesidir [4].

The International Conference of Materials and Engineering Technology

Grafen, altıgen bir ağa bağlanan ve bu ağın karbon atomlarından oluştuğu tek tabakalı bir malzemedir. Grafen, karbon nanotüplerin (CNT) ve büyük fullerenlerin imalatında yapı taşı olarak kullanılmaktadır. Grafen, oda sıcaklığında kuantum Hall etkisinin gözlemlenmesi, yüksek mekanik mukavement, uygun ısıl iletkenliği, yüksek Young modülü, kırılma direnci gibi birçok üstün fiziksel özelliklere sahip olmaktadır. Grafen oksitin indirgenmesi birden fazla indirgeyici ajanlar kullanılmaktadır. Hidrazin hidrat, dimetil hidrazin, dimetil formaldehit gibi indirgeyici ajanlar termal yöntemler ve ultraviyole ışınlamaları kullanılarak indirgenmektedir [5]. Esas olarak, grafen grafen oksitin (GO) kimyasal olarak indirgenmesi redüklenmiş grafen oksitin (rGO) kenar ve bazal düzlemlerinde yer alan az miktarda oksijen içeren fonksiyonel gruplar büyük öneme sahip olmaktadır [6]. Redüklenmiş grafen oksit, karbon ailesinin yeni allotropu olarak; rGO'yu destekleyen nanopartiküller ile iyi bir temas sağlayabilmesi için mükemmel bir destek malzemesi, sp2 hibritleşmesi yapmaktadır.Karbon yapısının düzlemsel formunu oluşturma ve r-GO/TiO<sub>2</sub> nanokompozitlerinde elektron – holl çiftlerinin rekombinasyonunun etkili bir şekilde engelleme gibi yapı içerisinde birçok benzersiz işleve sahip olmaktadır [7].

Sıkıştırma, bükülme mukavemeti ve sertliği gibi birçok mekanik özelliği geliştirmek adına reçinelere dolgu maddeleri ilave edilmektedir. Geliştirilmiş fiziksel özellikler arasında, polimerizasyon büzülmesinde bir azalma ve elastiklik modülünde bir artış ortaya çıkmaktadır. Kompozit yapıda ki reçinelerin fiziksel ve mekanik özellikleri, yapı içerisine katkılanan dolgu maddelerinin özelliklerinden etkilenmektedir. Bir grafen katmanındaki karbon – karbon (C=C) kimyasal bağı muhtemelen doğada bilinen genişletilmiş bir sistemdeki en güçlü kimyasal bağdır. Grafen allotropları sorunsuz bir şekilde toplanmış grafen katmanları olduğu için, keşfedildiği günden itibaren bu nanoyapıların mükemmel mekanik özelliklere sahip olduğu ve bu özelliklerin miktarının ölçülmesi nanoteknoloji alanında büyük ilgi görmektedir. Nanokompozit malzemelerin sertliği, kalıcı girinti veya penetrasyona direnç olarak tanımlanır. Sertlik test ölçüm yöntemlerinde, belirli bir yük altında hareketli prop yüzeye batırılarak ve bir girinti oluşturularak ölçüm yapılır.

Bu çalışmada,TiO<sub>2</sub> nanopartikülleri ve r-GO/TiO<sub>2</sub> nanokompozit yapıları sentezlenip SEM,XRD kullanılarak karakterize edilmiştir. Nanokompozitlerin sertlikleri Vicker sertlik cihazı kullanılarak belirlenmiştir.



### 2. Materyal Ve Yöntem

#### 2.1. Materyaller

Grafit tozları (powder<45 $\mu$ m $\geq$ 99.99%, Sigma Aldrich), Sülfürik asit (H<sub>2</sub>SO<sub>4</sub>,95-97%, Merck), Sodyum Nitrat (NaNO<sub>3</sub>), Potasyum Permanganat (KMNO<sub>4</sub>), DMF(N-N-Dimethylformamide, $\geq$ 99%, Sigma Aldrich) ve Hidrojen peroksit (H<sub>2</sub>O<sub>2</sub>, %30, Sigma Aldrich), Titanyum (IV) izopropoksit (Sigma Aldrich, %97), Etanol (Sigma Aldrich, %99,8) temin edilmiştir.

The International Conference of Materials and Engineering Technology

#### 2.2. Yöntem

#### 2.2.1. TiO<sub>2</sub> nanopartiküllerinin sentezi

TiO<sub>2</sub> nanopartiküllerini sentezlemek için ilk önce 20 mL Titanyum (IV) izopropoksit 140 mL etanol içerisinde 15 dakika için manyetik olarak karıştırıldı. Karıştırma işlemi tamamlandıktan sonra solüsyon 80°C'de 1 saat süre ile reflux işlemine tabi tutuldu. Reflux işleminden sonra Titanyum solüsyonu süzülüp 60°C'de kurutuldu. Kurutma işleminin ardından 500°C'de 3 saat süre ile kalsinasyon işlemi gerçekleştirildi. Saf Titanyum nanoparçacıkları elde edilmiştir.

#### 2.2.2. Grafen Oksitin Sentezi

Grafen oksit grafit tozu Hummer Metoduna göre sentezlenmektedir. İlk önce 69 ml sülfirik asit, 3 g grafit tozu, 1.5 g NaNO<sub>3</sub> bir 250ml erlen içerisinde karıştırıldı. Daha sonra çözelti sıcaklığının 0°C'ye inmesi için bir buz banyosu içerisine yerleştirilmiştir. Sıcaklık 0°C düştükten sonra 9 g potasyum permanganat çözelti içerisine ilave edildi. Potasyum permanganatın ilavesi bittikten sonra çözelti buz banyosu içerisinden çıkarılarak sıcaklığının oda sıcaklığına çıkması için beklemeye alındı. Daha sonra 138 ml iyonize su ilavesi yapıldı ve 98°C'de 15 dakika karıştırıldı ve 480 ml su ilavesi yapıldıktan sonra % 30'luk H<sub>2</sub>O<sub>2</sub> damlatılarak mevcut kahverengi rengin sarıya dönüşmesi sağlanarak oksitleme işlemi tamamlandı. Elde edilen grafen oksit birkaç kez etanol ve iyonize su ile yıkanarak pH dengesi sağlandı. Daha sonra süzüldükten sonra 60°C'lik fırında tamamen kuruması gerçekleşene kadar bekletilmektedir.

#### 2.2.3. rGO Sentezi

0.8 g GO tozları 400 ml DMF içerisinde ultrasonikasyonda çözündürülerek 6 saat reflux işlemi gerçekleştirildi. 6 saatlik işlemin sonunda rGO tozları süzülerek etanol ve su ile yıkanarak 60°C'lik fırında kurutuldu.

#### 2.2.4. rGO-TiO2 Nanokompozit Yapısının Sentezi

0.1 g rGO tozu 10 ml etanol ve 20 ml su ile karıştırılarak 1 saat sonikasyon işlemine tabi tutuldu. 1 saatlik işlemin sonunda 0.2 g anataz formundaki TiO<sub>2</sub> nanotozları rGO solüsyonunun içerisine ilave edildi ve 1 saatlik karışma süresinin sonunda solüsyon petri kaplarına dökülerek 60°C'lik fırında kurutuldu. Ve rGO-TiO<sub>2</sub> nanotozları elde edildi.





# 3. Sonuçlar Ve Tartışma

3.1. SEM ve XRD Analizi



Şekil 1. TiO2 nanopartikülünün (a)SEM görüntüsü, (b)EDS analizi

Şekil 1'de TiO<sub>2</sub> nanopartiküllerinin 100 nm'deki SEM görüntüsü gösterilmektedir. Buna göre, TiO<sub>2</sub> nanopartiküllerinin aglomera olduğunu gözlemliyoruz. TiO<sub>2</sub> nanopartiküllerinin küresel şeklini ve gözenekli yapısını SEM görüntüleri açıkça ortaya koymaktadır [8].EDS spekturumları Oksijen ve Titanyumun varlığını doğrulamaktadır.



Şekil 2. r-GO/TiO<sub>2</sub> nanokompozitinin (c)SEM görüntüsü, (d)EDS analizi

Şekil 2'de görüldüğü gibi, rGO tabakaları tamamen TiO<sub>2</sub> nanopartikülleri ile kaplanmıştır. Bu iki malzeme arasında mükemmel bir elektrik iletkenliği istiyorsak rGO levhaları ve TiO<sub>2</sub> nanopartikülleri arasındaki bu güçlü etkileşim kurmak oldukça önemlidir [9]. EDS spektrumları C,O ve Ti varlığını doğrulamaktadır.



Şekil 3. TiO<sub>2</sub> nanopartikülü ve r-GO/TiO<sub>2</sub> ait XRD diyagramları

Şekil 3'de görüldüğü gibi, Sentezlenen saf TiO<sub>2</sub> nanokompozitinin XRD sonuçlarına göre, TiO<sub>2</sub> nanopartikülü anataz formu için JCPDS (JCPDS No. 21-1272) kartları ile uygun yoğun kırınım pikleri göstermektedir. TiO<sub>2</sub> nanopartiküleri, 3 saat boyunca 500 ° C'de tavlama sonrasında polikristalin anataz fazına dönüşmektedir [10]. r-GO/TiO<sub>2</sub> nanokompozitinin kırınım pikleri, anataz formundaki TiO2'nin kırınım pikleri ile benzerlikler göstermektedir [11].

#### 3.2. Sentezlenen Nanokompozitlerin Sertlik Ölçümü

Sentezlenen TiO<sub>2</sub> nanopartikülleri ve rGO-TiO<sub>2</sub> nanokompozit yapısının mikrosertlik analizleri 245 mN (0,025) yük altında 10 saniye tutularak Hardness Vickers değerinde 5 sertlik değerinin ortalaması alınarak yapılmıştır. Şekil 4'de numunelerin sertlik değerleri grafik halinde gösterilmektedir.



Şekil 4. Nanokompozitlerin sertlik grafiği



 $TiO_2$  nanopartikülüne ait sertlik değerlerinin ortalaması 53,96 HV olarak ölçülmüştür. 500°C'de kalsine edilen  $TiO_2$  nanopartikülleri iyi mekanik özellikler ile sonuçlanmaktadır. Literatürde yapılan çalışmalarda, sıcaklığın artırılması ile sertliğin daha da arttığı tespit edilmiştir [12].

<b>Tablo 2</b> . r-GO/TiO <sub>2</sub> nanokompozitinin sertlik değerleri									
1 2		3	4	5					
79 (HV)	70 (HV)	79,2 (HV)	71,2 (HV)	72,3 (HV)					

r-GO /TiO<sub>2</sub> nanokompozitinin ortalama sertlik değeri 74,34 HV olarak ölçülmüştür. Bir karbon allotropu olan grafen, grafit ve pırlantaya yakın mekanik özellikler göstermesi beklenmektedir. Yapının sahip olduğu karbon – karbon (C=C) kovalent bağı en güçlü bağlardan olmaktadır ve yapı içerisine katıldığında oldukça iyi mekanik özellikler gösterdiği görülmektedir [13].

#### 4. Sonuçlar

Sentezlenen TiO<sub>2</sub> nanopartikülü ve r-GO/TiO<sub>2</sub> nanokompozit yapılarının sertlik ölçümünde TiO<sub>2</sub> nanopartillerinin içerisine destek maddesinin ilave edilmesi ile yapının önemli ölçüde mekanik olarak iyileştiği tespit edilmiştir.

#### Referanslar

- 1. Zhao, W., et al., Synthesis of Ag/TiO2/graphene and its photocatalytic properties under visible light. Materials Letters, 2016. **171**: p. 182-186.
- 2. Maruthamani, D., D. Divakar, and M. Kumaravel, Enhanced photocatalytic activity of TiO2 by reduced graphene oxide in mineralization of Rhodamine B dye. Journal of Industrial and Engineering Chemistry, 2015. **30**: p. 33-43.
- 3. Galińska, A. and J. Walendziewski, Photocatalytic water splitting over Pt– TiO2 in the presence of sacrificial reagents. Energy & Fuels, 2005. **19**(3): p. 1143-1147.
- 4. Luo, L., et al., Hydrothermal synthesis of fluorinated anatase TiO2/reduced graphene oxide nanocomposites and their photocatalytic degradation of bisphenol A. Applied Surface Science, 2015. **353**: p. 469-479.
- 5. Didehban, K., L. Mohammadi, and J. Azimvand, Preparation of RGO/Fe3O4/poly (acrylic acid) hydrogel nanocomposites with improved magnetic, thermal and electrochemical properties. Materials Chemistry and Physics, 2017. **195**: p. 162-169.
- Sohail, M., et al., Synthesis of well-dispersed TiO2@ reduced graphene oxide (rGO) nanocomposites and their photocatalytic properties. Materials Research Bulletin, 2017. 90: p. 125-130.



UWE

- 8. Singh, R., S.G. Kulkarni, and S.S. Channe, Thermal and mechanical properties of nanotitanium dioxide-doped polyvinyl alcohol. Polymer bulletin, 2013. **70**(4): p. 1251-1264.
- 9. Morais, A., et al., Nanocrystalline anatase TiO 2/reduced graphene oxide composite films as photoanodes for photoelectrochemical water splitting studies: the role of reduced graphene oxide. Physical Chemistry Chemical Physics, 2016. **18**(4): p. 2608-2616.
- 10. Vishwas, M., et al., Effect of tio2 nano-particles on optical, electrical and mechanical properties of poly (vinyl alcohol) films. Procedia Materials Science, 2014. **5**: p. 847-854.
- 11. Yang, Y., et al., One-step hydrothermal synthesis of surface fluorinated TiO2/reduced graphene oxide nanocomposites for photocatalytic degradation of estrogens. Materials Science in Semiconductor Processing, 2015. **40**: p. 183-193.
- 12. Oktar, F., Hydroxyapatite–TiO2 composites. Materials Letters, 2006. **60**(17-18): p. 2207-2210.
- 13. Lau, K.-T., et al., Micro-hardness and flexural properties of randomly-oriented carbon nanotube composites. Journal of composite materials, 2003. **37**(4): p. 365-376.

# PVA – TİO<sub>2</sub> NANOKOMPOZİTİNİN ÜRETİMİ, KARAKTERİZASYONU VE ANTİBAKTERİYEL ÖZELLİKLERİNİN İNCELEMESİ

of Materials and Engineering Technology

#### DERYA TEKİN<sup>1</sup>, DERYA BİRHAN<sup>2</sup>, TANER TEKİN<sup>3</sup>

<sup>1</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, Erzurum, TÜRKİYE.
<sup>2</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, Erzurum, TÜRKİYE.
<sup>3</sup> Atatürk Üniversitesi, Mühendislik Fakültesi, Kimya Mühendisliği, Erzurum, TÜRKİYE.

#### Özet

Son yıllarda, organik – inorganik nanokompozitler piller, sensörler, yakıt hücreleri, süperkapasitörler, güneş pilleri, biyosensörler antibakteriyel özelliklerin incelenmesi gibi farklı uygulamalardaki potansiyel kullanımları nedeniyle polimer - inorganik malzemelere olan ilgi artmaktadır. Bu polimerler arasında, Polivinilalkol (PVA), mükemmel film oluşturucu, emülsifiye edici ve yapışkan özelliklere sahip ticari olarak suda çözünebilen bir polimer türüdür. Bu bildiri kapsamında, TiO<sub>2</sub> nanopartikülleri ve PVA – TiO<sub>2</sub> nanokompoziti cast metodu kullanılarak sentezlenmiştir. Sentezlenen nanokompozitlerin morfolojik ve elementel analizi SEM-EDS, kristal yapıları ise XRD kullanılarak incelenmiştir. Titanyum (IV) izopropoksitten yola çıkarak TiO<sub>2</sub> nanopartikülleri sentezlenmiş, ikinci aşamada ise, TiO<sub>2</sub> nanopartikülleri PVA ile modifiye edilmiştir. PVA – TiO<sub>2</sub> nanokompozitinin SEM sonuçlarına göre, filmlerinin yapılarında kümelerin ortaya çıktığı görülmektedir. EDS sonuçları oksijen ve titanyumun varlığını kanıtlamaktadır. XRD sonuçları, TiO<sub>2</sub> nin anataz fazını doğrulamakta ve TiO<sub>2</sub> nanopartiküllerinin polimer ile modifiye edilmesi TiO<sub>2</sub> piklerini etkilememektedir.

Sentezlenen TiO<sub>2</sub> nanopartikülleri ve PVA – TiO<sub>2</sub> nanokompozitinin antibakteriyel aktiviteleri *Escherichia coli* (ATCC 25922) bakteri türü kullanılarak Time Kill Curve (zamana bağlı yok etme) yöntemi kullanılarak gerçekleştirilmiştir. Antibakteriyel aktivite deneme sonuçlarına göre, TiO<sub>2</sub> nanopartiküllerinin %14,42, PVA – TiO<sub>2</sub> nanokompozitinin ise % 19,23 oranında bakteri giderdiği tespit edilmiştir.

Anahtar Kelimeler: TiO<sub>2</sub> nanopartikülleri, PVA – TiO<sub>2</sub> nanokompoziti, Antibakteriyel aktivite

#### 1.Giriş

Nanomalzemelerin benzersiz özelliklerini mikro biyolojide uygulama çabalarıyla yapılan materyal araştırması, gelecekteki antimikrobiyal teknolojilerini genişletme konusunda büyük bir potansiyele sahiptir. Antimikrobiyal ajanlar, çevre, gıda, sentetik tekstiller, ambalajlama, sağlık ve tıbbi bakım ürünlerindeki bir dizi endüstriyel uygulama için oldukça önemli olmaktadır [1]. Nanoteknoloji, gıda ürünleri için ambalajlama ve depolama uygulamalarında kullanılan fotokatalitik nanopartiküller ve polimer nanokompozit filmler formunda gelişmiş antimikrobiyal kaplama elde etmeye yardımcı olabilir [2]. Polimer esaslı nanokompozitler, son yıllarda yüksek performanslı hafif malzemeler elde etmek amacıyla önemli ölçüde dikkat çekmektedir [3]. Polivinilalkol (PVA), mükemmel film oluşturma, emülsifiye edici ve yapışkan özelliklere sahip önemli, suda çözünebilen bir polimer türüdür. Polivinil alkol (PVA), kokusuz, toksik olmayan, biyouyumlu özelliklere sahip olup doku mühendisliği, ilaç dağıtımı, eklem kıkırdak ve biyosensörler gibi biyoteknolojik

uygulamalarda yaygın olarak kullanılmaktadır [4]. PVA, iyi film morfolojisi nedeniyle mükemmel bir matris malzemesidir. Bununla birlikte, PVA, neme karşı oldukça hassas olmaktadır, buda polimerin dayanıklılığını ve dengesini azaltmaktadır [5]. Bu probleme çözüm olarak, döküm yöntemi kullanılarak TiO<sub>2</sub> nanopartikülleri ile birlikte, PVA matriksli nanokompozit malzemeler sentezlenmiştir.

Titanyum dioksit (TiO<sub>2</sub>), kimyasal kararlılık, mükemmel optik geçirgenlik, iyi elektriksel özelliklere sahip olduğundan nanokompozitlerin geliştirilebilmesi için en önemli dolgu maddesi olmaktadır [6]. TiO<sub>2</sub> boyaya duyarlı güneş hücreleri, fotokatalizörler, su arıtma ve antimikrobiyal çalışmalarda yaygın olarak kullanılmaktadır [7]. TiO<sub>2</sub> tozları sol – jel tekniği kullanılarak ince film üretiminde kullanılmaktadır.

Bu çalışmada, TiO<sub>2</sub> nanopartikülleri sentezlenip PVA matriks üzerine dopp edilmiştir. Sentezlenen TiO<sub>2</sub> nanopartikülleri ve PVA- TiO<sub>2</sub> nanokompoziti SEM, XRD teknikleri kullanılarak karakterize edilmiştir. Antibakteriyel aktiviteleri için ise, *E.coli* bakterisi kullanılmıştır.

#### 2. Materyal Ve Yöntem

#### 2.1. Materyaller

Titanyum (IV) izopropoksit (Sigma Aldrich, %97), etanol (Sigma Aldrich, %99,8), Polivinilalkol (Mw 9,000-10,000,80% hydrolyzed,PVA, Sigma Aldrich) ve distile su kullanılmıştır.

#### 2.2. Yöntem

#### 2.2.1. TiO2 nanopartiküllerinin sentezi

TiO<sub>2</sub> nanopartiküllerini sentezlemek için ilk önce 20 mL Titanyum (IV) izopropoksit 140 mL etanol içerisinde 15 dakika için manyetik olarak karıştırıldı. Karıştırma işlemi tamamlandıktan sonra solüsyon 80°C'de 1 saat süre ile reflux işlemine tabi tutuldu. Reflux işleminden sonra Titanyum solüsyonu süzülüp 60°C'de kurutuldu. Kurutma işleminin ardından 500°C'de 3 saat süre ile kalsinasyon işlemi gerçekleştirildi. Saf Titanyum nanoparçacıkları elde edilmiştir.

#### 2.2.2. PVA – TİO2 nanokompozitinin sentezi

1 g polivinil alkol (PVA) tozları 25 mL iyonize su içerisinde 70°C'de 1 saat süre ile karıştırıldı. Diğer tarafta 25 mL iyonize su içerisinde sentezlenen saf TiO<sub>2</sub> nanopartikülleri 30 dakika boyunca ultrasonikasyon işlemine tabi tutuldu. Sonikasyon işleminin ardından PVA solüsyonu titanyum solüsyonunun içerisine damla damla ilave edildi ve 3 saat boyunca karıştırıldı. Karışma işlemi tamamlandıktan sonra solüsyon cam petri kaplarında 40°C'de 2 gün süre ile kurutulmuştur.

#### 2.2.3. Antibakteriyel Test

Sentezlenen TiO<sub>2</sub> nanopartikülleri ve PVA-TiO<sub>2</sub> nanokompozitinin antibakteriyel aktiviteleri *Escherichia coli* (ATCC 25922) bakteri türü kullanılarak Time Kill Curve (zamana bağlı bakteri sayısında azalma) yöntemi kullanılarak gerçekleştirilmiştir. Sentezlenen nanokompozitlerin optik yoğunluğu 600 nm'lik dalga boyu (OD600) ile absorbans değerleri okunarak nanokompozitlerin bakteri gideriminde ne kadar etkili olduğu incelenmiştir.





3. Sonuçlar ve Tartışma

## 3.1. SEM ve XRD Analizi



Şekil 1. TiO<sub>2</sub> nanopartikülleri (a) SEM görüntüsü, (b) EDS spektrumu

Şekil 1'de TiO<sub>2</sub> nanopartiküllerinin 100 nm'deki SEM görüntüsü gösterilmektedir. Buna göre, TiO<sub>2</sub> nanopartiküllerinin aglomera olduğunu gözlemliyoruz. TiO<sub>2</sub> nanopartiküllerinin küresel şeklini ve gözenekli yapısını SEM görüntüleri açıkça ortaya koymaktadır [8].EDS spekturumları Oksijen ve Titanyumun varlığını doğrulamaktadır.



Şekil 2. PVA-TiO2 nanokompoziti (c) SEM görüntüsü, (d) EDS spektrumu

Şekil 2'de PVA-TiO<sub>2</sub> nanokompozitine ait 1  $\mu$ m'deki SEM görüntüsü görülmektedir. Buna göre, PVA-TiO<sub>2</sub> filmlerinin yapılarında kümelerin ortaya çıktığı görülmektedir. Kompozit filmler için elde edilen morfoloji, literatürdede bildirildiği gibi tozla doldurulmuş polimer kompozit malzemelere benzer özellikler göstermektedir [9]. EDS spekturumları Oksijen ve Titanyumun varlığını doğrulamaktadır.



Şekil 3. TiO2 nanopartikülü ve PVA-TiO2 nanokompozitine ait XRD diyagramı

Şekil 3'de sentezlenen saf TiO<sub>2</sub> nanopartiküllerinin XRD sonuçlarında TiO<sub>2</sub> nanopartikülünün anataz formu için JCPDS (JCPDS No. 21-1272) kartları ile uygun yoğun kırınım pikleri göstermektedir. TiO<sub>2</sub> nanopartiküleri, 3 saat boyunca 500°C'de tavlama sonrasında polikristalin anataz fazına dönüşmektedir. Sentezlenen PVA-TiO<sub>2</sub> nanokompozitinin XRD sonuçlarında PVA – TiO<sub>2</sub> nanokompozitinde, bir anataz TiO<sub>2</sub> fazı sergilenmektedir ve JCPDS (No. 21-1272) kartı ile de uyumluluk gösterdiği görülmektedir. PVA -TiO<sub>2</sub> nanopartiküllerinin kristalleşme sağladığı ve sadece anataz fazının gözlemlenebildiğini görülmektedir.

#### 3.2. Antibakteriyel Aktivite

Hazırlanmış olan numunelerin başlangıç ve 24 saat sonundaki absorbans ve *E. Coli* değerleri Tablo 1'de verilmiştir.

Maddeler	Başla	ngıç	24 saat sonunda			
	Absorbans	CFU/mL	Absorbans	CFU/mL		
Kör Numune	0.125	$1,0*10^8$	0,936	9,36*10 <sup>7</sup>		
TiO <sub>2</sub>	0.125	1,0*10 <sup>8</sup>	0,801	8,01*10 <sup>7</sup>		
PVA/TiO <sub>2</sub>	0.125	1,0*108	0,756	7,56*107		

Tablo 1. Nanokompozitlerin eklemiş olduğu ve referans E. Coli bakterilerinin absorbans ve CFU değerleri



Şekil 4. Nanokompozitlerin 24 saat sonunda bakteri konsantrasyonunda meydana getirdiği azalma grafiği

TiO<sub>2</sub> nanopartiküllerinin kullanılmış olduğu numunelerdeki bakteri sayısında %14,42 oranında azalma meydana gelmektedir. Anataz formundaki TiO<sub>2</sub> nanokompozitlerin bakteriyel aktiviteleri, *E.coli*'nin bakteriyel büyümesinin inhibisyonu ile değerlendirildi. Bu sonuçlar, TiO<sub>2</sub> nanopartiküllerinin güneş ışığının etkisi ile daha iyi antimikrobiyal aktivite sergilediğini göstermektedir. PVA – TiO<sub>2</sub> nanokompozitinin kullanılmış olduğu numunelerdeki bakteri sayısında %19,23 oranında azalma meydana gelmektedir. Titanyum oksit nanoparçacıklarının diğer fotokatalizörlerden daha küçük bir tanecik boyutuna sahip olmasına rağmen, geniş bant aralığından dolayı daha aktif hale getirmek için UV ışığı ile uyarılması gerekmektedir.

#### 4. Sonuç

Yapılan çalışma sonucunda PVA matrisi üzerine dopp edilen TiO<sub>2</sub> nanopartikülleri bakteri konsantrasyonunda % 19,23 oranında bir giderim göstermiştir. TiO<sub>2</sub> nanopartikülleri modifiye edilerek antimikrobiyal özelliklerinin iyileştirildiği tespit edilmiştir.

### Referanslar

- Rajeshkumar, S. and C. Malarkodi, In vitro antibacterial activity and mechanism of silver nanoparticles against foodborne pathogens. Bioinorganic chemistry and applications, 2014. 2014.
- 2. Dhanasekar, M., et al., Ambient light antimicrobial activity of reduced graphene oxide supported metal doped TiO2 nanoparticles and their PVA based polymer nanocomposite films. Materials Research Bulletin, 2018. **97**: p. 238-243.
- 3. Zhou, T., et al., The preparation of high performance and conductive poly (vinyl alcohol)/graphene nanocomposite via reducing graphite oxide with sodium hydrosulfite. Composites Science and Technology, 2011. **71**(9): p. 1266-1270.
- Zhu, Y., et al., Nanoindentation and thermal study of polyvinylalcohol/graphene oxide nanocomposite film through organic/inorganic assembly. Applied Surface Science, 2015. 349: p. 27-34.



- 5. Hdidar, M., et al., Influence of TiO2 rutile doping on the thermal and dielectric properties of nanocomposite films based PVA. Journal of Alloys and Compounds, 2018. **750**: p. 375-383.
- 6. Rathod, S.G, et al., Pressure sensitive dielectric properties of TiO 2 doped PVA/CN-Li nanocomposite. Journal of Polymer Research, 2015. **22**(2): p. 6.
- 7. Ahmadpoor, P., A.S. Nateri, and V. Motaghitalab, The optical properties of PVA/TiO2 composite nanofibers. Journal of applied polymer science, 2013. **130**(1): p. 78-85.
- 8. Singh, R., S.G. Kulkarni, and S.S. Channe, Thermal and mechanical properties of nanotitanium dioxide-doped polyvinyl alcohol. Polymer bulletin, 2013. **70**(4): p. 1251-1264.
- 9. Li, S., et al., Facile green synthesis of Degraded-PVA coated TiO2 nanoparticles with enhanced photocatalytic activity under visible light. Journal of Physics and Chemistry of Solids, 2019. **129**: p. 92-98.



laterials and Engineering Technology

#### ERKUT YALÇIN <sup>1</sup>, AHMET MÜNİR ÖZDEMİR <sup>2</sup>, MEHMET YILMAZ<sup>3</sup>, BAHA VURAL KÖK <sup>4</sup>

<sup>1</sup> Firat University, Engineering Faculty, Department of Civil Engineering, Elazig, TURKEY.

<sup>2</sup> Firat University, Engineering Faculty, Department of Civil Engineering, Elazig, TURKEY.

<sup>3</sup> Firat University, Engineering Faculty, Department of Civil Engineering, Elazig, TURKEY.

<sup>4</sup> Firat University, Engineering Faculty, Department of Civil Engineering, Elazig, TURKEY.

#### Abstract

The concept of asphalt self-healing has become popular among researchers in recent years. Correct and timely maintenance and repair of asphalt pavements prolong service life. Performing and planning these processes may be interrupted due to economic and environmental reasons. Therefore, some methods are being studied to accelerate the self-healing of asphalt. In this study, some of these methods are discussed and a general literature review is presented. One of the most promising methods to accelerate the self-healing of asphalt is the "Microcapsule Method". In the microcapsule method, the rejuvenators applied on the asphalt surface are encapsulated and added to the asphalt mixture. Then, when the cracks come into contact with the capsules, the capsules explode and the rejuvenators release into the crack and heal the cracks. In many types of research, it was found that the healing rate of asphalt mixtures that containing microcapsule, increased and service life was prolonged.

Keywords: Asphalt, Self-Healing, Microcapsule, Bitumen.

#### **1. Introduction**

Bitumen, which obtained by refining petroleum, is a viscoelastic material that commonly used in road and airport pavements. The flexibility of the asphalt mixture is high in the first period of its service life. After service life, these properties are weakened due to repeated traffic loads and changing environmental conditions (especially temperature and moisture) and the bituminous binder becomes fragile (1). This leads to different types of deterioration in the flexible pavement, particularly in the formation of cracks. Fatigue and low-temperature cracks are two of the different types of deterioration in bituminous mixtures.

Fatigue cracks are often caused by repeated heavy wheel loads. Fatigue cracks are parallel to the axis of the pavement, starting in the form of fine cracks ranging in length from a few decimeters to tens of meters, and turning into crocodile cracks. Fatigue cracks are caused by exceeding the fatigue strength of the pavement as a result of a certain repetition of heavy traffic loads (2-4). Moreover, inadequate compaction and/or inadequate drainage of the base, sub-base and/or foundation layers, poor construction techniques, environmental and climatic conditions (freezing effect, humidity

changes, etc.) leads to fatigue cracks. Low, medium and high-intensity cracks have shown in Figure 1.1.

of Materials and Engineering Technology



Fig. 1 Fatigue cracks of low (a), medium (b) and high (c) intensity (5).

Thermal (low temperature) cracks take place in the transverse direction which occurs perpendicular to the flow of traffic caused by increased shrinkage stresses caused by shrinkage of the asphalt pavement in cold climatic weather rather than the effects of traffic. Cracks that occur due to the repetition of temperature changes in pavements are called thermal fatigue cracks, while cracks that occur as a result of sudden cooling in pavements are called low-temperature cracks (6). Low-temperature cracks generally occur in the transverse plane at intervals of 6-9 meters, since the transverse shrinkage of the pavement is easier than its longitudinal shrinkage.

The most important reason for these cracks is the stiffness of the asphalt and its sensitivity to temperature. Asphalt, which exhibits a rigid behavior at low temperatures, can show fracture under tensile stress without deformation. Therefore, using soft and less sensitive to temperature asphalt in cold areas can reduce such cracks. Also, the amount of bitumen used in the mixture, aging of the bitumen, the rigidity of the mixture, air temperature, the number of cold days, the thickness of the pavement and the friction forces between the layers should be considered (7,8).

Fault in planning or disruptions due to economic reasons leads to a decrease in the service capability of the pavement and high-cost reconstructions. To overcome these problems, a new approach and new alternative maintenance methods have been investigated. One of these new approaches is self-healing asphalt.

#### 2.Self-Healing of Asphalt

Recently, scientists have turned to alternative maintenance methods because of economic and environmental reasons. One of these alternative methods is the self-healing technique of bituminous materials. This method reduces the maintenance cost of asphalt pavements and increases the service life of the pavements. To better understand the self-healing method of asphalt pavements, it is important to think and investigate all the factors that contribute to this method.

When bituminous materials are subjected to load repetition, their stiffness and strength will decrease. The process of the formation of micro-cracks during cyclic loading and then the transformation into macro-cracks has been investigated by many researchers (9-11). The recovery of the stiffness and strength of the material and the prolongation of fatigue life were determined for the first time by the rest period fatigue test in the 1960s.

Self-healing materials and pavements are expected to remain durable and safe throughout their service life. Prolonged use of asphalt pavements leads to fatigue cracks. As fatigue cracks increase, the likelihood of pavements to deteriorate increases(12).

Bituminous materials have self-healing capability for the repair of microcrack damage. They can recover the damages and significantly reduce maintenance costs and extend service life (13).

Recently, three techniques have been developed to improve the rate of asphalt pavements recovery. These are the induction method, microwave method, and microcapsule method.



#### **2.1.Induction Method**

The viscoelastic and thermoplastic nature of asphalt mixtures gives the material the ability to self-repair cracks. One of the most promising and effective methods for accelerating the recovery event is the addition of externally heatable conductive particles to the mixture by applying electromagnetic induction.

The International Conference

aterials and Engineering Technology

The process of generating electric current by placing the conductor in an alternative magnetic field is called Faraday electromagnetic induction. This method is often called induction heating. Recently, induction heating techniques have improved the temperature of asphalt pavements and heal cracks at high temperatures. (14).

This process increases the level of self-healing and closes the cracks. Figure 2.1 illustrates how cracks are closed by conductive fiber use and induction heating.



Fig. 2 Induction heating mechanism (15)

#### 2.2. Microwave Method

The microwave method is another method used to heal cracks caused by the deterioration of bituminous hot mixtures. In this method, as in the induction method, healing is achieved by increasing the surface and internal temperature of the asphalt mixture.

The potential of using steel slag in asphalt mixture by microwave heating as a self-healing material has been studied by Yihan et al. In this study, microwave heating rate and thermal conductivity of asphalt mixtures were tested. The results show that asphalt mixtures that containing steel slag are self-healing materials. (16).



Fig. 3 Schematic representation of crack width measurements before and after microwave heating (17)



#### 2.3. Microcapsule Method

Rejuvenators generally apply to the asphalt surface can be encapsulated to overcome deterioration and be added to the asphalt mixture during production. When a crack occurs, the microcapsules in contact break and release the rejuvenator it contains. Briefly, the microcapsule method is a system that bursting of capsules embedded in asphalt when the pressure reaches a certain limit value and releasing some amount of rejuvenator to restore the original properties of the pavement.

The International Conference

of Materials and Engineering Technology

Hot mix asphalt loses its capability over time due to oxidation. Traditionally, to regain the original properties of asphalt pavement, applying rejuvenators to the road surface. However, the problem at this point is that for a rejuvenator to be successful, it must be able to diffuse in the pavement surface. Moreover, the application of the rejuvenator reduces the slip resistance of the pavement. Also, the rejuvenator contains a large number of aromatic compounds that may be harmful to the environment. To solve these problems, the above-mentioned encapsulated rejuvenators are presented as a new concept in road construction. Figure 2.3 shows an experiment dipping a porous stone into a rejuvenator (cooking oil) to produce capsules. This approach is stated to be one of the first steps in smart pavement (18).



**Fig. 4** Optical micrographic of core materials of capsules: (a) Porous stone used; (b) Rejuvenators embedded porous stone (18)

In the microcapsule method; When cracks occur, they break some of the capsules in contact, as shown in Figures 2.4 and 2.5, the oil in the broken capsules contacts the bitumen in the cracks and increases the healing capacity. This process is seen to repair and significantly reduce cracks and is also thought to prevent the spread of new cracks. The basic assumption is that the capsules will break if fatigue cracks and thermal stresses apply a high strain on the capsules.

These rejuvenators will penetrate the bitumen and help reduce its stiffness and restore its chemical balance.



UWE Bristol

Fig. 5 Recovery mechanism with encapsulated rejuvenator (19)



Fig. 6 A cross-section of capsules embedded in asphalt concrete (18)

According to Su and its team, it is necessary to determine the stress to break the shell of the microcapsule to allow the rejuvenator in the microcapsule shell to penetrate and disperse. After the microcapsule shell is broken, the rejuvenator will bring back the properties of the bitumen by contacting the bitumen in the crack and increase the healing capacity of the cracks (20).

In the study conducted by Garcia and his team, the ratio of asphaltenes to maltes was determined in un-aged binders. Rejuvenators were used to provide the asphaltene / maltene ratio before aging in the bituminous binder samples. These products have been used to reduce the loss of fine particles and cracks on the surface. However, these rejuvenators are encapsulated and mixed into asphalt



effective, which in turn reduces shear resistance. The idea behind this practice is that the pressure in the capsules rises above a certain limit value, breaking the capsule and releasing the rejuvenator. The study focused on the properties of these capsules. Four different types of rejuvenator were encapsulated and the effects of the capsules examined. On the other hand, the release mechanisms of the capsules have been tried to be analyzed. Finally, the capsules were added to a porous asphalt mixture and their appearance was examined under a microscope when the asphalt sample was broken under indirect tensile tests. According to the results obtained, the effect of microcapsules on the rejuvenation of bitumen and optimum shell thickness of microcapsules was determined (21). In the study conducted by Qui et al, it is stated that the self-repairing properties of bituminous binders have been known for a long time. Previous researchers on the rheology of bituminous materials have generally focused on self-healing (such as elastic behavior) after load application. Since these experiments are both long-term and complex, a simpler self-repair experiment is presented in this study. In this method, fraction-repair-fraction and image processing are used together. From the results obtained, the load-carrying rate of the fracture increased for the second time as self-repair time and ambient temperature increased. The improvement in strength values and microscopy images consisted of two stages; In the first step, the crack was closed and in the second step the strength was increased. From the results obtained, it is determined that the proposed method is more simple and effective for self-repair of bituminous binders (22).



Fig. 7 Observation of crack repair at different times (0, 1, 3, and 18 hours) by fluorescence microscopy (22)

Su et al, in their study, assuming the basis of their previous work, tried to examine the thermal stability, mechanical stability, and interface stability of microcapsules in bitumen. The results indicate that these microcapsules containing rejuvenator are resistant to repeated temperature changes in the melted bitumen. The microcapsules have been shown resistance to temperature changes and mixing pressure due to their elastoplastic deformation ability. Moreover, chemical bonds improved the interface stability between bitumen and shell (23).

Garcia et al. evaluated the effects of capsules containing fluid oil (low viscosity) in the asphalt mixture when broken under simulated traffic loads. For this purpose, capsules containing sunflower oil were produced and mechanical composition and strength of the capsules were described. The capsules were then added as aggregate to the hot mix asphalt and compacted. To measure the effectiveness of the capsules that added to the hot bituminous mixture, samples were subjected to different numbers of periodic loading, rested and re-tested. As a result, it was observed that the capsules were broken during gradual periodic application and that the oil released from the capsules softened (healed) the bitumen in the asphalt mixture during resting processes (24).



**Fig. 8** (a) Asphalt sample before, testing (b) Asphalt sample after testing with broken capsules (24)

Su et al. examined the properties of bituminous hot mix samples containing microcapsules that containing waste cooking oil (WCO) to improve aging bitumen in situ. The thermal stability of micro WCOs in melting bitumen and resistance to repeated temperature changes are shown in Figure 2.8. From the results obtained, it was determined that WCOs can easily penetrate the aged bitumen as a rejuvenator. Aged bitumen showed improvement in initial properties, including penetration value, softening point value and viscosity value after treatment (25).



Fig. 9 Investigation of the healing of the morphology of micro WCO samples in repeated changes between -10°C and 50°C by fluorescence microscopy (25)



**Fig. 10** The capillarity behaviors of rejuvenator in self-healing bitumen by microcapsules, (a–c) a microcrack was generated by liquid N2 with the width about 10–15 μm, the microcrack

propagates and pierce the shells of microcapsules, (d and e) the liquid of rejuvenator leaked out from microcapsules and flowed into the microcapsules, and (f) movement trace and direction of rejuvenator during the capillarity. (26)

Al-Mansoori et al., calcium-alginate capsules were made which can release cooking oil through a mechanical trigger and mixed into the asphalt mixture to improve self-healing properties in their study. The physical, mechanical and self-healing properties of the asphalt mixture containing these capsules were evaluated for the first time. The capsules were strongly bonded to the asphalt mixture and the results showed that the mechanical performance of asphalt samples with and without capsules in water sensitivity, particle loss, and permanent deformation tests was similar. This means that the capsules used for self-healing of asphalt can be safely used on roads without affecting quality. It was found that 52.9% of the initial strength of the cracked asphalt mixture containing the capsule improved at 20°C, whereas this ratio was 14.0% in the encapsulated asphalt mixture (27).

#### Results

Deteriorations such as low-temperature or fatigue cracking occur mainly due to traffic and climate in asphalt pavements. Expensive and time-consuming maintenance activities are required due to the deterioration of road superstructures. New concepts are being explored to reduce these processes and one of them is the self-healing asphalt. Induction, microwave and capsule use are the most commonly used methods for self-healing. the microcapsule method is a system of the bursting of capsules embedded in asphalt when the pressure reaches a certain limit value and releasing some amount of rejuvenator to restore the original properties of the pavement.

From the studies, it was determined that the capsule was successful for spontaneous healing of the asphalt, in that it allowed the aging binder to heal. However, this approach has the disadvantage that the capsules cannot be reused after breaking. The use of capsules in asphalt mixtures is still in its infancy and should be further developed in the coming years. A reasonable amount of microcapsule should be added to the asphalt mixture to ensure proper dispersion. Besides, the



development of a multi-stage self-healing process in asphalt mixtures should be the focus of future research in this area.

#### Acknowledgments

This study was performed under TUBİTAK (Scientific and Technological Research Council of Turkey) Research Project MAG-217M570. The financial contribution of TUBİTAK is gratefully acknowledged.

#### References

- 1. Sun, D., Sun, G., Zhu, X., Guarin, A., Li, B., Dai, Z., Ling, J. A Comprehensive Review On Self-Healing Of Asphalt Materials: Mechanism, Model, Characterization and Enhancement. Advances in Colloid and Interface Science, 2018, 256, 65-93.
- **2.** Yilmaz, M. The Effects of Addition of Asphaltite on the Mechanical Properties of Bitumen and Bituminous Hot Mixes. Ph.D. Thesis. Firat University. Institute of Science and Technology. Elazig. 2011.
- **3.** ŞengÖz, B., Topal, A. Use of Asphalt Roofing Shingle Waste in HMA, Construction and Building Materials. 2005, 19(5): 337-346.
- **4.** Kaşak, S. Investigation of Usability of Diatomite Instead of Fiber as Additive in Stone Mastic Asphalt Mixture. Master Thesis. Gazi University, Institute of Science and Technology. Ankara. 2007.
- **5.** General Directorate of Highways Turkey (KGM). Highways Flexible Pavements Design Guide. Technical Research Department. Pavement Branch Directorate. 2008.
- 6. Edwards, Y., Taşdemir, Y., Isacsson, U. Effects of Commercial Waxes on Asphalt Concrete Mixtures Performance at Low and Medium Temperatures. Cold Regions and Science Technology, 2006, 45(1): 31-41.
- 7. Tunç, A. Road Materials and Applications. Ankara. Nobel Press Distribution. 2007.
- 8. Lav, A.H., Lav, M.A. Shell Bitumen Guide. Istanbul. İSFALT Scientific Publications, 2004, 334p.
- **9.** Yalcin, E., Norambuena-Contreras, J., Garcia, A., Yılmaz, M., 2018. Self-Healing Methods Used in Asphalt Mixtures. 20th International Conference on Pavement Engineering. Paris. France. March, 2018, 1219-1225.
- **10.** Francken, L. Fatigue Performance of a Bituminous Road Mix Under Realistic Best Conditions. Transportation Research Record, 1979, 712, 30-34.
- **11.** Van Dijk, W., Moreaud, H., Quedeville, A., Uge, P. The Fatigue of Bitumen and Bituminous Mixes. 3rd int. Conference on the Structural Design of Asphalt Pavements. Ann Arbor, Michigan, USA, 1972.
- 12. Hager, M.D., Greil, P., Leyens, C., Van der Zwaag, S., Schubert, U.S. Self-Healing Materials. Materials Today, 2010, 22(47): 5424-5430.
- **13.** Chung, K., Lee, S., Park, M., Yoo, P., Hong, Y. Preparation and Characterization of Microcapsule-Containing Self-Healing Asphalt. Journal of Industrial and Engineering Chemistry, 2015, 29, 330-337.
- **14.** Liu, Q. Induction Healing of Porous Asphalt Concrete. Ph.D. Thesis. Faculty of Civil Engineering and Geosciences, 2012, TU Delft, Netherlands.
- **15.** Garcia, A., Schlangen, E., Van De Ven, M., Van Vliet, D. Induction Heating of Mastic Containing Conductive Fibres and Fillers. Materials and structures, 2011, 44, 499-508.



- **17.** Norambuena-Contreras, J., Gonzalez-Torre, I. Influence of the Microwave Heating Time on the Self-Healing Properties of Asphalt Mixtures. Applied Sciences, 2017, 7, 1076.
- **18.** Garcia, A., Schlangen, E., Van De Ven, M., Sierra-Beltran, G. Preparation of Capsules Containing Rejuvenators for Their Use in Asphalt Concrete. Journal of Hazardous Materials, 2010, 184, 603-611.
- **19.** Tabakovic, A., Schlangen, E. Self-Healing Technology for Asphalt Pavements. Self-Healing Materials, 2015, 285-306.
- **20.** Su, J.F., Qiu, J., Schlangen, H. Self-Healing Bitumen by Microcapsules Containing Rejuvenator. ICSHM 2013: Proceedings of the 4th International Conference on Self-Healing Materials, Ghent, Belgium, 2013, Ghent University; Delft University of Technology.
- **21.** Garcia, A., Schlangen, E., Ven, M. Properties of Capsules Containing Rejuvenators for Their Use in Asphalt Concrete. Fuel, 2011, 90, 583-591.
- **22.** Qiu, J., Ven M., Wu, S., Yu, J., Molenaar, A. Evaluating Self-Healing Capability of Bituminous Mastics. Experimental Mechanics, 2011, 52, 1163-1171.
- **23.** Su, J.F., Qiu, J., Schlangen, E. Stability Investigation of Self-Healing Microcapsules Containing Rejuvenator for Bitumen. Polymer Degradation and Stability, 2013, 98, 1205-1215.
- **24.** Garcia, A., Jelfs, J., Austin, C.J. Internal Asphalt Mixture Rejuvenation Using Capsules. Construction and Building Materials, 2015, 101, 309-316.
- **25.** Su, J.F., Qui, J., Schlangen, E. and Wang, Y.Y. Investigation the Possibility of a New Approach of Using Microcapsules Containing Waste Cooking Oil: In Situ Rejuvenation for Aged Bitumen. Construction and Building Materials, 2015, 74, 83-92.
- **26.** Su, J.F., Schlangen, E., Wang, Y.Y. Investigation the Self-Healing Mechanism of Aged Bitumen Using Microcapsules Containing Rejuvenator. Construction and Building Materials, 2015, 85, 49-56.
- **27.** Al-Mansoori, T., Micaelo, R., Artamendi, L., Norambuena-Contreras, J., Garcia A. Microcapsules for Self-Healing of Asphalt Mixture Without Compromising Mechanical Performance. Construction and Building Materials, 2017, 155, 1091-1100.



# DISCONTINUOUS PRECIPITATION IN METALLIC ALLOYS

## ZAKARIA BOUMERZOUG

Department of Mechanical Engineering, University of Biskra, B.P. 145, Biskra, 07000, Algeria

#### Abstract

Discontinuous precipitation is a solid state phase transformation. It is generally formed at grain boundaries of metallic alloys. This paper presents a literature review on previous research works on discontinuous precipitation. The mechanisms of initiation and growth of this reaction are presented. In addition, the main parameters which affect its mechanism are listed. The past, present and future research questions are formulated.

Keyword: Discontinuous precipitation, metallic alloys, phase transformation.

#### **1.Introduction**

The 1.1. Definition

Discontinuous precipitation (DP) (or cellular reaction) is a solid state decomposition reaction that converts supersaturated solid solution  $\alpha_0$  into a two phases  $\alpha + \beta$  behind a migrating reaction front (RF). [1]:  $\alpha_0 \rightarrow \alpha + \beta$ 

The cellular reaction originates at high angle grain boundaries by the formation of stable  $\beta$ -plate enriched in one of solute elements [2]. The discontinuous precipitation (DP) is diffusive solid state phase transformation during which a supersaturated solid solution decomposes into a usually lamellar structure consisting of alternate lamellae of new phase  $\beta$  and solute depleted  $\alpha$  phase having the same crystal structure as initial phase  $\alpha_o$  (Fig.1) [3]. The term "cellular precipitation" can be used instead of "discontinuous precipitation", because the shape of precipitates is as cells with lamellar structure. During growth reaction of this cell, the branching mechanism of branching is also illustrated. This branching is done by formation of new  $\beta$  lamella by branching at the RF in the area indicated by narrow.





The reaction front, which is initially a grain boundary (GB), moves over the supersaturated matrix and is generally capable of supporting a composition gradient across it and a composition profile along it [4,5]. The reaction generates discontinuous changes in crystal orientation and solute concentration across the boundary-reaction front [2]. For this reason, it is called discontinuous precipitation. Figure 2 shows a typical of cellular precipitation in aged Mg-8 % Al. alloy at 150°C. The product phases formed as an aggregate of lamellar structure consisting of alternate lamellae of  $\alpha$  and  $\beta$  phases. In this micrograph, the phase  $\beta$  appears in white color.



Figure 2. Celluar precipitation in Mg-8 % Al. alloy aged at 150°C for 50 h.

Coarsening reaction is a second reaction which may also succeed the first finer structure formed after the discontinuous precipitation. The mechanism of this second reaction is done by a discontinuous process and the lamellae are larger.



Since its year of its discovery in 1931 [6] and as mentioned by Manna et al. [7], a discontinuous precipitation has been observed in more than 131 binary alloys and 113 ternary alloys. In addition, the number of annual publications on the event has not ceased to increase [8]. However, discontinuous reaction can generate dramatic effects on the properties of materials. Consequently, to control the DP reaction, it is important to understand the mechanism and the kinetics of this reaction in alloy systems used in technology application such as functional materials, either to prevent it or to promote it [8]. As an example, Casias [9], found that discontinuous precipitation of lamellar  $M_{23}C_6$  is harmful to stress rupture life and ductility.

#### 2. Research questions on discontinuous precipitation

It is interesting to note that the most important research questions on discontinuous precipitation are divided in two categories. The first category is related to the understanding of the mechanisms of different stages of a discontinuous precipitation reaction (initiation, growth, coarsening). However, the second category is related to the understanding of the effect of the external parameters on the discontinuous precipitation such as, effect of the third element in binary alloy. In the next following section, previous investigations on three selected research questions will be presented:

2.1. Firs research question: Sites of initiation

Han et al. [10] have reported that numerous studies have suggested that in general the DP process starts at the grain boundary and the interface. In our previous investigations [11-16] the occurrence of discontinuous precipitation in grain boundaries and in other sites of the supersaturated phase  $\alpha_0$  was confirmed.

2. 2. Second research question: Mechanisms of initiation

Two main models are considered: precipitate-induced boundary migration and precipitation on migrating boundaries:

2.2.1. Precipitate-induced boundary migration

This model established by Tu and Turnbull [17] after investigation of Pb–Sn alloy. They observed that the first step for cell formation is the nucleation of a particle  $\beta$  at the grain boundary. Further growth of  $\beta$  may be determined by the habit plane with one of the two adjoining grains. Tu and Turnbull supposed that the orientation relationship is that of minimum interfacial energy. It is called also "pucker" mechanism. Figure 3 illustrates the different steps of the "pucker" mechanism.

Technology



Figure 3. Pucker mechanism for initiation reaction according to Tu and Turnbull [17].

Figure 4 shows an example of discontinuous precipitation in Mg- 8 wt. % Al alloy. The new second phase nucleates at grain boundary which takes a shape of "S" and it is known as S-mechanism. This mechanism reflects the high mobility of grain boundary which accelerates the diffusion process during discontinuous decomposition. This initiation reaction is close to the Tu and Turnbull mechanism "Pucker mechanism".



**Figure 4.** Microstructures of Mg- 8 wt. % Al after homogenization at  $420 \,^{\circ}$ C, quenched in water and followed by ageing treatment at 150  $^{\circ}$ C during 203 h.

2.2.2. Precipitation on migrating boundaries

Fournelle and Clark [18] considered the migration of a grain boundary due to its curvature and advanced a model for the development of a cell nucleus. The migrating grain boundary depletes the area behind it of solute atoms by forming precipitates.



Figure 5. Development of a precipitation cell according to Fournelle and Clark [18].

2.3. Second research question: Parameters affecting DP.

In this part, two parameters will be considered. Abdou et al [19] found that the discontinuous precipitation rate depends strongly on the degree of deformation in Ni-In Alloy. In our previous investigation, We have studied the effect of plastic deformation by compression on the occurrence of discontinuous precipitation in Al-30% Zn alloy after ageing at two different temperatures (348 and 423 K) has been studied. This reaction is observed only for low degree of deformation (Figure 6). For higher deformation, the alloy is occupied only by deformation bands (Figure 7). which represent favorite sites of finer continuous precipitates [14].



Figure 6. Microstructure of Al-30 wt% Zn alloy during aging at 348 K for 5 h [14].



Figure 7. Microstructure of Al-30 wt. % Zn alloy after 45% compressive strain and aged at 348 K for 5 h [14].

Semboshi et al. [20] investigated the effect of boron doping on cellular discontinuous precipitation for age-hardenable Cu–Ti alloys. They found that the volume fraction of the discontinuous precipitates is lower in the Cu–4Ti–0.03B alloy than the Cu–4Ti alloy.



#### 3. Future scope of DP

There are several scientific challenges that require future focus. As mentioned by Zieba [3], the understanding the mechanism and kinetics of the discontinuous precipitation requires the use of very sophisticated analytical tools [3]. The objective will be to measure the solute redistribution across the reaction front and within solute depleted lamella, especially for new types of discontinuous reactions. The other challenge is giving more attention to the effect of third element on the discontinuous precipitation in binary alloys. We must note that the discovery of this discontinuous precipitation in the meteorite [21], gave a broader scope to the scientific investigation on this phase transformation.

The International Conference of Materials and Engineering Technology

#### 4. Conclusion

A discontinuous precipitation is among of phase transformation at solid state. Discontinuous precipitation is a diffusion reaction, and it has been observed in more than two hundred metallic alloys. This reaction can generate dramatic effects on their properties. The present review has been undertaken with an objective to present this reaction and the different research questions which remain as challenges such as the effect of external parameters on its development.

#### References

- 1. Gust, W. in "Phase transformation", The Institution of Metallurgists, 1979, London, (Vol. 1, p. II/27.
- 2. Williams, DB, and Butler, EP. Grain boundary discontinuous precipitation reactions.1981, Int Mater Rev 3:153–183.
- 3. Zieba, P., Recent developments on discontinuous precipitation, Arch. Metall. Mater.2017, 62, (2): 955-968.
- 4. Cahn, JW. The kinetics of cellular segregation reactions. Acta Metall. 1959, 7:18–28.
- 5. Solorzano, IG, Purdy, GR, and Weatherly, GC. Studies of the initiation, growth and dissolution of discontinuous precipitation product in aluminum-zinc alloys. Acta Metall 1984, 32:1709–1717.
- 6. Ageew, N, and Sachs, G. Ro<sup>°</sup>ntgenographische Bestimmung der Lo<sup>°</sup>slichkeit von Kupfer in Silber. Z Phys. 1930, 66:293–30.
- 7. Manna, I., Pabi; S.K. and Gust, W. Discontinuous reactions in solids, <u>International</u> <u>Materials Reviews</u>, 2001, 46, (2):53
- 8. Suguihiro, N. M., and Solorzano, I. G. On the initiation, growth, and coarsening of discontinuous precipitation in Cu-10 at.% Co alloy , J Mater Sci. 2016, 51:71–81.
- 9. Casias, A. M. thesis, influence of composition on precipitation behavior and stress rupture properties in inconel 740 series superalloys, Colorado School of Mines, 2012.
- 10. Han, S. Z. , Ahn, J. H. , You, Y. S. , Lee, J. , Goto, M. , Kim, K. , and Kim, S. , Met. Mater. Int.2018, Vol. 24, (1): 23-27.
- 11. Boumerzoug Z., Fatmi M., Effect of heat treatments on discontinuous precipitation kinetics in Al-30 wt.% Zn alloy, Materials Characterization, 2009, 60 (8): 768-774.
- Boumerzoug, Z., and Hamana, D. Different sites of discontinuous precipitation and mechanisms of dissolution in Cu-9 wt.% Sb alloy, Materials chemistry and physics, 2001, 69 (1-3):10-18.
- Hamana, D., Boumerzoug, Z., Fatmi, M., and Chekroud, S. Discontinuous and continuous precipitation in Cu-13 wt.% Sn and Al-20 wt.% Ag alloys, Materials chemistry and physics, 1998, 53 (3): 208-216.



- of discontinuous precipitation, Journal of materials science, 2005, 40 (12): 3199-3203.
- 15. Fatmi, M., and Boumerzoug, Z. Influence of plastic deformation on occurrence of discontinuous precipitation reaction in Ni-3 at% In alloy, Physica B: Condensed Matter, 2010, 405 (19): 4111-4115.
- 16. Hamana, D., Boumerzoug, Z., and Saheb, N. Cellular precipitation from phase boundaries in Cu-9 wt% Sb alloy, Philosophical magazine letters, 1995, 72 (6) : 369-374.
- 17. Tu,KN., and Turnbull, D. Morphology of cellular precipitation of tin from lead-tin bicrystals. Acta Metal, 1967, 15: 369-376.
- 18. Fournelle, RA, and Clark, JB. The genesis of cellular precipitation reaction. Metal Trans. 1972, (3):2757–2767.
- 19. Abdou; S., and El-Hafez; H.A. Influence of Plastic Deformation on Occurrence of Discontinuous Reaction in Ni-In Alloy, WJET, 2018, 6 (2): 492-503.
- 20. Semboshi; S., Ikeda; J., Iwase, A., Takasugi, T., and Suzuki, S. Effect of Boron Doping on Cellular Discontinuous Precipitation for Age-Hardenable Cu-Ti Alloys, Materials (Basel). 2015, 8(6): 3467-3478.
- 21. Grokhovsky, V. J. and Bevan, A. W. R. Plessite formation by discontinuous precipitation reaction from y-Fe,Ni in Richardton (H5) ordinary chondrite, Nature, Letter published on 27 January 1983



# BONCUKLANMA GÖRÜNTÜLERININ İŞLENMESİNDE FARKLI FİLTRELERIN KULLANIMI

of Materials and Engineering Technology

#### ABDURRAHMAN TELLİ<sup>\*1</sup>

<sup>1</sup> Çukurova Üniversitesi, Tekstil Mühendisliği Bölümü, Adana, TÜRKİYE.

#### Özet

Son yıllarda örme kumaşların boncuklanma derecelerinin belirlenmesi için dijital görüntü işleme çalışmalarına başvurulmaya başlandığı görülmektedir. Çünkü sübjektif değerlendirme yönteminde insan faktörü önemli seviyede etkili olmaktadır. Bu durum çeşitli anlaşmazlıklara sebebiyet vermektedir. Bu çalışmada, MATLAB yazılımı ile boncuklanma görüntülerinin işlenmesinde farklı filtrelerin kullanımı incelenmiştir. Örme kumaştan alınan üç numuneye dört farklı devirde boncuklanma testi uygulanmıştır. Öncelikle numuneler subjektif olarak değerlendirilmiştir. Daha sonra görüntüleri alınan numunelerde dört farklı temel filtreleme tekniği içeren görüntü işleme çalışmaları gerçekleştirilmiştir. Elde edilen bulgular sübjektif değerlendirme sonuçlarıyla karşılaştırılmıştır. 2D Gaussian filtreleme ve 2D Median filtreleme tekniklerinde görüntü ile daha uyumlu sonuçlar elde edilmiştir. Toplam alan, boncuk ve tüy sayısı temel olarak dikkate alındığında 2D Median filtreleme'de daha başarılı sonuçlar elde edildiği görülmüştür. Çalışma boncuklanma görüntülerinin işlenmesinde filtre seçiminin önemli seviyede etkili olduğunu göstermektedir.

Anahtar Kelimeler: örme kumaş, boncuklanma, Martindale, görüntü işleme, MATLAB

#### 1. Giriş

Boncuklanma, kumaşın kendi kumaşına karşı veya farklı yüzeylere hareketi ile oluşmaktadır. Boncuklanma test sonuçları kullanılacak giysinin kalitesini ve ömrünü tespit etmede önemli bir yer tutmaktadır. Sürtünmeye maruz bırakıldığında tüm kumaş yüzeylerinde boncuklanma gözlenmektedir. Ancak boncukların oluşma hızı, boncukların kumaş yüzeyinden ayrılma hızından daha yüksek olduğunda, boncuklanma rahatsız edici seviyelere gelmektedir. Özellikle örme kumaşların boncuklanması önemli bir problemdir. Çünkü örme kumaşlar, kumaş yapıları nedeniyle diğer kumaşlardan daha fazla boncuklanma eğilimindedir. Daha önceki çalışmalar genel olarak, boncuklanma üzerine etki eden parametreler, boncuk oluşumuna yönelik farklı yöntemler, boncuklanma derecesini veren değerlendirme teknikleri ve bitirme işlemlerinin etkisi üzerine yoğunlaşmaktadır. Lif, iplik, dokuma, örme, boyama ve terbiye gibi parametrelerin boncuklanma direnci üzerindeki etkilerini açıklamaya yönelik çok sayıda çalışma yapılmıştır [1-4]

Örme kumaşların boncuklanma eğilimi, kutulu boncuklanma, taklalı serbest düşme ve Martindale yöntemi gibi laboratuvar koşullarında farklı yöntem ve cihazlarla test edilmektedir. Bu yöntemlerde, kumaşların günlük yaşamdaki koşulları simüle edilmektedir. Kumaş yüzeyinde oluşturulan boncuklar eğitimli ve deneyimli uzmanlar tarafından gözle değerlendirilmektedir. Ayrıca, bu değerlendirme standart fotoğraflar ile desteklenmektedir [5, 6]. Ancak bu sübjektif değerlendirme yönteminde insan faktörü önemli seviyede etkili olmaktadır. Telli (2019) tarafından bu yöntemin güvenilirliği araştırılmıştır. Bu araştırmanın sonuçları, uzmanlar arasında güçlü bir korelasyon bulunduğunu göstermektedir. Ancak çalışmada on iki farklı kumaş üzerinde üç uzman tarafından hiçbir örneğe aynı boncuklanma derecesi verilmemiştir. Uzmanlar arasındaki görsel algıdaki farklılıklar aynı sonuçları elde etmeyi zorlaştırmaktadır. Duyarlılığın yarım puan azaltılması ile benzer bulgular sağlanabilmiştir. Boncuklanmanın değerlendirilmesinde görüntü işleme gibi objektif yöntemlere daha fazla ihtiyaç duyulduğu belirtilmiştir [7].

Boncuklanma görüntülerinin işlenmesi ilk olarak Konda et al. (1990) tarafından gerçekleştirilmiştir. Bir boncuklanma görüntüsünden boncuk boyutlarının dağılım eğrisinin, toplam boncuk sayısının, boncukların toplam ve ortalama alanının elde edilebileceğini göstermiştir [8]. Önemli sayıda çalışma standart fotoğrafları kullanarak boncuklanma parametrelerini açıklamaya çalışmıştır [9]. Sınırlı sayıda çalışma ise görüntülerin dokusal parametreleri üzerine odaklanmıştır [10]. Furferi et al. (2015) ve Eldessouki (2018) objektif değerlendirme teknikleri ve aşamaları ile ilgili literatürü kronolojik olarak sıralamaktadır [11, 12]. Bu çalışmada, MATLAB yazılımı ile boncuklanma görüntülerinin işlenmesinde farklı filtrelerin kullanımı incelenmiştir.

#### 2. Materyal ve Yöntem

Bu çalışmada 240 g/m<sup>2</sup> ağırlığında %72 Pamuk %20 Yün %8 Elastan içeren Punta-Di-Roma örme kumaş kullanılmıştır. Kumaştan alınan üç numunede 1000, 2000, 3000, 5000 devirlerde boncuklanma testi gerçekleştirilmiştir. Boncuklanma TS EN ISO 12945-2 standardına göre "Nu-Martindale Aşınma ve Boncuklanma Test Cihazı"nda uygulanmıştır. Daha sonra iki deneyimli personel tarafından standart fotoğraflar yardımıyla ışık kabininde gözle sübjektif olarak kumaşların değerlendirilmesi yapılmıştır. Değerlendirme sonrası farklı devirlerdeki boncuklanma dereceleri sonuçları Tablo 1'de verilmektedir.

<b>Tablo 1.</b> Ivalitatic Rainașa alt farkii devinerdeki obneakialina derecelen sonaçian							
Devir	1000	2000	3000	5000			
Sonuç	4-5	4	3-4	2-3			

Tablo	1.	Numune	kumaşa	ait	farklı	devir	lerdeki	boncul	klanma	derecel	leri	sonuç	lar
-------	----	--------	--------	-----	--------	-------	---------	--------	--------	---------	------	-------	-----

Numunelerin başlangıçta ve her bir devir sonrasında görüntüleri dijital tarayıcı kullanılarak 300x300 dpi 24 bit renkte alınmıştır. Alınan görüntüler boncuklandırılmış bölgeleri içerecek şekilde 900x900 piksel olarak boyutlandırılmıştır. Daha sonra görüntü işleme çalışmaları gerçekleştirilmiştir. Görüntü işleme çalışmaları MATLAB R2018a paket programında yapılmıştır. Tarayıcıdan RGB formatında (mXnX3 uint 8) alınan görüntüler, önce iki boyutlu (mXn uint8) 0 ile 256 arasında grinin farklı tonlarını ifade eden matrislere dönüştürülmüştür. Bu işlemden sonra üzerinde filtreleme yapabilmek için matris 0-1 değerler aldığı "mXn double" formatına getirilmiştir [13, 14]

Bu matrislerde dört farklı temel filtreleme tekniği uygulanmıştır. Bunlar medfilt2 (2D median filtering), imgaussfilt (2D Gaussian filtering of images), stdfilt (local standart devitiation of image) ve rangefilt (local range of image) şeklindedir. Filtre uygulanmış görüntülerde histogram eşitleme tekniği kullanılarak görüntü bölütleme için en uygun eşik değeri (thresholding value=0,49804) belirlenmiştir [15]. Bölütlemede Otsu'nun global eşikleme algoritması kullanılmıştır. Daha sonra tercih edilen büyüklüğün altındaki değerler maskelenmiştir. Böylelikle görüntü içerisindeki boncuk ve tüylerin görüntü arka planından ayrılması sağlanmıştır. Maskelemede boncuklanma öncesi kumaş görüntüleri referans olarak kullanılmıştır. Bu durumda medfilt2 ve imgaussfilt


filtrelemenin kullanıldığı görüntülerde maskeleme "open mask (shape=disk radius=4)", stdfilt ve rangefilt filtrelemenin kullanıldığı görüntülerde "close mask (shape=disk radius=5)" şeklinde olmuştur. Sonuç olarak (mXn logical) formatında matrisler elde edilmiştir. Bu matrislerden "Image Region Analyzer" kullanılarak görüntüler bölgesel olarak analiz edilmiştir. Tespit edilen toplam alan, boncuk ve tüylerin sayısı belirlenmiştir. Kullanılan üç numunedeki sonuçların ortalamaları alınmıştır. Elde edilen bulgular sübjektif değerlendirme sonuçlarıyla kıyaslanmıştır.

# 3. Bulgular ve Tartışma

Şekil 1'de örnek olarak örme kumaşın ilk numunesinin boncuklanma öncesi ve 2000 devir sonrası görüntüleri yer almaktadır.



Şekil 1. Örme kumaşın ilk numunesinin boncuklanma öncesi ve 2000 devir sonrası görüntüleri

Kumaşın ilk hali ile kıyaslandığında görüntüde kenarlarda yoğunlaşmaya başlamakla birlikte tamamında hafif bir tüylenme görülmektedir. Ayrıca boncukların kısmi olarak oluşmaya başladığı da açıkça görülmektedir. 2000 devir sonrası görüntüde belirgin boncuklar işaretlenmiştir (Şekil 1). Şekil 2-5'de ise medfilt2, imgaussfilt, stdfilt ve rangefilt filtrelemenin kullanıldığı görüntü işleme çalışmaları sonrası elde edilen görüntüler yer almaktadır.



Şekil 2. Medfilt2 filtrelemede histogram eşitleme (solda) ve maskeleme (sağda) sonrası elde edilen görüntüler



Şekil 3. Imgaussfilt filtrelemede histogram eşitleme (solda) ve maskeleme (sağda) sonrası elde edilen görüntüler



Şekil 4. Stdfilt filtrelemede histogram eşitleme (solda) ve maskeleme (sağda) sonrası elde edilen görüntüler



**Şekil 5.** Rangefilt filtrelemede histogram eşitleme (solda) ve maskeleme (sağda) sonrası elde edilen görüntüler

Şekil 1'deki 2000 devir sonrası boncuklanma görüntüsündeki belirgin boncuklar üzerinden bir değerlendirme yapıldığında, medfilt2 ve imgaussfilt filtrelerinin kullanıldığı görüntülerdeki boncukların konum olarak orijinal kumaşa daha benzer alanlarda oluştuğu gözlemlenmektedir. Stdfilt ve rangefilt filtrelerin kullanıldığı görüntülerde ise maskeleme sonrası öne çıkan boncuklar ile orijinal kumaşta yer alan boncuk konumlarının uyumlu olmadığı görülmektedir. Stdfilt ve rangefilt filtrelerin çalışmada kullanılan örme kumaş için uygun olmadığı düşünülerek görüntülerdeki bölütlenmiş boncuk ve tüylerin bölgesel analizlerinin yapılmasına gerek duyulmamıştır. Değerlendirme dışında tutulmuştur. Medfilt2 ve imgaussfilt filtrelerinin kullanıldığı görüntü matrislerinden "Image Region Analyzer" kullanılarak yapılan bölgesel analiz sonuçlarından tespit edilen toplam alan, boncuk ve tüy sayıları Tablo 2'de yer almaktadır.

of Materials and Engineering Technology

Dovin	Subjektif	Adet Medfilt2 Imgaussfilt		Toplam Alan	
Devir	Değerlendirme			Medfilt2	Imgaussfilt
0	5	138	24	9624	1445
1000	4/5	241	69	19554	4945
2000	4	271	74	22518	5402
3000	3/4	302	74	24622	5271
5000	2/3	374	98	31482	7310

Tablo 2. Bölgesel analiz sonuçlarından tespit edilen toplam alan, boncuk ve tüy sayıları

Tablo 2 incelendiğinde her iki filtrede de boncuklanma derecesi düştükçe toplam alan, boncuk ve tüy sayılarının arttığı görülmektedir. Ancak hem adet ve hem de toplam alanda imgaussfilt filtreye göre medfilt2'de daha yüksek değerler elde edilmiştir. Şekil 2 ve 3'deki maskeleme sonrası görüntülerden de görüleceği üzere, medfilt2'nin rahatsız edici tüylerin algılanmasına daha fazla katkı yaptığı söylenebilmektedir. Imgaussfilt filtreleme boncukların tespitinde medfilt2'ye benzer sonuçlar verirken, rahatsız edici tüylerin algılanmasında eksik kaldığı görülmektedir. Elde edilen bulguları sübjektif değerlendirme sonuçlarıyla kıyaslamak için Pearson korelasyonu kullanılmıştır. Korelasyon analizi sonuçları Tablo 3'de sunulmaktadır.

		Bağıntı Katsayısı
		( <b>r</b> )
Adat	Medfilt2	0,963
Adet	Imgaussfilt	0,883
Toplam Alan	Medfilt2	0,955
Topiani Alan	Imgaussfilt	0,885

|--|

Elde edilen bağıntı katsayısı değerleri hem adet ve hem de toplam alanda her iki filtrenin de sübjektif değerlendirme sonuçlarıyla negatif yönlü ve güçlü bir ilişkisi olduğunu göstermektedir (Tablo 3). Imgaussfilt filtrelemede toplam alanda adet sayısına göre kısmen daha yüksek bağıntı katsayısı değeri gözlemlenmiştir. Medfilt2'de ise tersi durum söz konusudur. Medfilt2 filtresinde hem adet ve hem de toplam alan için imgaussfilt filtrelemeye göre daha yüksek bağıntı katsayısı değerleri elde edilmiştir. Sonuçlar örme kumaşların boncuklanmasının objektif olarak değerlendirilmesine giden yolda medfilt2 filtreleme tekniğine daha fazla öncelik verilmesi gerektiğini göstermektedir.

# 4. Sonuç

Stdfilt ve rangefilt filtreleme tekniklerinden sonra görüntülerdeki boncuk ve tüylerin bölütlenmesinin çalışmada kullanılan örme kumaş için uygun olmadığı tespit edilmiştir. Imgaussfilt ve medfilt2 tekniklerinde görüntü ile daha uyumlu sonuçlar elde edilmiştir. Subjektif boncuklanma dereceleri ile karşılaştırıldığında ise görüntü özniteliklerinden toplam alan, boncuk ve tüy sayıları temel olarak dikkate alındığında medfilt2'de daha başarılı sonuçlar elde edildiği görülmüştür. Çalışma boncuklanma görüntülerinin işlenmesinde filtre seçiminin önemini

The International Conference of Materials and Engineering Technology göstermektedir. Sonraki çalışmalarda daha fazla sayıda ve çeşitte kumaş numuneleri ile filtreleme tekniklerinin incelenmesi kumaş boncuklanmalarının objektif olarak değerlendirilmesine önemli katkılar sağlayacaktır.

MET

### Teşekkür

Bu çalışma Çukurova Üniversitesi BAP Birimi tarafından desteklenmiştir (Proje Kodu: FBA-2017-9938).

#### Kaynakça

- 1. Ukponmwan, J. O., Mukhopadhyay, A., & Chatterjee, K. N., Pilling, Textile Progress. 1998, 28(3), 1-57.
- 2. Özdil, N., Kumaşlarda Fiziksel Kalite Kontrol Yöntemleri, E.Ü. Tekstil ve Konfeksiyon Araştırma Uygulama Merkezi Yayınları Yayın No: 21. 2003, ISBN No: 975-483-579-9, Bornova-İzmir, 120s.
- 3. Özçelik, G., Kumaş Boncuklanma Özelliğinin Objektif Olarak Değerlendirilmesi ve Tahminlenmesi Üzerine Bir Araştirma, E.Ü. Fen Bilimleri Enstitüsü Doktora Tezi. 2009, Bornova-İzmir, 291s.
- 4. Telli A., The Relationship between Yarn Properties and Pilling Resistance in Knitted Fabrics, 3rd International Symposium on Innovative Approaches in Scientific Studies (ISAS 2019). 19-21 Apr 2019, pp.238-240.
- 5. Tekstil Kumaşlarda yüzey tüylenmesi ve boncuklanma yatkınlığının tayini Bölüm 2: Gelistirilmis Martindale metodu. TS EN ISO 12945-2/Nisan 2002.
- 6. Kayseri, G. Ö., & Kırtay, E., Farklı Ölçüm Yöntemleri İle Kumaş Boncuklanma Eğiliminin Değerlendirilmesi. Tekstil ve Mühendis. 2011, 18(84).
- 7. Telli A., The Reliability of Subjective Assessment in The Determination of Pilling Resistance of Knitted Fabrics, 4<sup>th</sup> International Mediterranean Science and Engineering Congress (IMSEC 2019), 25-27 Apr 2019, pp.420-422.
- 8. Konda, A., Xin, L. C., Takadera, M., Okoshi, Y., & Toriumi, K., Evaluation of pilling by computer image analysis, Journal of the textile Machinery Society of Japan. 1990. 36(3), 96-107.
- 9. Kim, S. C., & Kang, T. J., Image analysis of standard pilling photographs using wavelet reconstruction, Textile Research Journal. 2005, 75(12), 801-811.
- 10. Eldessouki, M., & Hassan, M., Adaptive neuro-fuzzy system for quantitative evaluation of woven fabrics' pilling resistance, Expert Systems with Applications. 2015, 42(4), 2098-2113.
- 11. Furferi, R., Governi, L., & Volpe, Y., Machine Vision-Based Pilling Assessment: A Review, Journal of Engineered Fabrics & Fibers (JEFF). 2015, 10(3).
- 12. Eldessouki, M., Evaluation of fabric pilling as an end-use quality and a performance measure for the fabrics, In Applications of Computer Vision in Fashion and Textiles. 2018 (pp. 147-187).
- 13. Telli, A., & Özkan, İ., The Objective Evaluation Of Pilling Tendency Of Knitted Fabrics Through Digital Image Processing, ITTC- 7<sup>th</sup> International Technical Textiles Congress, 10-12 October 2018, 309-312.
- 14. Telli, A., & Özkan, İ., Objective Measurement of Pilling Resistance in Knitted Fabrics with Image Processing Techniques. 2018, 25(112).
- 15. Xin, B., Hu, J., & Yan, H., Objective evaluation of fabric pilling using image analysis techniques. Textile Research Journal. 2002, 72(12), 1057-1064.



of Materials and Engineering Technology

## HATICE AYLIN KARAHAN TOPRAKCI<sup>1,2</sup>, AYSE TURGUT<sup>1,2</sup>, OZAN TOPRAKCI<sup>1,2</sup>, MUHSINE ZORCAN<sup>1,2</sup>

#### Corresponding Author: aylin.toprakci@yalova.edu.tr

<sup>1</sup>Yalova University, Engineering Faculty, Polymer Engineering Department, Yalova, TURKEY. <sup>2</sup>Yalova University, Institute of Science, Yalova, TURKEY.

#### Abstract

Polyacrylonitrile (PAN) nanocomposite fibers were fabricated by electrospinning. Halloysite nanotubes (HNTs) were used at various concentrations. In order to obtain uniform, bead-free nanocomposite fibers and optimize the nanofiber morphology, some spinning conditions and solutions were changed. The morphology of the nanocomposite fibers was analyzed by scanning electron microscopy. The results showed the importance of the processing conditions. Increased feed rate and increased filler concentration led to larger fiber diameter.

Key words: Electrospinning, Nanofibers, PAN nanofibers, PAN nanocomposite fibers, Nanocomposite

#### 1. Introduction

Polyacrylonitrile is one of the most used polymers in the fiber industry. In addition to its superior fiber forming properties, its mechanical properties are good enough to be used for many applications including textiles, filtration, membranes and so on. Since traditional spinning techniques limit the fiber diameter in the micron range; electrospinning is commonly used in order to obtain nanofibers. Since electrospinnig is a versatile method, solution properties, processing and ambient conditions affect the final properties of the nanofibers. One of the most significant contribution of the electrospinning is to provide tunable nanofiber morphology. By this method, fiber diameter, fiber porosity, surface area can be optimized by changing solvent type, polymer concentration, aaplied voltage, neddle-collector distance, feed rate, filler type, filler ratio and so on [1-5].

Halloysite, a naturally abundant aluminosilicate with hollow tubular structure nanotube and a diameter around 10-200 nm. Halloysite nanotubes (HNTs) have been used for many applications including cosmetics, household and personal care products, pest repellents, pharmaceuticals and so on. In addition to those, promising applications can be given as: template synthesis of some nanomaterials, controlled and sustained release of some chemicals, molecular sieves, ion adsorption, reinforcement for polymer composites. Even low amounts of HNT addition results

higher modulus, hardness, impact resistance. It also prevents heat and mass transport due to its nanotube structure and it is used as a flame retardant or thermal stabilizator for nanocomposites [6-9].

In this study, nanocomposite HNT/PAN fibers were fabricated by using electro-spinning method. Various spinning solutions were prepared in order to see the effect of filling ratio and composition. In addition to that processing conditions were changed to obtain tunable morphology. The morphology of the nanocomposite fibers was carried out by scanning electron microscopy.

# 2. Materials and Methods

Polyacrylonitrile (PAN) and solvent dimethyl form amide (DMF) were purchased from Merck. The filler halloysite nanotubes (HNTs) were supplied from Esan, Turkey.

PAN and HNTs were used after the drying (80 °C for 24h). 15 % wt PAN containing polymer solutions were prepared by magnetic stirring at room temperature for 24 h. HNTs were first dispersed in DMF by using a ultrasonicator for 24 h. Then polymer solution and HNTs solutions were combined and mixed by using a ultrasonicator for 24 h.

Electrospinning was carried out by using Inevenso Electrospinning System. Electrospinning conditions are given in Table 1. As given in Table 1, polymer concentration, applied voltage, needle-collector distance and feed rate were kept constant only filler concentration was changed. Filler concentrations were 2 and 4 % wt and feed rates were 0.5 and 1 ml/h.

Table 1. Electrospinning conditions							
Sample #	Polymer Concentration (% wt)	Filler Concentration (% wt)	Voltage/Distance (keV/cm)	Feed Rate (ml/h)			
1	15	2	15/15	0.5			
2	15	2	15/15	1.5			
3	15	4	15/15	0.5			
4	15	4	15/15	1.5			

Surface morphology of the nanocomposite fibers were analyzed by a Scanning Electron Microscopy (SEM) (Inspect S50, FEI Corp.). Fiber diameter was calculated by Image J.

# 3. Results and Discussion

In order to investigate the fiber formation and morphology, SEM analysis was carried out. Fiber morphology and fiber diameter distributions of the samples are shown in Fig. 1-4. Spinning conditions and average fiber diameters are given in Table 2. All samples showed homogeneous fiber formation and random orientation was achieved for all samples.

The International Conference

of Materials and Engineering Technology

Fig. 1 and 2 are given in order to compare the feed rate at low filler concentration. As obvious from the images by increasing the feed rate, fiber diameter increased. While 0.5 ml/h feed rate resulted in average fiber diameter around 305 nm, fiber diameter increased to 347 nm for 1.5 ml/h feed rate. Approximately 13.8 % increase in fiber diameter was obtained.

Fig. 3 and 4 are given in order to compare the feed rate at higher filler concentration. Parallel with the previous results, fiber diameter increased by increasing the feed rate. While 0.5 ml/h feed rate resulted in average fiber diameter around 361 nm, fiber diameter increased to 506 nm for 1.5 ml/h feed rate. Approximately 40.2 % increase in fiber diameter was obtained. As a result, regardless of the filler concentration, fiber diameter increased when feed rate was increased. This was probably caused by the increased volume of the spinning material between needle and collector. Since amount of the material was higher at higher feed rate, the net force on unit volume of spinning material decreased. As a result of this jet stretching decreased and fiber diameter increased [10].

Sample #	Filler Concentration (% wt)	Feed Rate (ml/h)	Average Fiber Diameter (nm)	Increase in Fiber Diameter (%)
1	2	0.5	305	-
2	2	1.5	347	13.8 (Compared with sample 1)
3	4	0.5	361	18,4 (Compared with sample 1)
4	4	1.5	506	40.2 (Compared with sample 3) 45.8 (Compared with sample 2)

Table 2. Electrospinning conditions and avarage fiber diameter



Figure 1. SEM image and fiber diameter distribution of 2% wt HNT/PAN nanocpmposite fibers feed rate: 0.5 ml/h



Figure 2. SEM image and fiber diameter distribution of 2% wt HNT/PAN nanocpmposite fibers feed rate: 1.5 ml/h

In addition to feed rate, effect of filler concentration was also investigated. As given in Table 2, at the same feed rate, higher filler concentration led to higher fiber diameter and that was assumed to be caused by higher solution viscosity. At higher filler concentrations, viscosity of the spinning solution increases and jet stretching decreases and that results in larger fiber diameter [11].



Figure 3. SEM image and fiber diameter distribution of 4% wt HNT/PAN nanocpmposite fibers feed rate: 0.5 ml/h



Figure 4. SEM image and fiber diameter distribution of 4% wt HNT/PAN nanocpmposite fibers feed rate: 1.5 ml/h

**4. Conclusions:** Electrospun HNT/PAN nanocomposite fibers were fabricated under various conditions successfully. Regardless of the condition, all samples showed a homogeneous, random fiber morphology. Depending on the composition of the spinning solution and processing conditions, fiber diameter and porosity were found to be changed. Increased feed rate and filler concentration led to increase in fiber diameter.

Acknowledgments: We would like thank to Esan for halloysite nanotubes.

#### **References:**

1. Wu, Q.-Y., et al., *Interactions between polyacrylonitrile and solvents: density functional theory study and two-dimensional infrared correlation analysis.* The Journal of Physical Chemistry B, 2012. **116**(28): p. 8321-8330.



Bunsell, A.R., Handbook of properties of textile and technical fibres. 2018: Woodhead 2. Publishing.

- 3. Lewin, M., Handbook of fiber chemistry. 2006: Crc press.
- 4. Doshi, J. and D.H. Reneker, *Electrospinning process and applications of electrospun fibers*. Journal of electrostatics, 1995. 35(2-3): p. 151-160.
- 5. Ramakrishna, S., An introduction to electrospinning and nanofibers. 2005: World Scientific.
- Liu, M., et al., Recent advance in research on halloysite nanotubes-polymer 6. nanocomposite. Progress in polymer science, 2014. 39(8): p. 1498-1525.
- Joussein, E., et al., *Halloysite clay minerals-a review*. 2005, De Gruyter. 7.
- Maksimovic, Z. and J. White. Infrared study of chromium-bearing halloysites. in Proc. Int. 8. Clay Conf. Madrid. 1973.
- Saif, M.J., H.M. Asif, and M. Naveed, PROPERTIES AND MODIFICATION METHODS 9. OF HALLOYSITE NANOTUBES: A STATE-OF-THE-ART REVIEW. Journal of the Chilean Chemical Society, 2018. 63(3): p. 4109-4125.
- Zafarulla Khan, F.K., et al., Morphology, mechanical properties and surface 10. characteristics of electrospun polyacrylonitrile (PAN) nanofiber mats. IJAENT, 2015. **2**(3): p. 15-22.
- Makaremi, M., R.T. De Silva, and P. Pasbakhsh, Electrospun nanofibrous membranes of 11. polyacrylonitrile/halloysite with superior water filtration ability. The Journal of Physical Chemistry C, 2015. 119(14): p. 7949-7958.

# EFFECTS OF SOLVENT RATIO ON ELECTRICAL AND MORPHOLOGICAL PROPERTIES OF CARBON NANOFIBER FILLED POLYMER NANOCOMPOSITES

# AYSE TURGUT<sup>1,2</sup>, OZAN TOPRAKCI<sup>1,2</sup>, HATICE AYLIN KARAHAN TOPRAKCI<sup>1,2</sup>,

Corresponding Author: aylin.toprakci@yalova.edu.tr

<sup>1</sup>Yalova University, Engineering Faculty, Polymer Engineering Department, Yalova, TURKEY. <sup>2</sup>Yalova University, Institute of Science, Yalova, TURKEY.

#### Abstract

Melt processing and solution casting are the most common conventional techniques in the polymer industry for the production of composites that have thermoplastic character. Thermoplastic elastomers (TPEs) consist of thermoplastic (hard) and rubbery (soft) phases and both phases provide different properties. Thermoplastic phase controls the melt processing and mechanical deformation behavior of the material. On the other hand rubbery phase controls the elasticity of polymeric material. In a melting process the polymer is heated under pressure and shear to be melted. However, polymers are dissolved in a suitable solvent system in the solution casting process. Process conditions are crucial for both processing is the most common way of processing of TPEs, in the case of nanocomposite fabrication, melt processing may not be enough to obtain homogeneous filler dispersion. In this study carbon nanofiber filled nanocomposites were prepared by the combination of solution casting and melt processing; electrical and morphological properties of the nanocomposites were examined.

**Keywords:** Thermoplastic elastomers, Solvent ratio, Electrical conductivity, Solvent casting, Melt processing

#### **1.Introduction**

Melt and solution processing are two basic techniques in the polymer processing. Polymers can be classified depending on their thermal processing characteristics such as thermoplastics and thermosets. Melt processing is basically melting of the solid polymeric raw material at elevated temperatures under pressure and shear. Injection molding, extrusion, blow molding, compression molding, transfer molding, calendaring and thermoforming are the most commonly used processing in the polymer industry. On the other hand, for the solution casting, polymer must be dissolved in the proper solvent system. Almost every thermoplastic polymer has a specific solvent system that can dissolve in it. Dissolution of the polymer starts with the diffusion of solvent molecules into the polymer chains and finishes with disentanglement of polymer chains in the solvent. After complete dissolution, polymer solution is cast onto a plate and solidify after evaporation of the solvent [1-3].

Thermoplastic elastomers (TPEs) are the polymers that can be processed by both melt and solution based technics. TPEs are block copolymers that consist of at least two different monomer units with having rubbery (soft) and thermoplastic (hard) phases. Soft segments are responsible for the elasticity of the material and have high amount of chain mobility. On the other hand, hard segments represent the thermoplastic properties and form physical cross-links for soft segments. Hard segments also control mechanical deformation resistance of the material under deformation. Chemical structure of the monomer units, block ratio, length of segments have various effects on physical, mechanical, morphological and electrical properties of the TPEs [3, 4].

Having high deformation rates, high resilience and low creep performance make TPEs very crucial materials to obtain flexible conductive polymer composites. Most of the commercial polymers are electrical insulators and conductive fillers can be used to change the electrical properties of the polymers. Vapor grown carbon nanofibers (VGCNFs) are electrically conductive nanofillers that have a high aspect ratio, high thermal conductivity and good mechanical properties [5, 6].

Processing conditions and material properties have significant effects on final performance of the products and repeatability of the process. Mixing and solidification conditions, temperature, pressure, shear, residence time, cycle time, polymer molecular weight, solvent type, solvent ratio, filler properties and filler concentration are critical parameters [7-9]. In terms of conductive polymer composites, polymer, filler and solvent properties are the three basic components. In this study, the effect of solvent ratio in polymer solution as a function of filler concentration was studied and electrical and morphological properties of composites were examined. On this purpose, two polymer solutions with different polymer to solvent ratios were prepared. In the composite production step, combination of solution casting and melt processing techniques were used.

#### 2. Materials and Methods

Styrene Ethylene Butylene Styrene (SEBS) block copolymer (linear) with S/EB ratio of 13/87 (Kraton G1657) was used. Tolune (Merck, Germany) was used a solvent. VGCNFs were purchased from Sigma–Aldrich, USA. Average diameter of VGCNFs is 130 nm and length of the fibers changes from 20 to 200  $\mu$ m.

VGCNFs filled nanocomposites were fabricated in 3 steps. In the first step, two SEBS solutions were prepared. Polymer and toluene were mixed in the ratio of 1:2 and 1:6, respectively. Complete dissolution was achieved by a magnetic stirrer after 24 hours of mixing. In the second step relevant % wt. of nanofiller was mixed with proper amount of polymer solution by a planetary high shear mixer (Kurabo-Mazerustar-KK250, Japan) for 90 sec at 1600 rpm. Mixing stages have very critical effects on electrical and morphological properties of nanocomposites. After mixing process, all the mixtures were poured into the petri dishes and placed in a vacuum oven at 50 °C for 12 hours. In the last step, solidified nanocomposite films were compression molded into 0.7-0.9 mm thick films by using a laboratory type hot press at 180 °C for 10 min. under 0,3 MPa of pressure.

Morphology of VGCNF and nanocomposites were by scanning electron microscope (FESEM) (5 kV, METU Central Research Laboratory) without any coating on the surface.

The volume resistivity of each VGCNFs containing composites were determined by Keithley Model 6517B electrometer and Model 8009 resistivity chamber in accordance with the ASTM D-257 standard. Before conducting the electrical measurements, specimen thicknesses were measured



als and Engineering Technology

#### 3. Results and Discussion

Morphological properties of the composites were characterized by using Scanning Electron Microscope (SEM). It is obviously seen from SEM images that, solvent ratio is very critical in terms of composite morphology. In the case of low solvent ratio, dispersion of VGCNFs in the polymeric matrix was limited and agglomerate formation could be seen as given in Fig. 1a (right top side). On the other hand, VGCNFs dispersed homogeneously into polymer matrix when high amount of solvent used as in Fig.1b. This behavior also affected electrical properties and mechanical properties of the nanocomposites.



(a) Polymer: Solvent = 1:2



(b) Polymer: Solvent = 1:6

**Figure 1.** SEM images of 5 wt. % VGCNF filled SEBS composites at different solvent ratios. Polymer: Solvent (a) 1:2 and (b) 1:6

In order to detect the effects of solvent ratio on electrical properties, volume resistivity of the composites were measured. As seen from Table 1, solvent ratio was found significant in terms of electrical resistivity. Regardless of the polymer:solvent ratio, two sample sets showed classical percolation behavior. However, sample set with the 1:2 polymer:solvent ratio showed the percolation concentration at lower concentration region when compared with the other set (1:6 polymer:solvent ratio). While the sample with 1 wt.% VGCNFs filled composite (1:2 polymer:solvent ratio) showed eight orders of magnitude decrease in volume resistivity; the other set showed two orders of magnitude change for the same filler concentration. As previously showed in the morphology section, this was caused by the filler dispersion.

Filler Concentration (% wt)	Volume Resistivity (Ωcm) (Polymer:Solvent-1:2)	Volume Resistivity (Ωcm) (Polymer:Solvent-1:6)
0	7,82E+16	5,37E+16
1	2,98E+08	6,44E+14
3	2,23E+07	1,21E+11
5	3,79E+06	5,89E+06

**Table 1.** Volume Resistivity of Composites as a Function of Filler Concentration

#### 4. Conclusions

Conductive flexible polymer nanocomposites were fabricated as a function of filler concentration. In the fabrication process two different solvent ratio was used to observe the effects of solvent ratio

Technology



solvent ratio was found significant in terms of morphological and electrical properties.

#### Acknowledgments

This work was supported by Yalova University, Project No. 2018/YL/0006, Yalova University, Project No. 2018/YL/0019 and TUBITAK 2210-C National Graduate Scholarship Program in High Priority Technological Areas (TUBITAK 2210-C Öncelikli Alanlara Yönelik Yurt İçi Yüksek Lisans Burs Programı)

### References

- 1. Miller-Chou, B.A. and J.L. Koenig, A review of polymer dissolution. Progress in Polymer Science, 2003. 28(8): p. 1223-1270.
- 2. El-Sonbati, A., Thermoplastic elastomers. 2012: BoD-Books on Demand.
- 3. Fried, J.R., Polymer science and technology. 2014: Pearson Education.
- 4. Grady, B., S. Cooper, and C. Robertson, Chapter 13-Thermoplastic Elastomers. The Science and Technology of Rubber (Fourth Edition), Academic Press, Boston, 2013: p. 591-652.
- Al-Saleh, M.H. and U. Sundararaj, A review of vapor grown carbon nanofiber/polymer 5. conductive composites. Carbon, 2009. 47(1): p. 2-22.
- 6. Holden, G., Thermoplastic elastomers, in Applied Plastics Engineering Handbook. 2011, Elsevier. p. 77-91.
- 7. Modesti, M., S. Besco, and A. Lorenzetti, Effect of processing conditions on the morphology and properties of polymer nanocomposites. Optimization of polymer nanocomposites properties", ed. by V. Mittal, Publisher: WILEY-VCH Weinheim, 2010: p. 369-405.
- 8. Das, N., T. Chaki, and D. Khastgir, Effect of processing parameters, applied pressure and temperature on the electrical resistivity of rubber-based conductive composites. Carbon, 2002. **40**(6): p. 807-816.
- 9. Hu, N., et al., Effect of fabrication process on electrical properties of polymer/multi-wall carbon nanotube nanocomposites. Composites Part A: Applied Science and Manufacturing, 2008. **39**(5): p. 893-903.



of Materials and Engineering Technology

# HATICE AYLIN KARAHAN TOPRAKCI\*1,2, OZAN TOPRAKCI<sup>1,2</sup>

Corresponding Author: aylin.toprakci@yalova.edu.tr

<sup>1</sup>Yalova University, Engineering Faculty, Polymer Engineering Department, Yalova, TURKEY. <sup>2</sup>Yalova University, Institute of Science, Yalova, TURKEY.

#### Abstract

The aim of this study was to investigate the effects of processing conditions and spinning solution concentration on the electrospinning process of polyvinylidene fluoride and nanofiber morphology. In order to do this, parameters were changed during the electrospinning process.

**Key words:** Electrospinning, nanofibers, Polyvinylidene fluoride (PVdF) nanofibers, Nanowebs, Polymeric fiber

### 4. Introduction

Polyvinylidene fluoride (PVdF) is a unique polymer because of its superior properties including low weight, good mechanical properties (high strength, toughness, abrasion resistance); good thermal stability, low thermal and electrical conductivity, high chemical corrosion and UV resistance, good barrier properties to most gases and liquids. It can be used for wide range of applications from piping to membrane technology and to secondary batteries in various forms. Although it can be processed by various methods based on melt or solution based processes such as extrusion, injection molding, phase inversion; in order to obtain nanomaterials electrospinning can be used [1-5].

Electrospinning is a very common method used for fabrication of nanofibers from polymer solution under electrical force. Since electrospinning system has many components such as voltage source, feeding pump, syringe, collector and polymer solution; various fibers can be obtained. In otherwords fiber morphology is tunable depending on the processing conditions [6, 7].

In this study, PVdF fibers were fabricated by using electro-spinning method. Various spinning solutions were prepared in order to see their effects on fiber morphology. The morphology of the nanocomposite fibers was analyzed by scanning electron microscopy. Processing conditions and spinning solution concentration were found to be significant in terms of fiber morphology.

#### 5. Materials and Methods

Polyvinylidene fluoride (PVdF) with the molecular weight of 275000 g/mol was purchased from Sigma Aldrich. Acetone and dimethyl formamide (DMF) were used as solvents were purchased from Merck. All chemicals were used without further purification.



PVdF was used after the drying in a vacuum oven at 60 °C for 24h. Spinning solutions were prepared at three concentrations as 10 wt %, 15 wt % and 20 wt % by magnetic stirring at 70 °C temperature for 24 h. Acetone/DMF ratio was determined as 60:40 v:v.

Electrospinning was carried out by using Inevenso Electrospinning System (Fig. 1). Electrospinning conditions are given in Table 1. As given in Table 1, applied voltage, needle-collector distance, were kept constant; polymer concentration and feed rate were changed.



Figure 1. Electrospinning system

Sample #	Polymer Concentration (% wt)	Feed Rate (ml/h)	Voltage/Distance (kV/cm)
1	10	0.26	15 kV/15 cm
2	10	0.4	15 kV/15 cm
3	10	0.52	15 kV/15 cm
4	15	0.26	15 kV/15 cm
5	15	0.4	15 kV/15 cm
6	15	0.52	15 kV/15 cm
7	20	0.26	15 kV/15 cm
8	20	0.4	15 kV/15 cm
9	20	0.52	15 kV/15 cm

714

Surface morphology of the nanocomposite fibers were analyzed by a Scanning Electron Microscopy (SEM JEOL JSM-6400) Fiber diameter was calculated by Image J.

#### 6. Results and Discussion

In order to investigate the fiber formation and morphology, SEM analysis was carried out. Fiber morphology and fiber diameter distributions of the samples are shown in Fig. 2-5. Spinning conditions and average fiber diameters are given in Table 2. As can be seen from SEM images and fiber diameter distribution graphs (Fig. 5), all samples showed random orientation with different fiber morphology.

As expected higher polymer concentration led to higher fiber diameter. That was caused by the higher solution viscosity value obtained at higher polymer concentration. At higher polymer concentrations, viscosity of the spinning solution increased, jet stretching decreased and that resulted in larger fiber diameter. While 10% PVdF showed average fiber diameter around 128 nm, 15% and 20% PVdF containing solutions had fiber diameter around respectively. In addition to that the fibers obtained from the solution with 20 % wt PVdF showed the widest fiber distribution. This is probably caused by the clogging of the needle. As previously mentioned PVdF solution was prepared at 70 C. Even we did not observe any clogging for 10 or 15% PVdF containing spinning solutions, 20% wt PVdF containing sample clogged the needle after 30 minutes. Since feeding was not homogeneous during that time, we obtained fibers from nanometers to micrometers. On the other hand, bead formation was observed for 10 % wt PVdF containing spinning solution. This was caused by lower viscosity.



Figure 2. SEM image of 20% wt PVdF containing spinning solution (Feed rate: 0.4 mL/saat, 15 kV/15 cm)

Technology



Figure 3. SEM image of 15% wt PVdF containing spinning solution (Feed rate: 0.4 mL/saat, 15 kV/15 cm)



Figure 4. SEM image of 10% wt PVdF containing spinning solution (Feed rate: 0.4 mL/saat, 15 kV/15 cm)



Figure 5. Fiber diameter distrubition for %20, %15 and %10 PVdF (Feed rate: 0.4 mL/saat, 15 kV/15 cm)

The International Content           Table 2. Electrospinning conditions and avarage fiber diameter						
Sample #	Filler Concentration (% wt)	Feed Rate (ml/h)	Average Fiber Diameter (nm)			
2	10	0.4	128			
5	15	0.4	509			
8	20	0.4	1259			

and Engineering Technology

**4. Conclusions:** Electrospun PVdF fibers were were fabricated under various conditions successfully. Regardless of the condition, all samples showed random fiber morphology. Depending on the composition of the spinning solution and processing conditions, fiber morphology, diameter and porosity were found to be changed. Increased polymer concentration led to increase in fiber diameter.

**Acknowledgments:** This project was founded by Yalova University. BAP (Scientific Research Project) Project No: 2015/BAP/101, Funtional Nanofiber Production, (Fonksiyonel Nanolif Üretimi)

### **References:**

- 1. Kritzer, P. and J.A. Cook, *Nonwovens as separators for alkaline batteries an overview*. Journal of the Electrochemical Society, 2007. **154**(5): p. A481-A494.
- 2. Costa, C.M., M.M. Silva, and S. Lanceros-Mendez, *Battery separators based on vinylidene fluoride (VDF) polymers and copolymers for lithium ion battery applications*. Rsc Advances, 2013. **3**(29): p. 11404-11417.
- 3. Saunier, J., et al., *Thin and flexible lithium-ion batteries: investigation of polymer electrolytes.* Journal of power sources, 2003. **119**: p. 454-459.
- 4. Hwang, K., B. Kwon, and H. Byun, *Preparation of PVdF nanofiber membranes by electrospinning and their use as secondary battery separators.* Journal of membrane science, 2011. **378**(1-2): p. 111-116.
- 5. Huang, F., et al., *Effect of temperature on structure, morphology and crystallinity of PVDF nanofibers via electrospinning.* e-Polymers, 2008. **8**(1).
- 6. Ding, B. and J. Yu, *Electrospun nanofibers for energy and environmental applications*. 2014: Springer.
- 7. Homaeigohar, S. and M. Elbahri, *Nanocomposite electrospun nanofiber membranes for environmental remediation*. Materials, 2014. **7**(2): p. 1017-1045.



of Materials and Engineering Technology

# AYSE TURGUT<sup>1,2</sup>, OZAN TOPRAKCI<sup>1,2</sup>, HUSEYIN CAGDAS ASLAN<sup>1,2</sup>, HATICE AYLIN KARAHAN TOPRAKCI<sup>1,2</sup>,

Corresponding Author: aylin.toprakci@yalova.edu.tr

<sup>1</sup> Affiliation: Yalova University, Engineering Faculty, Polymer Engineering Department, Yalova, TURKEY.
<sup>2</sup> Affiliation: Yalova University, Institute of Science, Yalova, TURKEY.

#### Abstract

Polymeric blends consist of at least two different homopolymers or copolymers. Blending is significant in terms of obtaining new materials with desired properties. Polypropylene is a stiff thermoplastic polymer at room temperature that has high elastic modulus, tensile strength and relatively low tensile strain and impact strength. On the other hand, to optimize its mechanical and impact properties and to obtain tunable properties various elastomers can be used and thermoplastic elastomers (TPE) are one of the good candidates. Since TPEs contain soft and rigid segments in their structure, properties of PP can be modified with the appropriate amount of TPE. In this study polypropylene (PP) and styrene–ethylene butylene–styrene block (SEBS) copolymer were blended at different ratios by using a traditional melt compounding system and the mechanical properties of the blends were investigated as a function of blend ratio.

**Keywords:** Polymer blends, Thermoplastic, Thermoplastic elastomer, Extrusion process, Impact strength

#### **1. Introduction**

Polymer blends consist of at least two different homopolymers or copolymers. Blending is one of the easiest ways to obtain new materials with desired properties. These new materials possess the properties of components depending on the ratios in the polymeric matrix. Polymeric blends are important for the production of new and cost-effective products. By blending, many important properties can be modified such as mechanical, thermal, electrical and optical properties, toughness and processability. In polymer industry in addition to blending, different techniques are used to get desired material properties such as modification of polymers by using random copolymerization, graft and block copolymerization. In all these methods, blending is found to be more feasible and economic. Polymer blending can be carried out by solution or melt processing. However in industry, traditional polymer blending is carried out by extruders. Polymers are generally dry mixed and fed into the extruder; melting and mixing are carried out simultaneously in the barrel [1-4].

In order to fabricate polymeric blends from melt, extrusion systems are used. Extrusion is the basic process for melt blending in which polymers are melted and blended. Basically by the effect of



The International Conference of Materials and Engineering Technology thermal energy, shear and pressure; solid raw materials turn to viscous fluid at elevated temperatures. This system is used in fabrication of electronics, household devices, automotive, packaging and medical products. Literally, a basic extrusion system consists of a feeding hopper that provide polymeric raw material pass into the extrusion machine, a barrel that have thermocouples around it, a dynamic screw that have special design depending on aimed product and causes melting of raw material into barrel and transformation of it through the die at elevated temperatures under pressure and high shear, a die that give the final shape of molten product and lastly, screw driving unit for the rotation movement of screw into the barrel. In addition to them, degassing unit has very crucial importance on appearance, physical and mechanical properties of the finished products. It provides the removal of low molecular weight monomers and moisture from the molten polymer [5-8].

In the extrusion system, single or twin screws can be used depending on the aim of the process. Single screw extruders mostly used for the melting of semi finished or finished products to give final shape such as films, sheets, pipes and profiles. On the other hand, twin screw extruders that might have co-rotating or counter- rotating screws are chosen for the mixing, compounding process, dispersion of components with each other and chemical modification. Ratio of screw length to screw diameter (L/D) is a parameter that directly determines the capacity of the extrusion system. The tunable process parameters are feeding rate (g/min), screw speed (rpm), temperature values in the barrel (°C) [5-11].

Many commodity thermoplastics can be blended with elastomers to enhance the impact properties. PP is a stiff thermoplastic material that has high mechanical properties with low tensile strain and impact strength at room temperature. For the optimization of the impact properties of PP, elastomeric structures can be used. SEBS is a thermoplastic elastomer that has both elastomeric and thermoplastic domains in its structure and due to the elastomeric domains SEBS polymer has high flexibility and also high impact resistance. Depending on the ratio of the components tunable mechanical and impact properties can be obtained for SEBS/PP blends [3, 12-16]. In this study, parallel with this purpose, effects of SEBS/PP ratio on mechanical properties of the blends were investigated.

#### 2. Materials and Methods

Polypropylene homopolymer ISPLEN PP070G2M with the melt flow index of 12 g/10 min. was supplied by Repsol Chemicals. SEBS block copolymers with S/EB ratio of 20/80 and melt flow index of 14 g/10 min. was purchased from Kraton.

Both polymers were dried at 80°C in a vacuum oven for 2 hours and following that SEBS and PP were dry mixed in a container at different ratios. Blend ratios of SEBS/PP were 0/100, 25/75, 50/50, 75/25, 100/0 wt %. Dried mixed samples were fed into the co-rotating twin screw extruder (GM, Gülnar Makina with L/D = 25 and D= 16 mm) with a screw speed of 300 rpm and barrel temperature profile was set as 50, 170, 180, 190 °C from feeding to die zone respectively (Fig. 1).



Figure 1. Extrusion system with barrel temperatures

In order to test the mechanical properties and film formation properties of the blends, filaments were cut into small pieces and compression-molded by using hot press at 180 °C for 10 min. under pressure. Samples can be seen in Fig. 2.



Figure 2. Photos of SEBS, PP and SEBS/PP blend films

Mechanical characterization of the blends was done by using on a universal load frame (Devotrans, DVT GPU/RD). For each blend type, 3 specimens were tested with the dimensions of 25 mm x 5mm (length x width).

# 3. Results and Discussion

Mechanical properties of blends vary depending on the content of thermoplastic elastomer into the structure. As seen from Table 1, with the increase in PP content elastic modulus and tensile strength increased. On the other hand, tensile strain decreased gradually by increasing the PP ratio. These findings are parallel with the literature [17, 18]. PP is a highly rigid polymer with relatively high modulus and tensile strength when compared with SEBS [12-14]. As given in Table 1 tensile strength of PP is 3 times higher than SEBS. In the case of elastic modulus the difference is more



drastic. PP showed a value around 55 times higher than SEBS. As a rule of thumb, the mixture represents the properties of the components and our results confirm that. As can be seen from results for the optimization of the mechanical properties of PP, SEBS can be used. Since SEBS is a thermoplastic elastomer with both elastomeric and thermoplastic domains, it lowers the elastic modulus and tensile strength of PP after blending. On the contrary to elastic modulus and tensile strength, tensile strain was increased by the addition of SEBS. That is probably caused by the elastomeric nature of the SEBS. Since it can be stretched to very high levels of strain SEBS/PP blends showed higher level of tensile strength compared to PP. All those properties will be useful in terms of modification of impact properties of the PP and by using SEBS/PP polymers at different ratios tunable properties can be obtained.

180	Table 1. Mechanical Properties of the Blends							
Samples	Elastic Modulus (MPa)	Tensile Strength (MPa)	Tensile Strain (%)					
100/0 SEBS/PP	5,90	7,52	9,15					
75/25 SEBS/PP	16,13	9,49	7,99					
50/50 SEBS/PP	83,59	12,91	7,17					
25/75 SEBS/PP	232,26	16,11	5,50					
0/100 SEBS/PP	325,28	21,50	0,16					

# 4. Conclusions

In this study in order to obtain tunable mechanical properties and also to enhance the impact resistance of PP, SEBS thermoplastic elastomer was added into PP structure at various levels. During production steps, melt blending and compression molding processes were used and mechanical characterization was carried out. As a result of the study, by increasing the SEBS ratio, elastic modulus and tensile strength were decreased due to elastomeric character of SEBS. Parallel with that tensile strain was increased by increasing the SEBS ratio.

# References

- 1. Paul, D. and J. Barlow. *BRIEF REVIEW OF POLYMER BLEND TECHNOLOGY*. in *Multiphase Polym, based on a Symp sponsored by Div of Polym Chem at Am Chem Soc* 175th Meet. 1979.
- 2. Utracki, L., *Polymer blends: fundamentals*, in *Polypropylene*. 1999, Springer. p. 601-605.
- 3. Parameswaranpillai, J., S. Thomas, and Y. Grohens, *Polymer blends: state of the art, new challenges, and opportunities.* Characterization of polymer blends: miscibility, morphology and interfaces. Germany: Wiley-VCH Verlag GmbH & Co. KGaA-Weinheim, 2015: p. 1-6.



MET

- 5. Lafleur, P.G. and B. Vergnes, Polymer extrusion. 2014: John Wiley & Sons.
- 6. Chokshi, R. and H. Zia, Hot-melt extrusion technique: a review. Iranian Journal of Pharmaceutical Research, 2010: p. 3-16.
- 7. Abeykoon, C., et al., A review and evaluation of melt temperature sensors for polymer extrusion. Sensors and actuators A: Physical, 2012. 182: p. 16-27.
- 8. Rauwendaal, C., Polymer extrusion. 2014: Carl Hanser Verlag GmbH Co KG.
- 9. Gadekar, M.S.S., J.G. Khan, and R. Dalu, Analysis of Process Parameters for Optimization of Plastic Extrusion in Pipe Manufacturing.
- Yeh, A.I. and Y.M. Jaw, *Effects of feed rate and screw speed on operating characteristics* 10. and extrudate properties during single-screw extrusion cooking of rice flour. Cereal chemistry, 1999. 76(2): p. 236-242.
- 11. Bhattacharya, S. and G.S. CHOUDHURY, Twin-screw extrusion of rice flour: Effect of extruder length-to-diameter ratio and barrel temperature on extrusion parameters and product characteristics. Journal of Food Processing and Preservation, 1994. 18(5): p. 389-406.
- 12. Gupta, A. and S. Purwar, Dynamic mechanical and impact properties of PP/SEBS blend. Journal of applied polymer science, 1986. 31(2): p. 535-551.
- 13. Maddah, H.A., Polypropylene as a promising plastic: A review. Am. J. Polym. Sci, 2016. **6**(1): p. 1-11.
- 14. Akkapeddi, M., Commercial polymer blends. Polymer blends handbook, 2003: p. 1023-1115.
- 15. Rabinovitch, E.B., J.W. Summers, and G. Smith, Impact modification of polypropylene. Journal of vinyl and additive technology, 2003. 9(2): p. 90-95.
- 16. Ignaczak, W., et al., Mechanical and thermal properties of PP/PBT blends compatibilized with triblock thermoplastic elastomer. Polish Journal of Chemical Technology, 2015. 17(3): p. 78-83.
- 17. Ismail, H., Thermoplastic elastomers based on polypropylene/natural rubber and polypropylene/recycle rubber blends. Polymer Testing, 2002. 21(4): p. 389-395.
- 18. Mohamad. N.. al., Mechanical and morphological et properties of polypropylene/epoxidized natural rubber blends at various mixing ratio. Procedia Engineering, 2013. 68: p. 439-445.

# DESIGN AND PERFORMANCE EVALUATION OF A SELF-COOLING CONCENTRATING PHOTOVOLTAICS SYSTEM

# Shoukat Alim Khan<sup>1,2</sup>, Yusuf Bicer<sup>1</sup>, Sami G. Al-Ghamdi<sup>1</sup>, Muammer Koç<sup>1</sup>

of Materials and Engineering Technology

<sup>1</sup> Division of Sustainable Development (DSD), Hamad Bin Khalifa University (HBKU), Qatar Foundation (QF), Doha, Qatar

<sup>2</sup> Faculty of Mechanical Engineering, The Ghulam Ishaq Khan Institute of Engineering Sciences and Technology, Topi, Swabi, Khyber Pakhtunkhwa 23640, Pakistan

#### Abstract

Effective heat dissipation form high concentrating photovoltaics (CPV) is a challenge to overcome, for its successful implementation in the market. This study designed a self-cooling thermal management system for CPVs and thermodynamically analyze its performance. The thermal energy from one row of CPV (CPV 1) is utilized in the absorption cooling (AbC) system as an input to produce cooling, which is used to cool down another row of CPV (CPV 2). In the proposed idea, both CPVs work simultaneously under same solar energy conditions and physically next to each other, hence, the demand of cooling energy of CPV 2 and supply of thermal energy from CPV 1 are continuously synchronized. Therefore, both rows of CPV are combined and treated as an overall selfcooling CPV system. The results of this investigation show that, under the designed conditions, first CPV system, having an installed capacity of 66.4 kW and operating temperature of 375.3 K with 35.4% electrical efficiency, has the ability to cool down the second CPV system with an installed capacity of 50.6 kW and operating temperature of 335.8 K with 37.4% electrical efficiency. The installed capacity of the second CPV system entirely depends upon the cooling capacity that can be provided by the first CPV system. A parametric study is made to analyze performance of the overall system for various working conditions and installed capacity. In addition, the designed system is analyzed for two different types of high-performance multi- junction solar cells and three different types of coolants: water, ethanol, and n-pentane. The optimum results are reported for the best working fluid configuration and working temperatures in the first and the second CPV systems, in order to achieve maximum concentration ratio limit and highest efficiency.

Keywords: Self-cooling CPV, concentrating photovoltaics, absorption cooling, solar thermal, exergy, efficiency.

1. Introduction

Solar Photovoltaics (PV) is an efficient technology that contributes the ever-increasing energy demand sustainably and cleanly [1-3]. Recently, new generation high efficiency multijunction solar cells (MJSC) with higher efficiency have been developed. These cells have proven efficiencies of up to 46% at standard testing conditions and up to 40% overall system efficiency [4,5]. The ongoing research on concentrating photovoltaics (CPV) will make them quite competitive by achieving efficiency levels greater than 50%.

CPV systems use optical devices such as mirrors and lenses to concentrate a large amount of solar radiation energy on a small cell area. Hence, decreasing the required cell area and replacing the expensive cell material with lower-priced optical devices, which significantly decreases the overall system cost. The increase in concentration ratio and efficiency significantly reduces the cell cost in CPV systems. However, there are specific challenges associated with this technique, one of the main challenges is the high cooling demand for a given small area rapidly [6,7].

In photovoltaics the incoming absorbed solar irradiation is mainly converted to electrical and heat energy [8,9]. Due to high concentration of light in CPV a high amount of heat is produced in small cell area. This results in an increase in cell temperature, if not dissipated efficiently, which eventually affects the electrical performance of the cell.

The design of a reliable heat dissipation system in a miniaturized component such as electronic cooling and CPV cell cooling has always been challenging. Therefore, an appropriate thermal management system is always an integral part of the successful design and operation of the CPV systems [10,11]. To achieve uniform cooling and higher heat transfer coefficient techniques like microchannel, heat pipes, jet impingement, microchannel-jet impingement, phase change materials andd spreader have been analyzed in the literature [12,13].

This study proposes designs and thermodynamically analyzes an overall self-cooling CPV system, following a previous investigation and its findings where nucleate pool boiling as a thermal management technique for CPV systems was thermodynamically analyzed [14]. This study uses the nucleate pool boiling technique for the extraction of the heat from CPV system. The thermal energy taken from one CPV system (called CPV 1, Figure 1) is used to produce cooling via absorption chiller and this cooling is used for another CPV system (called CPV 2, Figure 1). Hence, each CPV system acts as a cooling source for the other. As both the CPVs operate under same solar energy conditions physically next to each other, the demand for cooling energy of CPV 2 and supply of thermal energy from CPV1 are always synchronized. The specific objectives are:

- Design and thermodynamically analyzed the proposed self-cooling CPV system.
- Analyse performance of the designed system for different ambient conditions.
- To study the effects of different operating parameters of the designed systems on the system performance.
- Analyse the effect of different designed parameters on the performance of overall system.
- To select the optimum working liquid configuration and operating temperature for the best performance of designed system.
  - 2. System Description

The overall self-cooling CPV system composes of two CPVs: CPV 1 and CPV 2, and an absorption cooling unit. Thermal energy is extracted from each CPV by nucleate pool boiling technique. Then these two hot streams are connected using absorption chillers, as shown in Figure 1. The absorption cooling system use thermal energy form CPV 1 to produce cooling. The cooling produce by absorption chiller is used to reduce the working temperature of CPV 2. The complete self-cooling system is composed of:

- (i) CPV 1: Works as a heat source for absorption chiller.
- (ii) NBHT 1: Works to extract heat from CPV 1.





- (iii)
- (iv)



AbC: Uses the heat and converts into the cooling

Figure 1: The schematic diagram of overall self-cooling CPV system.



Table 1 represents the initial steady state designed of the proposed system.

Parameter	Explanation/Value	Reference					
CPV system							
CPV 1	66.4 kW						
CPV1 operating temperature	375.3 K						
CPV 1 area	220 m <sup>2</sup>						
<b>Cell type A:</b> Inverted Metamorphic multijunction (IMM) [15]							
Efficiency	40.8%	[15]					
Thermal coefficient	-0.052 %/K	[15]					
Cell type B: Four	r-junction inverted metamorphic (4J IMM) [16]						
Effeciency	45.3%	[16]					
Thermal coefficient	-0.2%/K	[17]					
Absorption cooling (AbC) system							
Working fluid	LiBr-water	[18]					
Metrological data							
Ambient temperature	301 K	[19]					
Atmospheric pressure	101.3 kPa						
Humidity (relative)	0.5	[19]					
DNI	$0.85 \text{ kW/m}^2$	[20]					
]	Thermal management system	L					
NBHT working fluid of CPV 1	Water						
NBHT working fluid of CPV 2	Water						
Nucleate boiling temperature for CPV 1	373K						
Nucleate boiling temperature for CPV 2	333 K						

**Table 1:** Initial input parameters for the steady-state design.

To satisfy different working conditions and achieve the optimum operating state these parameters are varied in the parametric study. Three different fluids water, ethanol, and n-pentane are comparatively employed as a coolant in NBHT sub-system.

2. System Analysis

Considered CPV with NBHT and AbC system is summarized here, the detail model can be found in the previous publication [14]. The following thermodynamic assumptions are made during this study [21,22]:

- The steady-state operation is considered for the initial design.
- The heat and pressure loss n the pipes are considered negligible in an AbC sub-system.
- The isobaric process is considered in the heat exchanger, generator, evaporator, and NBHT sub-systems.
- The saturated vapor condition at state point 10 and the saturated liquid conditions are considered at state points 1, 4 and 8 of AbC sub-system.
- Chemical reaction in AbC sub-system and hence change in chemical exergy is neglected [23].



For the initial design, performed at the steady-state conditions reported in Table 1, the installed capacity of 66.4 kW of CPV 1 operating at 375.3 K and 35.4% electrical efficiency has the ability to cool down the CPV 2 of 50.6 kW and 37.4% electrical efficiency working at a temperature of 335.8K, Table 2. The installed capacity of the system represents the electrical output of the respective system. Thermodynamic calculations are performed using EES (Engineering Equation Solver) software [24].

The International Conference

aterials and Engineering Technology

Parameter	CPV 1	CPV 2	Overall CPV system
Installation capacity (kW)	66.4	50.6	117
Operating electrical efficiency (%)	35.1	37.5	36.3
Critical heat flux (kW/m <sup>2</sup> )	844.5	426.7	-
Concentration ratio	1678	863	1202

Table 2: Initially designed steady state system.





Figure 2: The exergy efficiencies of major components and whole system

4. Parametric study

The following parametric studies are performed to understand the effects of ambient conditions, cell type, working fluid type, and installation capacity of system and subsystems. Also, parametric study is performed to select the optimal operating temperature of both the CPVs and optimum pair of working fluids, with respect to maximum overall efficiency and concentration ratio.

### **Effects of irradiation level**

In order to ensure the operation of the designed system for different ambient conditions, the effects of irradiation level are also studied by changing DNI values from 0.7 to 1.1 (kW/m<sup>2</sup>), as shown in Figure 3. The increase in irradiation causes in an increase in the incoming energy to the cell and hence decreases the concentration limit of the CPV system. However, the cell temperature remains constant as cell temperature depends mainly on the saturation temperature of the working liquid, the type of liquid, and the surface. The electrical output of the system, represented by overall installed capacity, increases with increase in the input energy such as irradiation level. The performance of the designed system is also verified for cell type B (4J-IMM), as shown in *Figure 4*.

This section aims to select the best working fluid among water, ethanol and n-pentane coolants for the individual NBHT thermal management system. By changing the NBHT temperature from 313K to 393K, the changes are observed in the efficiency and concentration ratio of all three working liquids, as shown in Figure 5. Water and n- pentane show the highest efficiency at low operating temperatures with 36.7 %, followed by ethanol. However, the maximum concentration ratio of the system with water as working fluid is quite high than that of ethanol and n-pentane. Ethanol shows excellent performance in efficiency at higher working temperatures, but the difference in the concentration ratio is quite lower than water. The concentration ratio of water is the highest because of its high critical heat flux, mainly due to its high latent heat value. The water has been selected as the most favorable working fluid concerning the concentration ratio and the efficiency.







**Figure 4:** Effects irradiation on overall installed capacity and system efficiency for selected cell type B. Table 3: The efficiency values of different liquids for same concentration ratio.

	Working fluid	C.R CPV2 900	C.R CPV2 1000	C.R CPV2 1100	C.R CPV2 1200	C.R CPV2 1300
CPV 2 (%)	Water	37.4	37.1	36.8	36.6	36.38
	Ethanol	35.8	35.4	34.9	34.5	Above cell working temperature limit
iciency of	n-pentane	Above cell working temperature	Above cell working temperature	Above cell working temperature	Above cell working temperature	Above cell working temperature
Eff		limit	limit	limit	limit	limit



Figure 5: Comparison of working liquid water, ethanol and n-pentane in NBHT sub-system for thermal management of CPV.

#### 5. Conclusions and Future Recommendations

The overall CPV system is divided into two portions such that a portion of CPV (CPV 1) provides the heat to run AbC sub-system and the produced cooling is used for thermal management of the other CPV system (CPV 2). At designed conditions, 66.4 kW of installed CPV 1 at the operating temperature of 375.3 and operating efficiency of 35.5% can maintain 50.6 kW of CPV 2 at the operating temperature of 335.8 K and operating efficiency of 37.5 %. The whole system works as a self-cooling system where no external energy is needed for the thermal management of the overall system.

# 6. References

1. Lamnatou C, Baig H, Chemisana D, Mallick TK. Environmental assessment of a buildingintegrated linear dielectric-based concentrating photovoltaic according to multiple lifecycle indicators. J Clean Prod 2016.doi:10.1016/j.jclepro.2016.04.094.

The International Conference

aterials and Engineering Technology

- 2. Haiping C, Haowen L, Heng Z, Kai L, Xinxin G, Boran Y. Numerical simulation and experimental analysis of an LCPV / T system under real operating conditions. J Clean Prod 2019;209:903–15. doi:10.1016/j.jclepro.2018.10.256.
- **3.** Al-soud MS, Hrayshat ES. A 50 MW concentrating solar power plant for Jordan. J Clean Prod 2009;17:625–35. doi:10.1016/j.jclepro.2008.11.002.
- **4.** Warmann EC, Atwater HA. Energy production advantage of independent subcell connection for multijunction photovoltaics. Energy Sci Eng 2016;4:235–44. doi:10.1002/ese3.125.
- **5.** Steiner MA, Geisz JF, Friedman DJ, Olavarria WJ, Duda A, Moriarty TE. Temperaturedependent measurements of an Inverted Metamorphic Multijunction (IMM) solar cell 2011:2527–32.
- 6. Zilli BM, Lenz AM, Nelson S, Souza M De, Secco D, Eduardo C, et al. Performance and effect of water- cooling on a microgeneration system of photovoltaic solar energy in Paraná, Brazil. J Clean Prod 2018. doi:10.1016/j.jclepro.2018.04.241.
- 7. Manikandan S, Selvam C, Poddar N, Pranjyal K, Lamba R, Kaushik SC. Thermal Management of Low Concentrated Photovoltaic Module With Phase Change Material. J Clean Prod 2019. doi:10.1016/j.jclepro.2019.02.086.
- **8.** Baljit SSS, Chan H, Sopian K. Review of building integrated applications of photovoltaic and solar thermal systems. J Clean Prod 2016. doi:10.1016/j.jclepro.2016.07.150.
- **9.** Boyaghchi FA, Nazer S. Assessment and optimization of a new sextuple energy system incorporated with concentrated photovoltaic thermal Geothermal using exergy, economic and environmental concepts. J Clean Prod 2017;164:70–84. doi:10.1016/j.jclepro.2017.06.194.
- **10.** Bellos E, Said Z, Tzivanidis C. The use of nanofluids in solar concentrating technologies: a comprehensive review. J Clean Prod 2018. doi:10.1016/j.jclepro.2018.06.048.
- **11.** Chen H, Ji J, Pei G, Yang J, Zhang Y. Experimental and numerical comparative investigation on a concentrating photovoltaic system. J Clean Prod 2017. doi:10.1016/j.jclepro.2017.11.058.
- **12.** Yang K, Zuo C. A novel multi-layer manifold microchannel cooling system for concentrating photovoltaic cells. Energy Convers Manag 2015;89:214–21. doi:10.1016/j.enconman.2014.09.046.
- **13.** Kabeel AE, Abdelgaied M. Performance enhancement of a photovoltaic panel with reflectors and cooling coupled to a solar still with air injection. J Clean Prod 2019. doi:10.1016/j.jclepro.2019.03.199.
- **14.** Khan SA, Bicer Y, Koç M. Design and analysis of a multigeneration system with concentrating photovoltaic thermal (CPV/T) and hydrogen storage. Int J Hydrogen Energy 2018.
- **15.** Steiner M, Siefer G, Schmidt T, Wiesenfarth M, Dimroth F, Bett AW, et al. 43 % Direct Sunlight Conversion Efficiency using 4J Cells with Achromatic Full Glass Lens 2016;080006. doi:10.1063/1.4962104.
- **16.** France RM, Geisz JF, Garcia I, Steiner MA, McMahon WE, Friedman DJ, et al. Design Flexibility of Ultrahigh Efficiency Four-Junction Inverted Metamorphic Solar Cells. IEEE J Photovoltaics 2016;6:578–83. doi:10.1109/JPHOTOV.2015.2505182.
- **17.** Kurtz S, Muller M, Marion B, Mcconnell R, Kimber A. Considerations for How To Rate Cpv 2011:80401.


- **18.** Herold KE, Rademacher R, A.Klein S. Absorption Chillers and Heat Pumps. 2nd ed. New York, NY: CRC Press; 2016.
- **19.** www.timeanddate.com. https://www.timeanddate.com/weather/qatar/doha/climate 2018.
- **20.** Globalsolaratlas:World Bank Group. World Bank Group 2018. http://globalsolaratlas.info/.
- **21.** Bicer Y, Dincer I. Analysis and performance evaluation of a renewable energy based multigeneration system. Energy 2016;94:623–32. doi:10.1016/j.energy.2015.10.142.
- **22.** Zafar S, Dincer I. Energy, exergy and exergoeconomic analyses of a combined renewable energy system for residential applications. Energy Build 2014;71:68–79. doi:10.1016/j.enbuild.2013.12.006.
- **23.** Dincer I, Ratlamwala TAH. Integrated Absorption Refrigeration Systems. Cham: Springer International Publishing; 2016. doi:10.1007/978-3-319-33658-9.
- 24. Klein. EES: Engineering Equation Solver | F-Chart Software : Engineering Software n.d.



## ANTEP FISTIĞI KABUKLARINDAN BİYOKÖMÜR ÜRETİMİ

## Ali KESKİN<sup>1</sup>, Elif ARANCI ÖZTÜRK<sup>2</sup>, Nadir ŞENGÜL<sup>2</sup>, Fırat ELİBOL<sup>2</sup>, Mesut AKTAŞ<sup>2</sup>, Mustafa BOYRAZLI<sup>2</sup>

<sup>1</sup>ETİ KROM A.Ş., AR-GE Merkezi Müdürlüğü, ELAZIĞ <sup>2</sup>Fırat Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği, ELAZIĞ

## ÖZET

Günümüzde dünyanın karşılaştığı enerji problemlerine alternatif olarak enerji kaynaklarından biri olarak biyokütle enerji kaynağı görülmektedir. Piroliz yöntemi, biyokütlenin teknolojiye uygun katı, sıvı ve gaz ürünlere dönüştürülmesini sağlar. Enerji ihtiyacımızın büyük bir bölümünü karşılayan fosil enerji kaynaklarının gelecekte tükenecek olması, yenilenebilir alternatif enerji kaynaklarının yaygın olarak kullanımını gerektirmektedir. Biyokütle enerjisi, yenilenebilir enerji kaynakları potansiyeli açısından çeşitlilik gösteren ülkemiz için, önemli bir seçenek olarak karşımıza çıkmaktadır. Bu çalışmada, Antep fistiği kabuklarının farklı süre ve sıcaklıklarda karbonizasyonu ile biyokömür üretimi amaçlanmıştır. Antep fistiği, ülkemizde ağırlıklı olarak Gaziantep yöresinde, dünyada Yakındoğu, Akdeniz Bölgesi ve Asya' nın batı bölgelerinde yetişmektedir. Deneysel çalışmalar, karbonizasyon işlemleri için özel olarak hazırlanmış vida kapaklı metal pota içerisinde farklı süre ve sıcaklıklarda gerçekleştirilmiştir. Karbon ve kükürt içeriği bakımından en iyi sonuçlar, 800 °C' de 60 dk. işlem gören numunede elde edilmiştir. Bu numunenin %88,75 C, %0,009 S içerdiği ve ısıl değerinin 7273 kal/g olduğu tespit edilmiştir. Karbonizasyon işlemi sonucunda numunelerin %63-84 arasında ağırlık kaybına uğradığı ve ortalama %3,4 kül içerdiği tespit edilmiştir.

Anahtar kelimeler: Karbonizasyon, Atık, Antep fistiği kabuğu, Biyokütle



## 1. Giriş

Enerji ihtiyacımızın büyük bir bölümünü karşılayan fosil enerji kaynaklarının gelecekte tükenecek olması, yenilenebilir alternatif enerji kaynaklarının yaygın olarak kullanımını gerektirmektedir. Yenilenebilir enerji kaynakları; güneş, rüzgâr, hidrolik, jeotermal, dalga ve biyokütle enerji kaynakları şeklinde sıralanabilir. Hızla artan enerji talebini karşılarken, kesikli olmayan ve çevreyle uyumlu enerji kaynaklarına yönelmek de önemlidir. Bu bağlamda, biyokütle enerjisi, yenilenebilir enerji kaynakları potansiyeli açısından çeşitlilik gösteren ülkemiz için, önemli bir seçenek olarak karşımıza çıkmaktadır [1].

The International Conference of Materials and Engineering Technology

Genellikle karbon, hidrojen, oksijen ve azottan oluşan hidrokarbon yapısı, biyokütle olarak adlandırılır. Biyokütle tipine bağlı olarak inorganik madde miktarı %1-15 arasında değişiklik göstermektedir [2].

Çeşitli biyokütle kaynakları ve farklı sistemlerde karbonizasyon ve/veya pirolizi konusunda birçok çalışma literatürde yer almaktadır [1-8].

Antep fistiği ülkemizde en fazla Gaziantep, Kahramanmaraş, Adıyaman, Şanlıurfa, Mardin, Kilis, Diyarbakır ve Siirt' te yetiştirilirken, dünyada ise Yakındoğu, Akdeniz Bölgesi ve Asya' nın batı bölgelerinde yetiştirilmektedir [9].

Antep fistiği ağaçlarından toplanan meyvelerin dışında iki tür kabuk bulunmaktadır. Bunlar kırmızı renkli yumuşak bir dış kabuk ve bunun altında meyveyi koruyan oldukça sert olan bir iç kabuktan oluşmaktadır. Yumuşak dış kabuk meyvenin %18' lik bölümünü oluşturmaktadır ve Türkiye' de yaklaşık yıllık 13.000 ton kırmızı dış kabuk atık olarak ortaya çıkmaktadır [10]. Diğer yandan sert iç kabuk ise meyvenin yaklaşık olarak %45' lik kısmını oluşturmaktadır [11].

TÜİK 2018 verilerine göre (Tablo 1) Türkiye' de toplam Antep fistığı ağaç sayısı 70.087' dir. Bunların 49.558' i meyve vermektedir ve üretilen toplam Antep fistığı ise yaklaşık olarak 240.000 tondur [12].

Bu çalışmada, atıl durumda olan ve genellikle fırınlarda ve sobalarda yakılan Antep fıstığı kabuklarından aktif karbon ve nano karbon yapılar üretebilmek için ilk aşama olan karbonizasyon işlemi ile biyokömür üretimi gerçekleştirilmiştir. Çalışmanın devamında aktif karbon üretimi ve nano karbon tüp üretiminin gerçekleştirilmesi planlanmaktadır.

Agaç sayısı							
Yıllar	Meyve veren	Meyve vermeyen	(Ton)				
2010	29.617	10.562	128.000				
2011	30.868	10.419	112.000				
2012	37.150	12.428	150.000				
2013	38.116	12.006	88.600				
2014	39.330	11.153	80.000				
2015	40.597	11.633	144.000				
2016	42.570	17.193	170.000				
2017	47.766	19.460	78.000				
2018	49.558	20.529	240.000				

Tablo 1. S	Son 8 yıla	ait Antep	fıstığı üretir	n verileri	[12]
------------	------------	-----------	----------------	------------	------



## 2. Materyal Metot

## -Materyal

Deneylerde piyasadan satın alma yoluyla temin edilen ve karbon, kükürt ve kalorifik değeri Tablo 2' de verilen Antep fistığı kabukları (Şekil 1) kullanılmıştır. Karbonizasyon işlemleri için vida kapaklı metal pota (Şekil 2) ve NÜVE/MF207 marka/model kül fırını kullanılmıştır (Şekil 3).

aterials and Engineering Technology

Tablo 2. İşlem görmemiş Antep fıstığı kabuklarının karbon ve kükürt analizi

%C	% S	Kalori(kal/g)
43,5	0,008	4318



Şekil 1. Karbonizasyon işlemleri için kullanılan Antep fıstığı kabukları



Şekil 2. Karbonizasyon işlemi için kullanılan özel hazırlanmış vida kapaklı metal pota





Şekil 3. Karbonizasyon işlemi için kullanılan kül fırını

## -Metot

İşlem görmemiş Antep fistiği kabukları, karbon, kükürt, kalorifik değer ve kül analizleri yapıldıktan sonra (Tablo 1), Şekil 2' de ebatları verilen kapaklı bir pota içerisine konularak Şekil 3' te görülen kül firininda 400, 600, 700, 800 ve 900 °C sıcaklıklarda, 60, 120, 360 ve 1440 dk. süre ile karbonizasyon işlemine tabi tutulmuştur. Karbonizasyon işlemi öncesi ve sonrasında numunelerin karbon kükürt analizleri Eti Krom Elazığ Ferrokrom Tesislerinde bulunan LECO CS 230 marka analiz cihazında, kalorifik değeri ise LECO AC 500 marka kalorimetre cihazında gerçekleştirildi. Karbonizasyon işlemi öncesi ve sonrası numuneler hassas terazi (Radwag AS 220/X) yardımıyla tartılarak ağırlık kayıpları hesaplanmış ve Tablo 3' te verilmiştir.

**Tablo 3.** Antep fıstığı kabuklarının sıcaklık ve süreye bağlı olarak hesaplanan ağırlıkkayıpları

		400 °C	600 °C	800 °C	900 °C
60 dk.	İlk Ağırlık (g)	36,7381	38,1791	37,0377	34,668
	Son Ağırlık (g)	13,2652	9,7463	8,7424	7,9561
	% Ağırlık Kaybı	63,89	74,47	76,4	77,05
	İlk Ağırlık (g)	38,0818	33,0573	33,2589	41,402
120 dk.	Son Ağırlık (g)	13,0018	8,3149	7,584	9,3805
	% Ağırlık Kaybı	65,86	74,85	77,2	77,34
360 dk.	İlk Ağırlık (g)	37,2816	33,4561	37,07	33,838
	Son Ağırlık (g)	12,1693	8,1652	8,02	7,4017
	% Ağırlık Kaybı	67,36	75,59	78,37	78,13
	İlk Ağırlık (g)	34,2115	37,1193	39,5174	33,581
1440 dk.	Son Ağırlık (g)	9,8821	7,94	6,7785	5,0823
	% Ağırlık Kaybı	71,11	78,61	82,85	84,87

Karbonize üründe kül analizi ASTM 5142 – 02a, 2003 standardına göre yapılmıştır. 1 g karbonize edilmiş Antep fistiği kabuğu, bir potaya konularak oda sıcaklığındaki firina yerleştirildi, firin sıcaklığı 750±10 °C çıkarılıp 60 dakika bu sıcaklıkta bekletilen numune,



fırından alındıktan sonra desikatörde soğutuldu. Hassas terazide tartılan numunelerin %kül içeriği (A) (1) numaralı denkleme göre hesaplandı. Yapılan analiz neticesinde karbonizasyon ürününde %3,4 kül olduğu belirlenmiştir.

$$A = \left(\frac{F-G}{W}\right) * 100$$
(1)

Burada;

F=kül ve potanın ağırlığı G= boş pota ağırlığı W= Başlangıç numune ve pota ağırlığı

## 3. Deney Sonuçları

Piyasadan temin edilen ve %43,5 karbon, %0,008 kükürt içerdiği tespit edilen Antep fistiği kabukları, 400 °C sıcaklıkta, 60, 120, 360 ve 1440 dakika süre ile karbonizasyona tabi tutulduktan sonra elde edilen karbon ve kükürt içerikleri ile ısıl değerler Tablo 4' te verilmiştir. Şekil 4' te karbonizasyon süresine bağlı olarak karbon ve kükürt değişimleri görülmektedir.

**Tablo 4.** 400 °C sıcaklıkta 60, 120, 360 ve 1440 dakika süre ile karbonizasyona tabi tutulan numunelerin karbon - kükürt içerikleri ve ısıl değerleri

Süre (dk.)	%C	%S	Isıl Değer (kal/g)
0	43,5	0,008	4318
60	66,53	0,0068	4800
120	76,84	0,059	7084
360	76,9	0,013	7080
1440	77.2	0,001	7100



**Şekil 4.** 400 °C sıcaklıkta 60, 120, 360 ve 1440 dakika süre ile karbonizasyona tabi tutulan numunelerin karbon ve kükürt içerikleri

Karbonizasyon işleminde önemli olan parametrelerden biri sıcaklık bir diğeri ise bu sıcaklıkta bekleme süreleridir. 400 °C sıcaklıkta gerçekleştirilen karbonizasyon işleminde artan süreye bağlı olarak sabit karbon oranında artış gözlenirken, kükürt içeriğinde önce bir artış olmuş, ardından önemli oranlarda düşüş olduğu görülmüştür. Biyokütlenin karbonizasyonu üzerine

yapılan çalışmalarda karbonizasyon verimini etkileyen en önemli parametrenin sıcaklık ve süre olduğu belirtilmektedir [2].

600 °C sıcaklıkta gerçekleştirilen karbonizasyon işleminde artan süreye bağlı olarak karbon, kükürt ve ısıl değer kapasitesindeki değişim Tablo 5 ve Şekil 5' te görülmektedir. Bu sıcaklıkta yine sabit karbon oranında rutin bir artış gözlenmiştir. Kükürt içeriği ve ısıl değer bakımından çok büyük değişimler olmamakla birlikte kararlılık gözlenmemiştir.

**Tablo 5.** 600 °C sıcaklıkta 60, 120, 360 ve 1440 dakika süre ile karbonizasyona tabi tutulan numunelerin karbon - kükürt içerikleri ve ısıl değerleri

Süre (dk.)	%C	%S	Isıl Değer (kal/g)
0	43,5	0,008	4318
60	83,16	0,001	7150
120	85,95	0,007	7140
360	86,26	0,027	7080
1440	86.64	0.03	7145



**Şekil 5.** 600 °C sıcaklıkta 60, 120, 360 ve 1440 dakika süre ile karbonizasyona tabi tutulan numunelerin karbon ve kükürt içerikleri

800 ve 900 °C sıcaklıklarda gerçekleştirilen karbonizasyon işleminde artan süreye bağlı olarak karbon, kükürt ve ısıl değer kapasitesindeki değişim sırasıyla Tablo 6 ve 7 ve Şekil 6 ve 7' de görülmektedir. Bu sıcaklıkta yine sabit karbon oranında rutin bir artış gözlenmiştir. Kükürt içeriği ve ısıl değer bakımından çok büyük değişimler olmamakla birlikte kararlılık gözlenmemiştir.

**Tablo 6.** 800 °C sıcaklıkta 60, 120, 360 ve 1440 dakika süre ile karbonizasyona tabi tutulannumunelerin karbon - kükürt içerikleri ve ısıl değerleri

Süre (dk.)	%C	%S	Isıl Değer (kal/g)
0	43,5	0,008	4318
60	88,75	0,009	7273
120	88,28	0,002	7270
360	88,17	0,001	7265
1440	84,85	0,0009	7100

Technology

aterials and Engineering



**Şekil 6.** 800 °C sıcaklıkta 60, 120, 360 ve 1440 dakika süre ile karbonizasyona tabi tutulan numunelerin karbon ve kükürt içerikleri

**Tablo 7.** 900 °C sıcaklıkta 60, 120, 360 ve 1440 dakika süre ile karbonizasyona tabi tutulannumunelerin karbon - kükürt içerikleri ve ısıl değerleri



**Şekil 7.** 900 °C sıcaklıkta 60, 120, 360 ve 1440 dakika süre ile karbonizasyona tabi tutulan numunelerin karbon ve kükürt içerikleri

Farklı sıcaklık ve sürelerde gerçekleştirilen karbonizasyon işlemleri aşağıdaki grafikte özetlenmiştir (Şekil 8). Şekil 8' e göre 800 °C' de 60 dakika bekletilen numune ile 900 °C' de 1440 dakika bekletilen numuneler arasında hem ısıl değer bakımından hem de karbon içeriği bakımından büyük bir fark görülmemektedir. Dolayısıyla Antep fıstığı karbonizasyon işlemleri için, 800 °C sıcaklık, 60 dakika işlem süresinin yeterli olduğu görülmektedir. Bu numunenin %88,75 C içerdiği ve ısıl değerinin ise 7273 kal/g olduğu görülmüştür. Aynı numunede %76,4 ağırlık kaybı gözlenmiş, %3,4 kül içerdiği tespit edilmiştir.



**Şekil 8.** Farklı sıcaklık ve sürelerde karbonizasyon işlemine tabi tutulan numunelerin karbon oranları

Karbonizasyon işlemleri sonucunda numunelerin kükürt ve kül içeriklerinin çok düşük oluşu, sabit karbon ve ısıl değer bakımından ise kayda değer sonuçlara sahip oluşu önemlidir. Fakat numunelerde karbonizasyon esnasında yüksek oranda ağırlık kaybı gözlenmiştir. Literatürde çam kozalakları, çay tesis atıkları gibi organik malzemelerin karbonizasyonu sırasında yaklaşık %60-76 ağırlık kaybından bahsedilmektedir [2, 8]. Ancak bu çalışmada özellikle yüksek sıcaklıklarda ve uzun sürelerde yapılan deneylerde %63-84 arasında bir ağırlık kaybı gözlenmiştir. Ağırlık kaybının yüksek olması ve işlem sonrasında elde edilen karbonize ürünün yoğunluğunun düşük olması işlemin ekonomikliğini olumsuz yönde etkileyecektir.

### 4. Sonuç ve Tartışma

Bu çalışmada, piyasadan satın alma yoluyla temin edilen, atıl durumda olan ve daha çok fırınlarda veya sobalarda yakılan Antep fistiği kabuklarının farklı süre ve sıcaklıklarda karbonizasyonu ile biyokömür üretiminin gerçekleştirilmesine çalışılmış ve aşağıdaki sonuçlar elde edilmiştir.

- Farklı sıcaklık ve sürelerde gerçekleştirilen karbonizasyon işlemleri sonucu 800 °C' de 60 dakika bekletilen numune ile 900 °C' de 1440 dakika bekletilen numuneler arasında hem ısıl değer bakımından hem de karbon içeriği bakımından büyük bir fark görülmemiştir. Bu nedenle, Antep fistiği kabuklarının karbonizasyon işlemleri için, 800 °C sıcaklık ve 60 dakika işlem süresinin yeterli olduğu anlaşılmıştır. Bu numunenin %88,75 C içerdiği ve ısıl değerinin ise 7273 kal/g olduğu görülmüştür. Aynı numunede %76,4 ağırlık kaybı gözlenmiş, %3,4 kül içerdiği tespit edilmiştir.
- Deneysel çalışmalar sonucunda numunelerin kükürt ve kül içeriklerinin çok düşük oluşu, sabit karbon ve ısıl değer bakımından ise kayda değer sonuçlara sahip oluşu önemlidir. Fakat numunelerde karbonizasyon esnasında yüksek oranda ağırlık kaybı gözlenmiştir.
- Bu çalışmada özellikle yüksek sıcaklıklarda ve uzun sürelerde yapılan deneylerde %63-84 arasında bir ağırlık kaybı gözlenmiştir. Ağırlık kaybının yüksek olması ve işlem sonrasında elde edilen karbonize ürünün yoğunluğunun düşük olması işlemin ekonomikliğini olumsuz yönde etkileyecektir.

## 4. Kaynaklar

**1.** Özçimen, D., Ersoy, M.A., 2009, Fındık Kabuğu Karbonizasyon Sonuçlarının İstatistiksel Değerlendirilmesi, İTÜ Mühendislik Dergisi Cilt:8 Sayı:1 Sayfa: 116-124.

The International Conference of Materials and Engineering Technology

**2.** Arancı, Ö.E., Boyrazlı, M., Benkli Y. E., 2015, Çay Tesis Atıklarının Karbonizasyonu, Metalürji Dergisi Sayı: 261 Sayfa: 76-79.

**3.** Tsai, W.T., Lee, M.K., Chang, Y.M., 2007, Fast Pyrolysis of Rice Husk: Product yields and compositions, Bioresource Technology, 98, 22-28.

**4.** Demirbaş, A., 2004, Determination of calorific values of bio-chars and pyro-oils from pyrolysis of beech trunkbars, Journal of Analytical and Applied Pyrolysis, 72, 215-219.

**5.** Ateş, F., Pütün, A.E., Pütün, E., 2006, Pyrolysis of two different biomass samples in a fixedbed reactor combined with two different catalysts, Fuel, 85, 12, 1851-1859.

**6.** Chen, G., Andries, J., Luo, Z., Spliethoff, H., 2003, Biomass pyrolysis/gasification for product gas production: the overall investigation of parametric effects, Energy Conversion and Management, 44, 1875-1884.

**7.** Onay, O., Koçkar, O.M., 2003, Slow, fast and flash pyrolysis of rapeseed, Renewable Energy, 28, 2417-2433.

**8.** Arancı, Ö.E., Bostancı, B., Benkli Y.E., Başgöz, Ö., Güler, Ö., Boyrazlı, M., 2017, Çam Kozalakları Karbonizasyonu, MSNG2017, 395-402.

**9.** T.C. Ekonomi Bakanlığı, İhracat Genel Müdürlüğü, Tarım Ürünleri Daire Başkanlığı, Antep Fıstığı Sektör Raporu, 2017.

**10.** Demiral, I., Gülmezoğlu, A.G., Şensöz, S., 2008, Production of Biofuel From Soft Shell of Pistachio (Pistacia vera L.), Chemical Engineering Communications, 196:1-2, 104-115.

**11.** Açıkalın, K., Karaca, F., Bolat, E., 2012, Pyrolysis of pistachio shell: Effects of pyrolysis conditions and analysis of products, Fuel, 95 169–177.

**12.** Türkiye İstatistik Kurumu (TÜİK), 2018, Bitkisel Üretim İstatistikleri Veri Tabanı, Meyveler, İçecek ve Baharat Bitkiler

# CORROSION PROPERTIES OF DISTALOY AE ALLOYS PREPARED BY P/M METHOD IN 1M KOH SOLUTION

## Ayşe Nur Acar<sup>\*1,3</sup>, Rasiha Nefise Mutlu<sup>2</sup>, Abdul Kadir Ekşi<sup>3</sup>, Ahmet Ekicibil<sup>4</sup>

<sup>1</sup>Cukurova University, Ceyhan Engineering Faculty, Mechanical Engineering Department, Ceyhan, Adana, Turkey

<sup>2</sup>Cukurova University, Sciences and Letters Faculty, Chemistry Department, Sarıçam, Adana, Turkey

<sup>3</sup>Cukurova University, Engineering Faculty, Mechanical Engineering Department, Sarıçam, Adana, Turkey

<sup>4</sup>Cukurova University, Sciences and Letters Faculty, Physics Department, Sarıçam, Adana, Turkey

#### Abstract

Powder metallurgy is widely attracted manufacturing method on the preparing materials due to provide net shape or near-net shape with good mechanical and surface behaviours; and metal and metal based materials prepared by powder metallurgy have important position in the industrial areas such as automotive. The distaloy AE alloy used in this study is a diffusion-bonded alloy and included of Fe, Ni, and Mo alloying elements. This alloy has good mechanical properties such as high strength. In this study; pressure effect on the corrosion behaviours of distaloy AE prepared by P/M method has been examined. Distaloy AE powder pressed on the 400 and 600MPa pressure and sintered at 1200°C temperature for 2 hrs under N<sub>2</sub> atmosphere. Before and after sintering process was carried out. The corrosion performance of green and sintered distaloy AE alloy electrodes has been investigated in 1M KOH by using EIS, polarization techniques. After electrochemical process, EIS measurement, current-potential, cyclic voltammogram curves of distaloy AE alloy samples were taken and, also OM (optic microscopy) images of samples before and after electrochemical process were taken.

Keywords: Powder metallurgy, Distaloy AE, corrosion resistance, OM images.

#### **1. Introduction**

Powder metallurgy method is one of producing methods of iron –based alloys and these alloys prepared by this method have usage areas in the industry such as aoutomotive. This method provides net shape or near-net shape to materials, and enables to reproductivity of this materials, and, also improves physical, chemical and surface properties of these materials [1-4]. Sometimes these properties of these materials can negatively be effected due to porosity existence on the microstructure of materials prepared by this method. In order to decrease porosity; alloy powders can be pressed on the high pressures and sintered at high temperatures under high-purity atmospheres [5].

Distaloy AE alloy used in this study is a diffusion –bonded alloy and has alloying elements such as Fe, Ni and Mo. Alloying elements diffuse into the Fe base metal and metallurgic binding between Fe and alloying elements and parts produced from this alloy has high durable, complex and precise microstructure [6-9] Also, high Ni content in this alloy and good manufacturing and shaping technique improve higher durability [10]. We prepared distaloy AE alloy samples with pressing

Technology

of Materials and Engineering



on the high pressures and sintered at high temperatures according to powder metallurgy technique in order words high density technology [5]. In this study; electrochemical properties of distaloy AE alloy samples prepared by this method examined. With increasing pressure; we observed and determined how chemical and structural properties of samples prepared from this alloy positively or negatively affected.

## 2.Material and Method

The distaloy AE alloy obtained from Höganäs has chemical composition consisting of carbon of 0.01% (wt%), nickel of 4.00% (wt%), copper of 1.5% (wt%), molybdenum 0.50% (wt%), and Fe in balance. This powder has irregular particle shape which is water atomized type.



Figure. 1. The SEM image of Distaloy AE powder

This distaloy AE powder was blended with lubricant in order to acquire homogeneous mixture and 37 g of distaloy AE alloy mixture placed into prismatic mold of  $(10 \times 15 \times 70)$ mm and this molded powder was pressed on the 400 and 600MPa pressures applying conventional press technique at RT (room temperature). Compacted distaloy AE specimens were sintered according to heat treatment process cycle under N<sub>2</sub> (g) atmosphere. Samples annealed on the 600 and 900°C temperatures for 30 min in order to delubricate, and debind for cohesion of grains; according to literature [11-13]. After then, samples sintered at 1200°C, for 2 hours and after sintered samples cooled to room temperature in furnace. Heating and cooling rate were selected as 5°C/min.

The corrosion behaviours of green and sintered distaloy AE alloy samples were studied. The test solution (1 M KOH ) was opened to the atmosphere, and the temperature was 298 K controlled. The electrochemical measurements were carried out using CHI (660 C) electrochemical analyzer under computer control. The three-electrode configuration was used. A platinum sheet (with 2 cm<sup>2</sup> surface area) and Ag/AgCl (3 MKCl) electrodes were used respectively as the auxiliary and the reference electrodes. Electrochemical impedance spectroscopy (EIS) measurements were performed at open circuit potential. The frequency range was between  $10^{-2}$ - $10^5$  Hz and amplitude was 5 mV. The polarization curves were recorded and the scan rate was 1 mV/sec. Surface properties of these alloy samples before and after corrosion test were examined using optic microscope.



## 3. Results and Discussion

The EIS measurements of green sample and sintered distaloy AE samples were obtained in 1 M KOH solution after 1 h immersion time was presented in Figure 2



Figure 2. The EIS Measurement of green and sintered distaloy AE alloy prepared on 400 and 600 MPa pressures

Resistance at the point where the curve starts is solution resistance ( $R_s$ ). The resistance value obtained by extrapolating the horizontal axis of the curve is polarization resistance ( $R_p$ ) which contains charge transfer resistance ( $R_{ct}$ ), diffuse layer resistance ( $R_{dl}$ ), film resistance ( $R_f = R_{f1}$  oxide film) +  $R_{f2}$  (film due to alloying elements) and all other accumulated kinds (corrosion products, any existing molecules or ions, etc.) ( $R_a$ ), ( $R_p = R_{por} + R_f$ ) and Rpor is pore resistance caused alkali corrosion ( $R_{por} = R_{ct} + R_{dl} + R_a$ ) [14-16]. The highest polarization resistance was obtained on the green distaloy AE sample prepared on the high pressure. In the case of sintered samples, the highest polarization resistance was acquired on the distaloy AE sample prepared on the high pressure. This means that in both green samples and sintered samples, as pressure increases, corrosion resistance increases.

The current-potential curves green and sintered distaloy AE alloy prepared on 400 and 600 MPa pressures were taken parallel to the impedance curve are shown in Figure 3.





Anodic process consists of dissolution reactions of alloying elements. [17]. The main component in this alloy is Fe. Other alloying elements also dissolve similarly in different potentials.

$$Fe + 2e^- \rightarrow Fe^{2+}$$
 (1)

As the corrosion event continues,  $O_2$  in the range ends and the event does not complete due to the passive oxides formed. In sintered samples, these reactions occur on the surface, whereas green samples mostly occur in pores. Fe<sup>+</sup> and Fe<sup>+2</sup> ion concentrations in the range will undergo hydrolysis as soon as possible [17].

$$Fe^{2+} + 2H_2O \rightarrow Fe(OH)_2 + 2H^+$$
 (2)

Corrosion becomes more stable as  $Fe (OH)_2$  became and then collected in the range to dissolve as iron oxide or rust [17].

For Fe-containing alloys in electrolyte; the active solubility behaviour of active iron occurs naturally. Magnetite formation in alkaline medium;

$$3Fe(0H)_2 + 20H^- \rightarrow Fe_3O_4 + 4H_2O + 2e^-$$
 (3)

$$3FeO + 2OH^- \rightarrow Fe_3O_4 + H_2O + 2e'$$
 (4)



In the abovementioned oxidation and reduction reactions (3) and (4), the transition of stainless steels from active solubility to passive solubility is seen. As is known, magnetite is an important compound in the corrosion resistance of steel due to passive film layer [18,19] and consists of oxide mixture consisting of  $Fe^{2+}$  and  $Fe^{3+}$  ions. Magnetite is chemically formulated as  $Fe_3O_4$  or  $FeO.Fe_2O_3$  [19].  $Fe_3O_4$  magnetite consists of an inverse cubic array spinel structure containing cubic array oxygen ions with all  $Fe^{2+}$  ions in the semi-octahedral regions.  $Fe^{3+}$  ions settle in the remaining octahedral and tetrahedral region.  $FeO.Fe_2O_3$  magnet; FeO is named as wustit;  $Fe_2O_3$  is called hematite [19.] Corrosion current decreases in sintered samples.

The Tafel curves of green and sintered Distaloy AE alloy samples are observed in Figure 4.



**Figure 4.** The Tafel curves of green and sintered distaloy AE alloy prepared on 400 and 600MPa pressures immersed in 1M KOH solution

The current-potential curve consists of two branches, including anodic and cathodic. The remaining portions of the right of the corrosion potential ( $E_{cor}$ ) are the anodic branch. The anodic reactions are given in equation (1);

On the other hand, the remaining portions of the left of the corrosion potential  $(E_{cor})$  are the cathodic branch. The main cathodic reaction is the reduction of the dissolved oxygen and evolution of hydrogen gas [14,20-21].

$$0_{2(g)} + 2H_20 + 4e^- \to 40H^-$$
 (5)

$$2H_20 + 2e^- \rightarrow H_{2(g)} + 20H^-$$
 (6)

In Figure 4, when the current-potential curves of green and sintered distaloy AE alloy samples prepared on 400 and 600MPa pressures are examined, it is seen the corrosion potential  $(E_{cor})$  values for different compact pressure. Corrosion currents were calculated from the Tafel



constants for these potentials.  $E_{cor}$  value of green sample pressed on the 400 MPa for 1 h immersion time is - 0.89 V.  $E_{cor}$  value of green sample prepared on the 600 MPa for 1 h immersion time is - 0.85 V.  $E_{cor}$  value of pressed 400 MPa and sintered for 1 h immersion time is - 0.75 V.  $E_{cor}$  value of sintered distaloy AE prepared on the 600 MPa and for 1 h immersion time is - 0.75 V Corrosion current decreases in sintered distaloy AE samples. The lowest corrosion current belongs to the sintered sample prepared on the 600 MPa.



**Figure 5.** The OM (optic microscope) images of green and sintered Distaloy AE alloy prepared on 400 and 600MPa pressures before and after corrosion (50x magnification)

In the Figure 5; The OM images of green and sintered Distaloy AE alloy samples before and after corrosion test observed. On the OM images of surfaces of both green and sintered samples; pressure and heat treatment effect observed. When increasing pressure; grain binding happens due to decreasing of porosity among grains. Heat treatment besides applied pressing technique play an important role on the bonding of more grains, pores become smaller. Because; atomic



After corrosion; on the examining OM images of green and sintered distaloy AE alloy samples; good surfaces observed on the sintered distaloy AE samples according to green distaloy AE samples. Thereby, the best surface appeared on the sintered alloy sample prepared on the high pressure. It was observed that for green samples; corrosion products diffused into the pore. Porosity existence decrease corrosion resistance, electrochemical activity on the surfaces increases and stable potential of distaloy AE samples decreased [23]. For sintered samples; corrosion products play a role as protective oxide film layer on the surfaces of samples.

### 4. Conclusion

In this study; the corrosion resistance behaviours and surface characterization of distaloy AE alloy samples prepared by powder metallurgy method were examined and optimum corrosion resistance and good surface on the distaloy AE samples determined. Following experimental results are given;

- With increasing pressure; corrosion resistance of distaloy AE alloy samples increased because closing of porosity and grain binding.
- Better corrosion resistance obtained on the sintered distaloy AE alloy samples prepared on the high pressure.
- Surface of sintered distaloy AE alloy samples prepared on the high pressure has smmoth surface after corrosion test.

**Acknowledgement:** The authors are greatly thankful to Cukurova University research funding (FBA-2018-11074)

## References

**1.** Giménez S., Vagnon A., Bouvard D. and Van der Biest O., Influence of the Green Density on the Dewaxing Behaviour of Uniaxially Pressed Powder Compacts, Materials Science and Engineering A.2006, (430), 277–284

**2.** Yılmaz R. and Ekici M.R., Üretim Parametrelerin Düşük Alaşımlı TM Çeliklerin Sertlik Ve Aşınma Özelliklerine Etkisi ISITES2015 Valencia –Spain, 2545-2554.

**3.** Zarebski, K. And Putyra ,P., Iron Powder-based Graded Products Sintered by Conventional Method and by SPS, Advanced Powder Technology.2015,(26) 401–408.

**4.** Pirayesh, N., Teimouri, M., H. Khorsand, H. And Khoie, M.M., Influence of P/M Distaloy AE Steel Porosities and Nitriding Process Parameters in Diffusion and Configuration of Nitrided Layers Through Advanced Liquid Salt Bath Nitriding Process Defect and Diffusion Forum. 2008, (273-276), 342-347



ME

6. Lindskog, P. The History of Distaloy, Powder Metallurgy. 2013, 56(5), 351-361.

7. Öksüz, K.E., Kumruoğlu, L.C. and Tur O., Effect of Sicp on the Microstructure and Mechanical Properties of Sintered Distaloy DC Composites, (5th International Biennial Conference on Ultrafine Grained and Nanostructured Materials, UFGNSM15), 11-12 November 2015, Tehran-Iran, Procedia Materials Science. 2015, (11), 49 – 54.

8. Karwan-Baczewska, J., The Properties of Fe-Ni-Mo-Cu-B Materials Produced Via Liquid Phase Sintering, Archives of Metallurgy and Materials. 2011, 56 (3), 789-796.

9. Öksüz, K. E., Akınalp, Y. A. and Kumruoğlu, L. C. Tribological Behavior of Distaloy DC Components, Materials Science Forum.2017, (907), 121-125.

**10.** Akbulut, I., and Ekşi, A.K., The Influence of Sintering and Nitriding Processes on Distaloy AE Powder Materials, C.Ü Fen ve Mühendislik Bilimleri Dergisi. 2012, 27(5), 128-137.

11. Pandya, S., Ramakrishna, K.S., Annamalai, A.R. and Upadhyaya, A., Effect of Sintering Temperature on the Mechanical and Electrochemical Properties of Austenitic Stainless Steel, Materials Science and Engineering A. 2012, (556) 271–277.

12. Butković, S., Oruč, M., Šarić, E., Mehmedović, M., Effect of Sintering Parameters on the Density, Microstructure And Mechanical Properties of the Niobium-Modified Heat-Resistant Stainless Steel GX40CrNiSi25-20 Produced by MIM Technology, Materiali in tehnologije / Materials and technology. 2012,46(2), 185–190.

13. Butković, S., Sinterability And Tensile Properties of Nickel Free Austenitic Stainless Steel X15CrMnMoN 17 11 3, Technical Gazette/Tehnički Vjesnik. 2013, 20(2), 269-274.

14. Mutlu, R.N., Ateş, S. Yazıcı, B., Al-6013-T6 and Al-7075-T7351 Alloy Anodes for Aluminium-Air Battery, International Journal of Hydrogen Energy.2017,(42), 23315 -23325

15. Rosalbino, F., Delsante, S., Borzone, G., and Angelin, E., Influence of Rare Earth Metals on the Characteristics of Anodic Oxide Films on Aluminium and Their Dissolution Behaviour in NaOH Solution, Corrosion Science. 2010, (52), 322-326

16.Solmaz, R., Investigation of the Inhibition Effect of 5-((E)-4-phenylbuta-1,3dienylideneamino)-1,3,4-thiadiazole-2-thiol Schiff base on Mild Steel Corrosion in Hydrochloric Acid, Corrosion Science.2010, (52), 3321-3330

17. Huo, Q., Zhang, G., Qju, Y. and Guo, X., The Crevice Corrosion Behaviour of Stainless Steel in Sodium Chloride Solution, Corrosion Science.2011,(53), 4065-4072.

18. Wang, L., Sun, J., Kang, B., Li, S., Ji, S., Wen, Z., and Wang X., Electrochemical Behaviour and Surface Conductivity of Niobium Carbide-Modified Austenitic Stainless Steel Bipolar Plate, Journal of Power Sources. 2014, (246), 775-782.

19. Freire, L., Catarino, M.A., Godinho, M.I., Ferreira, M.J., Ferreira, M.G.S., Simões, A.M.P., and Montemor, M.F., Electrochemical and Analytical Investigation of Passive Films Formed on Stainless Steels in Alkaline Media, Cement and Concrete Composites. 2012, 34(9),1075-1081.



**20.** Emregül, K.C. and Aksüt, A.A., The Behavior of Aluminum in Alkaline Media, Corrosion Science. 2000, (42), 2051-2067.

**21.** Baran, E. and Yazıcı, B., Effect of Different Nano-Structured Ag doped TiO2- NTs Fabricated by Electrodeposition on the Electrocatalytic Hydrogen Production, International Journal of Hydrogen Energy. 2016, (41) 2498-2511.

**22.** Yao, B., Zhou, Z., Duan, L., and Zhiyu Xiao, Z., Compressibility of 304 Stainless Steel Powder Metallurgy Materials Reinforced with 304 Short Stainless Steel Fibers, Materials. 2016, 9(161), 1-11.

**23.** Xu, W., Lu, X., Zhang, B., Liu, C., Lv, S., Yang, S., and Qu, X., Effects of Porosity on Mechanical Properties and Corrosion Resistances of PM-Fabricated Porous Ti-10Mo Alloy, Metals. 2018, 8(188), 1-13.

## SPORTSWEAR FABRIC SELECTION FOR DIFFERENT WEATHER CONDITIONS USING AHP AND TOPSIS ANALYSIS

of Materials and Engineering Technology

## BURAK SARI<sup>1</sup>, NİDA OĞLAKCIOĞLU<sup>2</sup>

<sup>1</sup> Bitlis Eren University, Fine Arts Faculty, Textile and Fashion Design Department, Bitlis, TURKEY. <sup>2</sup> Ege University, Engineering Faculty, Textile Engineering Department, İzmir, TURKEY.

#### Abstract

Sport activities have existed with the history of humanity and constitute an indispensable part of our daily life. Sports have no limitations such as social status, age or gender and are one of the most demanding leisure activities with various branches as amateur or professional. For this reason, the sports market is a giant industry which is fed by many sectors with unique needs. Textile is one of the primary related sectors of sports industry. This area, called sports textiles, includes all kinds of textile based applications to meet the needs of its users during sports. There are two basic expectations, namely safety and performance, from textile products used in sport activities. There is a need for sports textiles at different levels of security, depending on the location of sports such as indoor or outdoor and atmospheric conditions such as temperature, humidity or wind. In sports textiles, the basic capabilities of the product depend on the properties of the fabric. In this study, different fabric types were evaluated by AHP and TOPSIS method to perform fabric selection process for a sports product to be used in different atmospheric conditions. Water vapor and air permeability, thermal conductivity, thermal absorptivity and thermal resistance were used as evaluation criteria for the fabrics. Fabric samples were produced with different yarns and inlay types by using double-sided jacquard knitting pattern. The obtained thermal comfort values were evaluated mutually and the ideal types to be used in hot and cold conditions were determined among the alternatives.

Keyword: Sportswear, thermal comfort, AHP, TOPSIS, multi-criteria decision.

#### **1. Introduction**

One of the most related areas in terms of clothing comfort is sports textiles. Due to the fact that sport is a part of our daily life, the limit of comfort expectation in sportswear is increasing day by day. It has become almost impossible to use products that do not have sufficient comfort features both in functional sportswear for professional sports and in simple leisure wear. In the new sports industry, where the classical sports concept is destroyed, products with different characteristics are produced for each branch and the parameters for comfortable use are redefined for each sports branch. The positive effects provided by the comfortable garment in terms of physical and physiological aspects also motivate the athlete psychologically [1].

In general, sports textiles are expected to have high-level properties in terms of thermal comfort. For a textile product to be classified as having a high thermal comfort level, it must have a set of abilities to assist body functions for constant body internal temperature in accordance with outdoor conditions [2]. Body systems, which undertake variable tasks during sporting activity, cannot manage the whole flow regularly in the sense of possible discomfort. Thermal properties, water vapor permeability and air permeability are the parameters that determine the success level of thermal textiles in different tasks [3].



The International Conference of Materials and Engineering Technology Multi criteria decision making methods are the mathematical methods used for the most appropriate selection by evaluating the data of alternatives in the decision making process where multiple alternatives exist. Various models are used to perform comprehensive superiority analyzes among options to avoid making decisions based on a single person or a single event. AHP (Analytical Hierarchy Process) and TOPSIS (Technique for Order Preference by Similarity to Ideal Solution) are frequently preferred methods. The AHP method has a working system that establishes the hierarchical superiority of the criteria against each other and determines the percentage of impact on the decision. The AHP method is suitable for the analysis of both quantitative and qualitative data; it is preferred in many areas because it can structure complex problems hierarchically and can work together with other decision making methods. TOPSIS is defined as the distances of the options to the ideal solution created according to the objectives of the selection process. For the decision, the alternatives closest to the positive ideal solution and the farthest to the negative ideal solution are used. TOPSIS is preferred in many areas for decision making since it does not require complex calculations [4,5].

Multi criteria decision making methods are often preferred in the textile sector as in many other sectors. In particular, long-term investment decisions such as supplier [6–8], factory location [9], machine maintenance technique selection [10,11] were analyzed. In addition to this, there have been studies on raw material type selection [12] and rotor type selection on open-end spinning machines [13] under the influence of factors affecting yarn quality. There are few studies on fabric selection using comfort properties. In these studies, raw material selection for socks [14], handloom fabric selection for summer use [15] and raw material selection for sports fabrics [16] were examined.

In this study, the best fabric selection process was made with multiple decision making methods (AHP + TOPSIS) among the fabrics having different production parameters for use in different climatic conditions. Separate evaluation criteria were used for hot and cold conditions and the criteria for decision-making fabrics were chosen as general comfort characteristics.

## 2. Materials and Methods

The fabrics in the scope of the study were manufactured by using E20-38" Monach electronic jacquard circular knitting machine. The yarn and inlay types used in the production of doublelayer knitted fabrics are given in Table 1 and 600 denier textured PET yarns are used as inlay yarn. Other production parameters of the fabrics, such as tightness and elastane feeding tension have been kept constant.

Sample Code	Front face yarn	Back face yarn	Inlay design
F1		70/20 PA/EA	
F2		70/40 PA/EA	Empty
F3	150/40 1	70/40 PET/EA	
F4	150/48 denier PFT		1x1
F5	I LI	70/40 DET/E A	2x2
F6		/0/40 FEI/EA	3x1
F7			1x3

<b>Fable 1.</b>	Yarns	and	inlay	designs
-----------------	-------	-----	-------	---------

The standards and test devices used to investigate the comfort properties of fabrics are shown in Table 2.



<b>Comfort parameters</b>	Test devices	Standards
Thickness (mm)	Alambeta (Sensora)	-
Mass per area (g/m <sup>2</sup> )	-	EN ISO 12127
Thermal conductivity (W/mK)	Alambeta (Sensora)	-
Thermal absorptivity $(Ws^{1/2}/m^2K)$	Alambeta (Sensora)	-
Thermal resistivity (m <sup>2</sup> W/K)	Permetest (Sensora)	EN 31092
Water vapour permeability (%)	Permetest (Sensora)	EN 31092
Air permeability (l/m <sup>2</sup> /s)	FX3300 (Textest)	EN ISO 9237

# Table 2. Comfort properties, test devices and related standards.

Since it has different needs in terms of comfort, selection criteria for hot and cold weather conditions have been determined separately. One of the basic characteristics of a comfortable sports garment is the breathable surface structure and therefore water vapor and air permeability are used as common criteria for both climatic conditions. Thermal conductivity and absorptivity are considered as the selection criteria for hot weather condition, while thermal resistance is determined as the selection criteria for cold weather condition.

AHP method (9 point scale of relative importance) was used to calculate the weights of selection criteria for each weather conditions. By evaluating the mutual advantages, the weight values of each criterion were obtained to be used in the next steps. In TOPSIS method, these weight values were included in the calculations and the final rankings of fabric types were formed. Figure 1 illustrates the flow of multi criteria decision making process.



Figure 1. Multi criteria decision making model

## 3. Results and Discussion

Pair wise comparison matrixes for weight determinations using AHP method are given in Table 3 and Table 4. Consistency analysis showed that the judgments of both matrixes were consistent and acceptable (CR < 0.1).

Technology



Criteria	RWVP	AP	T. Con	T. Abs	Weight
RWVP	1	3	5	6	0.558
AP	1/3	1	3	4	0.259
T. Con	1/5	1/3	1	2	0.112
T. Abs	1/6	1/4	1/2	1	0.071
	$\lambda_{max}$			4.077	
Consistency index (CI)				0.026	
Consistency ratio (CR)				0.029	

Table 3. Pair-wise comparison matrix and relative weights of hot weather condition criteria

Table 4. Pair-wise co	mparison mat	ix and relative	e weights of col	d weather	condition	criteria
-----------------------	--------------	-----------------	------------------	-----------	-----------	----------

Criteria	RWVP	AP	T. Res	Weight
RWVP	1	5	2	0.568
AP	1/5	1	1/4	0.098
T. Res	1/2	4	1	0.334
$\lambda_{max}$			3.0	26
Consistency index (CI)		0.012		
Consistency ratio (CR)			0.0	21

While doing sports in a cold weather condition, body temperature increases due to sport activities, but it loses heat due to the outdoor environment. Since the perspiration occurs in cold weather, the water vapor formed between the skin and the garment must be effectively transported to the outer surface of the fabric. On the other hand, another important function expected from sportswear is the thermal resistance in order to keep the heat loss under control due to low temperature. In order to form a breathable structure, air permeability is a low desirable property and less effective than the other two factors.

Relative weights of comfort properties were determined separately for hot and cold conditions by using AHP method. Calculations were made by TOPSIS method for fabric selection by using weights obtained from pair-wise comparisons and measurement results of comfort properties. The weighted normalization matrixes for both conditions are shown in Tables 5 and 6.



Sample Code	RWVP	AP	T. Con	T. Abs
F1	0.281	0.153	0.037	0.022
F2	0.213	0.093	0.041	0.024
F3	0.240	0.093	0.041	0.024
F4	0.183	0.083	0.044	0.029
F5	0.178	0.082	0.044	0.029
F6	0.175	0.070	0.046	0.030
F7	0.183	0.090	0.043	0.028
Positive ideal solution (d +)	0.281	0.153	0.046	0.030
Negative ideal solution (d -)	0.175	0.070	0.037	0.022

 Table 5. Weighted normalization matrix of hot weather condition

Table 6.	Weighted	normalization	matrix of	f cold	weather	condition
----------	----------	---------------	-----------	--------	---------	-----------

Sample Code	RWVP	AP	T. Res
F1	0.286	0.058	0.056
F2	0.216	0.035	0.090
F3	0.244	0.035	0.109
F4	0.186	0.031	0.149
F5	0.181	0.031	0.149
F6	0.178	0.026	0.157
F7	0.187	0.034	0.138
Positive ideal solution (d +)	0.286	0.058	0.157
Negative ideal solution (d -)	0.178	0.026	0.056

The relative closeness and final ranking of the fabrics used for hot and cold weather conditions are indicated in Table 7. It was calculated that the sample with code F1, which is the best choice for hot weather conditions, had the obvious advantage, while the sample with code F3 was the best choice for cold weather conditions. The relative closeness values of the fabrics according to the cold conditions were found to be close to each other.

Table 7. Final	l ranking	of fabric	alternatives
----------------	-----------	-----------	--------------

Sample	Hot weather			Cold weather				
Code	<b>d</b> +	d -	Relative closeness	Rank	<b>d</b> +	d -	Relative closeness	Rank
F1	0.012	0.135	0.917	1	0.101	0.113	0.529	2
F2	0.091	0.045	0.330	3	0.099	0.052	0.344	7
F3	0.073	0.069	0.486	2	0.068	0.085	0.556	1
F4	0.121	0.018	0.132	5	0.104	0.093	0.472	3
F5	0.125	0.016	0.115	6	0.109	0.093	0.461	5
F6	0.134	0.012	0.083	7	0.113	0.101	0.471	4
F7	0.116	0.024	0.171	4	0.104	0.083	0.443	6



## 4. Conclusions

It is expected that a garment to be used in sports activities will be quite different from classic clothing in terms of comfort properties. For a comfortable product there is a need for a complex design stage that does not depend on a single parameter. In order to use ideal fabric structures, the comfort properties in relation to each other should be interpreted mutually. Therefore, the selection of fabric structures suitable for the purpose can be realized by methodological approaches such as multi criteria decision making methods. In the study, AHP and TOPSIS method were used together for fabric selection process for different weather conditions. While water vapor and air permeability play a dominant role for hot environment, it is seen that the ideal fabric for cold weather conditions stands out with its water vapor permeability as well as thermal resistance properties.

The International Conference of Materials and Engineering Technology

## References

- 1. Hayes SG and Venkatraman P. Materials and Technology for Sportswear and Performance Apparel. New York: CRC Press; **2016**. 342 p.
- 2. Bhatia D and Malhotra U. Thermophysiological Wear Comfort of Clothing: An Overview. J Text Sci Eng. **2016**;06(02).
- 3. R. Shishoo. Textiles for sportswear. Cambridge: Woodhead Publishing Limited; **2015**. 272 p.
- 4. Ishizaka A, and Nemery P. Multi-Criteria Decision Analysis: Methods and Software. Chichester, UK: John Wiley & Sons Ltd; **2013**. 296 p.
- 5. Triantaphyllou E. Multi-criteria Decision Making Methods: A Comparative Study. Boston, MA: Springer US; **2000**. 289 p.
- 6. Yeşim Yayla A, Yildiz A and Özbek A. Fuzzy TOPSIS method in supplier selection and application in the garment industry. Fibres Text East Eur. **2012**;93(4):20–3.
- 7. Zarbini-Sydani A, Karbasi A and Atef-Yekta E. Evaluating and selecting supplier in textile industry using hierarchical fuzzy TOPSIS. Indian J Sci Technol. **2011**;4(10):1322–34.
- 8. Güleş HK, Çağlıyan V, and Tuğba Ş. Supplier selection based on analytic hierarchy process methods in garment sector. Selcuk Univ J Inst Soc Sci. **2014**;Dr. Mehmet Yıldız Special Edition:159–70.
- 9. Ertuğrul I and Karakaşoğlu N. Comparison of fuzzy AHP and fuzzy TOPSIS methods for facility location selection. Int J Adv Manuf Technol. **2008**;39:783–95.
- Shyjith K, Ilangkumaran M and Kumanan S. Multi-criteria decision-making approach to evaluate optimum maintenance strategy in textile industry. J Qual Maint Eng. 2008;14(4):375–86.
- 11. Ilangkumaran M and Kumanan S. Selection of maintenance policy for textile industry using hybrid multi-criteria decision making approach. J Manuf Technol Manag. **2009**;20(7):1009–22.
- 12. Banwet DK and Majumdar A. Comparative analysis of AHP-TOPSIS and GA-TOPSIS methods for selection of raw materials in textile industries. Proc 2014 Int Conf Ind Eng Oper Manag Bali, **2014**;2071–80.
- 13. Majumdar A, Kaplan S and Göktepe Ö. Navel selection for rotor spinning denim fabrics using a multi-criteria decision-making process. J Text Inst. **2010**;101(4):304–9.



The International Conference

of Materials and Engineering Technology

UWE Bristol

- 15. Mitra A, Majumdar A, Ghosh A, Majumdar PK and Bannerjee D. Selection of handloom fabrics for summer clothing using multi-criteria decision making techniques. J Nat Fibers. **2015**;12(1):61–71.
- 16. Duru SC. Water-related comfort properties of silver-modified polyamide fabrics treated with wicking and antibacterial finishes. Cloth Text Res J. **2019** Aug 26;OnlineFirst:1–12.

## THE EFFECT OF USING SILVER BLENDED YARNS ON THE ABRASION RESISTANCE PROPERTY OF WOVEN FABRICS

The International Conference of Materials and Engineering Technology

## ZEHRA KAYNAR TAŞCI<sup>1</sup>, NİHAT ÇELİK<sup>2</sup>

<sup>1</sup>Çukurova University, Engineering Faculty, Textile Engineering Department, Adana, TURKEY. <sup>2</sup>Çukurova University, Engineering Faculty, Textile Engineering Department, Adana, TURKEY.

#### Abstract

In this study, the abrasion resistance properties were investigated of woven fabrics which contain silver and cotton fibres at different ratios. Fabrics were produced as shirting. Because the abrasion property is important shirting fabrics; the fabrics were tested with a Martindale Abrasion Tester. The abrasion resistance of the fabrics was evaluated according to their mass loss ratio after 3 different cycles (5000, 10000, 20000). It has been observed that silver fiber has a negative effect on abrasion resistance.

Keyword: Abrasion resistance, Cotton, Silver fibers, Yarn quality parameters, Woven fabric

#### **1. Introduction**

Silver is generally preferred in textile products for their antibacterial activity and electromagnetic shielding properties. There are several studies about silver in literature. In the studies silver is applied to textile surfaces in many forms. In literature, studies were conducted about antibacterial/antimicrobial textiles with silver nanoparticles and silver ions [1–6]. There are several studies to gaining antibacterial/antimicrobial activity to textile surfaces using silver nitrate in finishing processes [7-9]. It is known that for the purpose of obtaining antibacterial surface, silver is also used colloidal form [10-11]. Silver also finds usage area due to its electromagnetic shielding and conductivity properties. Duran and Kadoğlu [12] examined conductivity properties the woven fabrics that produced with silver/cotton blended ring yarns. They also studied [13] electromagnetic shielding characterization of woven fabrics produced with silver-containing (Ag/PA/Co) core yarns and silver-containing (Ag/PA-Co) blended yarns. In this study, silver was used as form of fiber by blending with cotton fibers. Ring yarns were produced with this fiber blend. Then, shirting woven fabrics were produced by using these yarns. In the study, it is aimed to give antibacterial properties to fabrics by using silver fiber. These yarns are used in the woven fabrics only weft directions considering the cost factor. The factors affecting abrasion resistance of fabrics are mainly classified as follows: Fiber, yarn, fabric properties and finishing processes. Fiber type, fiber fineness and fiber length are the main parameters that affect abrasion. Yarn structure, count, twist and hairiness are the main properties which affect abrasion of the textile fabrics. Fabric construction, thickness, weight,

properties which affect abrasion of the textile fabrics. Fabric construction, thickness, weight, the number of yarn (thread density) and interlacing per unit area are the fabric properties affecting abrasion. Finishing treatments, the types and concentration of the chemicals used in the treatment processes are also the parameters affecting the abrasion characteristics of the fabrics [14].

In the literature, there are many studies investigating the effect of yarn properties on abrasion resistance, such as works with fancy and chenille yarns, conventional and compact ring-spun yarns [15-20]. However, no study on the effect of silver fiber on abrasion resistance has been found.

The aim of this research is investigate the influence of silver fiber on the abrasion resistance of woven fabrics.

## 2. Materials and Methods

In this study, Ne 40/1 compact combed cotton (%100) warp yarns are used as raw material. Also, Ne 40/1 in 4 different types of weft yarns were produced by using Aegean cotton and Aegean cotton / Silver fibers (known under the trade name X-Static®). The yarns were provided from SANKO Textile Industries. According to fiber blend ratio the weft yarn types are given with codes in Table 1.

Table 1. Wert yarns and fiber blend ratios			
Weft yarn codes	Cotton fiber content (%)	Silver fiber content (%)	
G <sub>0</sub>	100	0	
G <sub>10</sub>	90	10	
G <sub>15</sub>	85	15	

 $G_0$  yarn contains %100 cotton fibers. It is encoded  $G_0$ , because there is no silver fibers. Other weft yarns are encoded as  $G_{10}$  and  $G_{15}$  according to X-static® percentage. Some manufacturing details of weft yarns are given in Table 2. All yarns were produced on the same conditions [21].

<b>Table 2.</b> Production datas for the weft yarn				
Output speed of comb (m / min)	100			
Sliver number on the comb (Ne)	0,120			
Output speed from draw-frame (m / min)	650			
Sliver number on the draw-frame (Ne)	0.120			
Roving number (Ne)	1.30			
Roving twist (tour/m)	54			
Ring twist (tour/m)	921			
Ring speed (rev / min)	14000			

In the study, woven fabrics were produced by using these yarns with different weft insertion order. All the fabrics were manufactured as 4/1 "S" direction and 3-hopping satin fabrics. The texture characteristics of a commercial shirting fabric were taken as a basis for production. The same type Ne 40/1, %100 combed cotton compact yarns were used as warp yarns in all fabrics. Combinations were obtained using different type weft yarns with different orders in the fabrics. Weft insertion plans in the fabrics are given in Table 3. According to Table 3, for example, K2B fabric includes two G0 yarns (with %0 silver fibre) and two G10 yarns (with %10 silver fiber) in weft direction of fabric [21].



	Weft			
Fabric codes	Go	G10	Go	G15
Ref. fabric (K0)	4	0	4	0
K2A	3	1		
K2B	2	2		
K2C	1	3		
K2D	0	4		
K3A			3	1
K3B			2	2
K3C			1	3
K3D			0	4

Desizing+bleaching, laundering+drying, singeing, mercerization and again laundering+drying pre-finishing processes were applied to all fabrics in finishing department. The fabrics were not colored. Finally, the fabrics were passed on the Babcock machine. In this manner, dimension stability of fabrics was provided.

All test specimens were conditioned under standard atmospheric conditions  $(20 \pm 2^{\circ} \text{ C} \text{ and } 65\pm2\%$  relative humidity) 24 hours and before the tests. Yarn properties were tested with Uster Tester 5. Abrasion tests were performed on a Martindale Abrasion Tester. The abrasion resistance of the fabrics was evaluated according to their mass loss ratio. Mass loss values were determined at the end of 5000, 10000 and 20000 rubbing cycles, according to TS EN ISO 12947-3 standards [22]. Also mass loss ratios of fabrics were calculated according to Formula 1. The pressure applied to the fabric during rubbing was 9 kPa as indicated for clothing.

Loss percentage in relevant property (%) = ((A-B)/A)\*100 (1)

A = relevant property before abrasion, and B = after abrasion [14].

## 3. Results and Discussion

Abrasion resistance of fabrics depends on fiber, yarn and fabric properties and finishing processes. Yarn structure, count, twist and hairiness are the main properties which affect abrasion of the textile fabrics [14]. In this context hairiness, mass unevenness and imperfections values of weft yarns including cotton and silver fibers at different ratios are given Table 4 [21, 23].

Weft yarn codes	Uster Hairiness Index (H)	Unevenness Values (% Um )	Coefficient of Mass Change (% CVm)
G0	5.72	11.05	9.20
G10	6.45	13.8	11.5
G15	6.32	15.2	12.7

Table 4. Hairiness	, unevenness and	imperfections	values of weft yarns
--------------------	------------------	---------------	----------------------

In the experimental study, abrasion resistances of fabrics were examined on the basis of mass loss. The mass loss values of the reference and silver containing fabrics at 5000, 10000 and 20000 cycles are given in Fig. 1. Also mass loss ratios of fabrics after abrasion test at 20000 rubbing cycles are seen at Fig. 2.



Figure 1. Mass values of samples after different rubbing cycles



Figure 2. Mass loss (%) after 20000 rubbing cycles

The mass loss value of the fabric without silver fibres (K0) was less as shown in Figure 2. As seen on the Table 4, generally, silver fiber content negatively affected of yarn properties. This is thought to be due to the lower cohesion between the cotton fibers and the silver fibers [23]. Fiber and yarn properties affect abrasion resistance. For this reason, a similar situation was observed in fabrics. The presence of silver fibers in the fabrics adversely affected the abrasion resistance. Since silver fibers are easily separated from the fabric surface during the rubbing, mass loss is faster than cotton fibers (Fig. 1). However, no correlation was found between the increase in silver fiber content in the fabrics and the abrasion resistance values.

**Acknowledgments:** This study was supported with MMF2012YL14 project number by Cukurova University Scientific Research Projects Department. We would also like to thank BOSSA T.A.Ş. R & D center for their valuable contributions.



## References

1. Wendler, F., Meister, F., Montigny, R. and Wagener M., "A New Antimicrobial ALCERU® Fibre with Silver Nanoparticles", Fibres & Textiles in Eastern Europe. January / December 2007, Vol. 15, No. 5 - 6 (64 - 65), page: 41-45.

The International Conference of Materials and Engineering

2. Wawro, D., et al, "Antibacterial Chitosan Fibres Containing Silver Nanoparticles", Fibres & Textiles in Eastern Europe. 2012, 20, 6B (96): 24-31.

3. Pupkevičiūtė, S. et al, "Formation and Antibacterial Property Analysis of Electrospun PVA Nonwoven Material with a Small Amount of Silver Nanoparticles", Fibres & Textiles in Eastern Europe. 2015, 23, 6(114): 48-54.

4. Üreyen, M. ve ark., "Antibacterial Efficacy And Laundering Durability Of Textile Fabrics Treated By Newly Developed Silver Doped Nano Scaled Bio-Antimicrobial Finishing Agent", The Journal Of Textiles and Engineers. 2008, 15(69).

5. Gutarowska, B. and Michalski, A., "Antimicrobial Activity of Filtrating Meltblown Nonwoven with the Addition of Silver Ions", Fibres & Textiles in Eastern Europe. 2009, Vol. 17, No. 3 (74) pp. 23-28.

6. Miaśkiewicz-Peska, E. and Łebkowska, M., "Effect of Antimicrobial Air Filter Treatment on Bacterial Survival", Fibres & Textiles in Eastern Europe. 2011, Vol. 19, No. 1 (84) pp. 73-77.

7. Filipowska, B. et al., "New Method for the Antibacterial and Antifungal Modification of Silver Finished Textiles", Fibres & Textiles in Eastern Europe. 2011, Vol. 19, No. 4 (87) pp. 124-128.

8. Ursache, M. Et al., "Investigation on the Effects of Antibacterial Finishes on Dyed Cotton Knitted Fabrics", Tekstil ve Konfeksiyon, 3/2011.

9. Polowinski, S. and Jantas, R., "Antibacterial and Catalytic Properties of Textiles with Modified Surfaces", Fibres & Textiles in Eastern Europe 2008, Vol. 16, No. 4 (69) pp. 104-107.

10. Montazer, M. ve ark., "Durable Anti-bacterial Nylon Carpet Using Colloidal Nano Silver", Fibres & Textiles in Eastern Europe. 2012; 20, 4(93): 96-101.

11. Lee, H.J. et al., "Antibacterial Effect Of Nanosized Silver Colloidal Solution On Textile Fabrics", Journal of Materials Science. 2003, 3:2199 – 2204.

12. Duran, D. and Kadoglu, H., "Gümüş/Pamuk Karışımlı İpliklerin değişen Voltajlardaki Elektriksel İletkenlik Davranışları", XII. Uluslararası İzmir Tekstil ve Hazır Giyim Sempozyumu 28 – 30 Ekim, 2010.

13. Duran, D. and Kadoglu, H., "Electromagnetic shielding characterization of conductive woven fabrics produced with silver-containing yarns", Textile Research Journal. 2015, Vol. 85(10) 1009–1021.

14. Ozdil, N., Kayseri, G., O. and Menguc, G., S., Analysis of Abrasion Characteristics in Textiles, Abrasion Resistance of Materials, Dr Marcin Adamiak (Ed.), ISBN: 978-953-51-0300-4, InTech. 2012, 119-146.

15. Candan, C. and Onal, L., Dimensional, Pilling, and Abrasion Properties of Weft Knits Made from Open-End and Ring Spun Yarns, Textile Research Journal. 2002, 72(2), 164-169.

16. Kumpikaitė, E., Ragaišienė, A. and Barburski, M., Comparable Analysis of the End-Use Properties of Woven Fabrics with Fancy Yarns. Part I: Abrasion Resistance and Air Permeability, Fibres & Textiles in Eastern Europe. 2010, Vol. 18, No. 3 (80) pp. 56-59.

17. Babaarslan, O. and Telli, A., A Study On Usage Of Chenille Yarn In Denim Fabric Production, Journal of Textiles and Engineer. 2013, 20: 92, 1-10.

18. Omeroglu, S. and Ulku S., An Investigation about Tensile Strength, Pilling and Abrasion Properties of Woven Fabrics Made from Conventional and Compact Ring-Spun Yarns, Fibres & Textiles in Eastern Europe. 2007, Vol. 15, No. 1 (60).

Technology

19. Tekeoğlu O. and Kavuşturan Y., Abrasion, Bursting and Bending Properties of Plain Knitted Fabrics from Chenille and Macaroni Yarns, Uludag University Journal of Faculty of Engineering and Architecture. 2007, 12(2).

20. Ulku, S., Ortlek, H.G. and Omeroglu, S., (2003). The Effect of Chenille Yarn Properties on the Abrasion Resistance of Upholstery Fabrics, Fibres & Textiles in Eastern Europe. 2003, Vol. 11, No. 3 (42).

21. Tasci Z.,K., The Design Of Silver Content Woven Fabric For Shirts, Production and Investigation Under Industrial Conditions, Cukurova University, Institute of Natural and Applied Sciences Department of Textile Engineering, MSc Thesis, ADANA, 2013.

22. TS EN ISO 12947-3, Textiles - Determination of the Abrasion Resistance of Fabrics by the Martindale Method - Part 3: Determination of mass loss.

23. Tascı, Z.K., and Celik N. Investigation of Some Quality Parameters of Yarns Produced with Cotton and Silver Fiber, 1. International Mediterranean Symposium, (MERSIN), 1-3 November 2018, Vol 6, 297.

# EFFECT OF PVA BASED SIZING MATERIAL ON TENSILE AND FRICTIONAL PROPERTIES OF RING-, SIRO-, COMPACT- AND COMPACT SIROSPUN YARNS

of Materials and Engineering Technology

## MEMIK BUNYAMIN UZUMCU <sup>\*1</sup>, TUBA BEDEZ UTE<sup>2</sup>, PINAR CELİK<sup>2</sup>, AYSEGUL EKMEKCI KORLU<sup>2</sup>, HUSEYIN KADOGLU<sup>2</sup>

 <sup>1</sup> Gaziantep University, Fine Arts Faculty, Fashion and Textile Design Department, Gaziantep, TURKIYE.
 <sup>2</sup> Ege University, Engineering Faculty, Textile Engineering Department, Izmir, TURKIYE.

#### Abstract

Even if new spinning systems or improvements in conventional systems allows us to produce yarns with higher quality properties, sizing is still crucial for weaving processes, in order to ensure fabric quality and uninterrupted production [1]–[5]. In this context, choosing the proper sizing material and sizing conditions is important. These settings could be different for different yarn types thanks to their different structures. In this study, Ne 20 yarns were produced in different spinning systems which were; ring, siro, compact and compact siro systems. Tensile strength, yarn-metal and yarn-yarn friction tests were done according to standards. Tensile strength results indicated that sizing increased this feature for all yarn types. Yarn-yarn friction differences for sizing concentration were not statistically significant. Yarn-metal friction results showed us that sizing material we used, which was a type of PVA initially increased this friction.

Keyword: Sizing, PVA, spinning systems, tensile strength, yarn-yarn friction, yarn-metal friction

### 1. Introduction

Special requirements of new type fabrics generate the need for yarns with better properties. This could be carried out by using different fibers and/or their blends or using different spinning systems which allow spinning yarns with different properties. In this context, if we want to change the properties of a yarn from same raw material, changing system or parameters of spinning is the only choice left. Since the introduction of ring spinning system in 1700s, which constitutes of yarn spinning, a lot has changed in this system and also new spinning systems were developed. Some of these modern spinning systems were made with alterations/modernizations in ring spinning systems. Siro, solo, compact, core and compact siro spinning systems are among these new systems.

Weaving process has three main movements, which are shedding, weft insertion and beat-up. In two of these three movements (shedding and beat-up) yarns are subjected to yarn-metal friction. Moreover, yarns in a loom is constantly under tension [1].

Warp yarns should be sized for gaining fine warping and efficient weaving. In some weaving mills, plied yarns are used without sizing, but this causes efficiency loss in weaving. Warp threads are subjected to tension and friction in the weaving machines as they pass through dropper, healds and reed, in the shedding and sley movements. As a result of these tension and friction, warp yarns are abraded and worn. Repeated movements cause abrasion on the yarn surface, so that sizing is carried out in order to increase the resistance of these threads.

Effects of sizing on weavability, causes of warp thread breakages, effects of sizing on warp breakages and different size types were investigated in various studies. This study focuses on the effects on frictional properties of yarns [2–5].

In this study, the effect of sizing concentration on tensile and frictional properties of yarns which were produced with different spinning systems was investigated. Within the scope of the study, the yarns were produced in the ring, siro, compact and compact-siro systems, and sized with the aqueous dispertion of PVA (non-ionic) prepared in different concentrations. Yarns were subjected to tests before and after sizing process. Test results were evaluated and the effects of sizing concentration on yarn quality were examined.

#### 2. Materials and Methods

In this study, 100% cotton warp yarns were produced in spinning machines at Ege University Textile Engineering Department's short staple yarn mill. Properties of the cotton used in this study, which were tested using HVI for raw cotton and AFIS for rovings produced with this raw cotton, are given in table 1. Two spinning machines were utilized for yarn productions. For minimizing the machine effects on yarn properties, same (12.000 rpm for spindle speed) or similar machine parameters (due to machine limitations) were used in productions. Ring and siro yarns were produced in Pinter Merlin SpA 1803 ring spinning machine, compact and compact siro yarns were produced in Rieter K45 compact spinning machine with yarn count of Ne 20 and twist coefficient of  $\alpha e 4.5$ . The winding process was performed in Schlafhorst Autoconer X5 winding machine at 1000 m/min winding speed in Ege University Textile Engineering Department's weaving mill; so that the sizing process would be done in package form and the IPI faults would be checked and cleaned before further investigations.

HVI r	esults of raw cotton	AFIS results of rovings	
SCI	222	Nep cnt/g	20
MIC	4,1	SCN cnt/g	0
STR	45,1	L(W) (mm)	25,7
LEN	35,2	L(W)(mm) %CV	0,0
UNF	88,2	L(W) %CV	31,9
SFI	11,1	SFC (w) %<12,7	4,1
ELG	4,7	UQL (w)	30,7
CSP	2603	L(n) (mm)	22,6
CG	22,1	cv L(n)(mm)	0,0
Rd	76,8	L(n) %CV	37,3
(+b)	10,5	SFC (n) %<12,7	10,8
Mat	87	5.0% (mm)	35,5
		5.0% (mm) %CV	0,0
		Fine mTex	193
		IFC %	3,4
		Mat ratio	1,00
		Total Cnt/g	18
		Dust Cnt/g	18
		Trasg Cnt/g	0
		VFM (%)	0,01

Table	1.	Fiber	properties
		1 10 01	properties



After winding the yarns, sizing step was carried out. For this application, aqueous dispertion of PVA (non-ionic) was selected as sizing material. This sizing material is a cold sizing material type. Polyvinyl alcohol is a water soluble polyhydroxy polymer which can be produced by hydrolysis of polyvinyl acetate and it is biodegradable. This polymer type is mostly used in textile sizing processes due to its strength, adhesion, flexibility and film forming properties [6]. KajiSeisakusho KS7 Unisizer machine was used for sizing, in Bez Textile's mill, according to the sizing prescription created. Three sizing concentrations were used, which were 4%, 6% and 8%. Sizing was carried out at the temperature of 55 °C.



Figure1. Kaji Seisakusho KS7 Unisizer

The main focus of this study was to determine more information about frictional properties of PVA sized yarns. However, we also tested different yarn properties, which were tensile strength and breaking elongation. Results of these will be given later in the results section for further information. Before tests, all yarn types were conditioned according to the standard. Lawson Hemphill CTT (Constant tension transport) device was used for yarn- metal pin friction and yarn-yarn friction tests. This dynamic transport system (Figure 2) has the ability to maintain specific yarn tension and let the yarn run at selected speed [7]. Tensile strength and breaking elongation tests were done with Lloyd LRX, according to the standards.



Figure 2. Lawson Hemphill CTT

The data obtained as a result of the tests foretold were analyzed, statistically. statistics software was used in these analyses. ANOVA test was performed and the information is presented in the results section.

## 3. Results and discussion

In this part, test results of sized and unsized yarns are given, in order to see the effect sizing material created on yarns spun with different spinning systems. Results are given on Table 2.

The International Conference of Materials and Engineering Technology

			Sized			
	Yarns	Unsized	4%	6%	8%	
			concentration	concentration	concentration	
Tensile Strength (cN/tex)	Ring	23,76	24,23	25,69	25,55	
	Siro	24,15	26,25	27,96	26,09	
	Compact	24,00	26,22	27,76	31,18	
	Compact siro	25,93	24,43	27,94	27,2	
Breaking Elongation (%)	Ring	11,86	12,64	11,46	9,07	
	Siro	11,64	10,68	10,99	9,36	
	Compact	10,19	10,99	10,06	9,32	
	Compact siro	12,11	11,06	11,2	10,3	
Yarn-pin friction	Ring	0,3523	0,403333	0,403333	0,4	
	Siro	0,299667	0,403333	0,396667	0,4	
	Compact	0,340333	0,396667	0,396667	0,406667	
	Compact siro	0,382267	0,4	0,393333	0,383333	
Yarn-yarn friction	Ring	0.2085635	0.25	0.26	0.26	
	Siro	0.2165	0.256667	0.28	0.266667	
	Compact	0.2076667	0.25	0.27	0.26	
	Compact siro	0.2096667	0.26	0.28	0.26	

Table 2. Test results of unsized and sized yarns (cN/tex)

Test results showed us that highest tensile strength results belonged to compact siro spun yarns. This was an expected result, in view of the literature and our previous studies [8, 9]. However, after sizing with non-ionic aqueous dispertion of PVA, which we used in this study as sizing material, sirospun yarns had the highest tensile strength with 4% and 6% concentration of the sizing material. This could be caused by adhesion of sizing material to the yarn surface. Sirospun yarns more surface area compared to compact and compact siro-spun yarns, due to its much hairy structure than these yarn types. Moreover, when we increased the concentration to 8%, tensile strength of yarns were decreased, except compact spun yarns. So, it is possible to say that 6% sizing concentration is more suitable than 4% and 8% sizing concentration; especially for ring-, siro- and compact siro-spun yarns in terms of tensile strength, and of course while using this type of sizing material (Figure 3).


UWE Bristol

Figure 3. Tensile Strength results

Breaking elongation results of sized and unsized yarns are given on table 2. It is evident from the table and Figure 4 that sizing generally decreased breaking elongation with some exceptions. Higher concentrations caused lower breaking elongation. Compact sirospun yarns had, mostly, the highest breaking elongation.





Determining yarn-metal and yarn-yarn friction properties are important. Especially, in weaving operations yarns are subjected frictions with metal surfaces and other yarns. Higher friction may cause damage on yarn and negatively affect end product quality. For this reason, these properties of yarns produced with different spinning systems and sized with different



concentration were investigated in this study. Yarn-pin friction results of sized and unsized yarns are given in table 2.



Figure 5 Yarn-pin friction results

When the results were investigated, it was found that this type of sizing increased yarn-metal friction (Figure 5). Sizing concentration did not affect yarns produced with different systems same way. Most of the differences were not statistically significant. However, it was clear that sizing increased yarn-metal friction values. It can be said that sizing material's friction coefficient may be higher than yarn, because yarn is generally covered with the sizing material after sizing process (Figure 6). Moreover yarn hairs are glued on the yarn surface and lubricating effect of the yarn is gone and friction surface area is increased (Figure 6). Here, it was important whether this frictional force negatively affected the yarn quality, or not. In fact, yarn was covered by a film layer around it. Further investigation with yarns after friction is needed in order to determine the effect of these frictional forces. But, it can be said that, this type of sizing may negatively effect metal parts of the production line where these frictional forces happen, such as heald. However, sizing is still needed for yarn to withstand the tensions in weaving.



Figure 6. SEM images of unsized (a,c) and sized (b,d) yarns





Yarn-yarn friction results are given in table 2. Results were similar to yarn-metal friction test results. Sizing material initially increased friction between the yarns (Figure 7). This could only be explained by sizing material's friction coefficient with itself and metal surface being higher than cotton's friction coefficient or surface structure change with sizing material. Sizing with lower concentrations resulted with lower yarn-yarn friction. However, statistical analysis

771

showed that differences between different concentrations were not statistically significant (p=0,143>0,05). It was also determined that spinning system effect on yarn-yarn friction was not statistically significant (p=0,085>0,05).

aterials and Engineering Technology

We also abraded yarns with yarns using same cycle counts. After this application, SEM images were taken (Figure 8). These images clearly show that after same abrasion cyles unsized ring-spun yarn (Figure 8-a) was really deformed. Deformation on unsized siro-spun yarn (Figure 8-b) was limited in comparison with ring-spun yarn. But the best abrasion resistance result after applying abrassion with the same yarn type was sized ring-spun yarn (Figure 8-c). This indicated that selecting an appropriate spinning system is important as well as sizing parameters and sizing material.



**Figure 8.** Unsized ring-spun (a), unsized siro-spun (b) and sized ring-spun (c) yarns with different magnifications (1:100x and 2:300x for ring-spun and 500x for the rest) after abrasion.

# 4. CONCLUSIONS

In this study, effects of sizing concentration and spinning systems on frictional properties of sized yarns were investigated. In order to do this, four types of Ne 20 yarns were spun in ring, siro, compact and compact siro systems. Tests of these yarns were carried out according to standards and results were investigated using statistics software.

Unsized yarn test results for yarns produced with different spinning systems were as expected. The highest yarn tenacity belonged to compact siro yarn, while the lowest tenacity belonged to the ring yarns. After sizing, tests were carried out again. The effects of yarn type and sizing concentration on tensile strength and yarn-metal friction were found to be statistically significant (p<0,05). However, the effects of these parameters on yarn-yarn friction was not significant (p>0,05). According to the test results, it can be seen that the increase of the sizing concentration generally increased the tensile strength of the yarns as expected. Moreover, sizing with the sizing material we used for this study, also increased frictions of yarn surface with metal and another yarn. We will be investigating the reason of this in our further studies.

772



# ACKNOWLEDGEMENTS

This study was conducted as a part of Ege University Scientific Research Projects with the project number 16-MUH-045. We would like to thank Ege University and Scientific Research Projects Coordinatorship. We also would like to thank Bez Textile for letting us use their mill to perform sizing operations and SÖKTAŞ for helping us about the raw materials, for this study. We also would like to thank Mr. Veli Gökçil and AKSA Acrylic for their valuable helps during yarn testing.

The International Conference of Materials and Engineering Technology

# References

- 1. Adanur S. Handbook of Weaving. CRC Press, 2001.
- 2. Faasen NJ, van Harten K. The effect of sizing on the weavability of cotton yarns. J Text Inst Trans 1966; 57: T269–T285.
- **3.** Dolecki SK. The Causes of Warp Breaks in the Weaving of Spun Yarns. J Text Inst 1974; 65: 68–74.
- **4.** Hesketh B, Bradbury E. The Effect of Sizing on Warp Breakage. In: Journal of the Textile Institute Proceedings. Taylor & Francis Group, pp. 272–276.
- **5.** Sultana S, Haque M, Nur H. Preparation and application of different size materials on the cotton yarn and investigating the effect of sizing on the tensile properties of cotton yarn. Bangladesh J Sci Ind Res 2014; 49: 25–30.
- **6.** Kroschwitz JI, Mark HF. Encyclopedia of polymer science and technology. 8th ed. Wiley-Interscience, 2004.
- 7. Menguc GS. A Research on Yarn and Fabric Characteristics of Acrylic/Wool/Angora Blends. Tekst ve Konfeksiyon 2016; 26: 40–47.
- 8. Xie C, Liu X, Xu B, et al. Research on the Compact-Siro Spun Yarn Structure. Fibres&Textiles East Eur 2015; 23: 54–57.
- Uzumcu MB, Sari B, Oglakcioglu N, et al. A Comparison on Physical Properties of Ring-, Compact- and Compact Siro- spun Yarns Produced from Different Fibers. In: International Technological Sciences and Design Symposium- ITESDES. 2018, pp. 1500–1506.



of Materials and Engineering Technology

# Mohamed H. RABIE<sup>1</sup>, Mohammad K. HASSAN<sup>2</sup>, Soheb M. SALIM<sup>1</sup>, Eman M. FAYYAD<sup>2</sup>, Mohammad Refa'at A. Rahim IRSHIDAT<sup>2</sup>, Nasser Abdullah N J ALNUAIMI<sup>2</sup>, Amit K. SHARMA<sup>3</sup>, Jeffrey S. WIGGINS<sup>3</sup>

<sup>1</sup> Department of Civil and Architectural Engineering, Qatar University, Doha, Qatar (m.rabie@qu.edu.qa, Soheb.salim@qu.edu.qa)

<sup>2</sup> Center for Advanced Materials, Qatar University, Doha, Qatar (mohamed.hassan@qu.edu.qa, emfayad@qu.edu.qa, mirshidat@qu.edu.qa, anasser@qu.edu.qa)

<sup>3</sup> Department of Polymer Science and Engineering, The University of Southern Mississippi, Hattiesburg Mississippi, United States (Amit.Sharma@usm.edu, Jeffrey.Wiggins@usm.edu)

#### Abstract

Carbon fiber reinforced polymer (CFRP) composites consist of two components: reinforcement materials (e.g. carbon fiber); and matrix (i.e. epoxy resin). Due to their extraordinary mechanical strength along the fiber direction and good fatigue resistance characteristics, CFRP composites are widely used in many applications from aircraft parts to strengthening and rehabilitation of reinforced concrete (RC) structures in civil engineering. In this presentation, we will discuss the results of two examples from our recent work on CFRP composites in aerospace and civil engineering applications. One study describes the introduction of new toughening agent via covalent attachment of polyhedral oligomeric silsesquioxane (POSS) nanoparticles to the crosslinked epoxy matrix. The second study outlines the utilization of different types of epoxy to achieve improved bond strength and adhesion between the carbon fiber and the concrete beam structures.

**Keyword:** Keywords: Carbon fiber reinforced composites, adhesion strength, composite toughening, three-point flexural test

#### **Epoxy Toughening in Aircraft Parts:**

Epoxy matrices possess great advantages over other polymers due to their combination of excellent modulus, strength, thermal capacity, solvent resistance, and so on, while being affordable. However, epoxy resins are brittle and often require a toughening phase or agent in composite applications. Commonly tougheners added to epoxy include rubbers or thermoplastics [1, 2]. These additives complicate material processing and evaluation so that it is desirable to design epoxies that are intrinsically tougher without sacrificing stiffness or desired glass transition temperature (Tg) for a given application.

*Herein*, we discuss our efforts to control structure and morphology of epoxy–POSS networks in the interest of designing well defined hybrids. The POSS cages were pre-reacted to form POSS–epoxy adducts which were then crosslinked to form the toughened hybrid networks (Fig. 1). The effect of pre-reaction temperature will be illustrated in terms of our goal of increasing POSS reaction rate to make POSS incorporation and dispersion into the epoxy network more



competitive with undesirable POSS phase segregation. The bulky inorganic POSS cage is known to reduce molecular mobility of the epoxy matrix and promotes aggregation. We speculated that any modification to the POSS–epoxy system that increases the rate of POSS reaction will improve dispersion by making POSS reaction into the growing network more competitive with phase segregation. POSS–epoxy reaction kinetics was monitored in situ via dynamic dielectric sensing, gel permeation chromatography, and <sup>29</sup>Si NMR spectroscopy. The influence of POSS–POSS and POSS–epoxy interactions on morphology and dynamic mechanical properties, including storage modulus and glass and sub-glass transitions, versus pre-reaction temperature, was investigated. Dynamic mechanical analysis (DMA) showed that incorporation of POSS nanoparticles at 5 wt % caused a Tg-related  $\Box$ -relaxation peak of composites to shift to slightly lower temperatures relative to the control samples (Fig. 2). Storage modulus versus temperature curves for all pre-reaction temperatures do not differ greatly among themselves but lie beneath that of the control unfilled samples. Altogether, DMA indicates a form of plasticization most likely due to the insertion of extra free volume by bulky POSS units [3].



Figure 1. Scheme of POSS - epoxy adduct formation.



Figure 2. Dynamic mechanical properties of hybrid epoxy networks showing the effect of POSS incorporation on Tg (left) and storage modulus (right).



# **Evaluation of Bond Strength between CFRP Composites and Concrete Beams:**

RC members strengthened with CFRP composites provide a noticeable improvement to the load capacity of RC members. Despite the recent advancements in using CFRP composites for strengthening, mechanical properties of epoxy is considerably weaker than the fiber. Therefore, epoxy is considered as the controlling factor for enhancing the efficiency of RC structures' mechanical properties [4, 5].

In this presentation, we will discuss the results of our study on new CFRP composites made using two commercially available epoxies (EPON 828 and EPON 862) and used to reinforce concrete prisms. A three-point bending test was conducted to test the bond strength between carbon fiber reinforced polymers and concrete. In addition, scanning electron microscopy (SEM), mechanical tensile and differential scanning calorimetry (DSC) tests were conducted to explore the internal structure of the CFRP laminates. It was clearly observed that concrete was intact to CFRP laminates as shown in (Fig.3), which indicates the good adhesion between CFRP laminates and concrete. The experimental results showed that optimum epoxy compositions were in samples having 100% wt./wt. of EPON 828 and 70:30% wt./wt. EPON 828/EPON 862 mixture (Tab. 1). Both achieved improved bond strength and better adhesion between the carbon fiber and the concrete structure. In addition to its high glass transition which proves its effectiveness for better fire resistance.

Sample	Sample code	Average load (kN)
1	BISA	15.28
2	70A/30F	16.25
3	50A/50F	15.21
4	30A/70F	16.28
5	BISF	16.39

<b>LADIC I.</b> I ICAULAI COLICOULO	Table	1.	Flexural	test	results.
-------------------------------------	-------	----	----------	------	----------

Figure 3. Concrete fragment intact with CFRP laminate after testing

#### **Conclusions:**

CFRP composites are widely used and will continue to be used a broad range of applications to provide mechanical strength, durability and light weight when compred to analogue materials. However, many issues will remian open for research activities including adhesion strength between the fibers and matrix, adhesion with materials like concrete, crack propagation, and developing new types of high performance epoxy matrices.

Acknowledgments: The authors would like to acknowledge the Qatar University funding through grant #: QUCG-CAM-2018\2019-2. M. H. Rabie would also like to thank the Office



of Graduate Studies at Qatar University for their financial support, under the Graduate Research Assistantship Program.

# **References:**

Kinloch A. J., Young R. J. Fracture Behavior of Polymers; Applied Science: London, 1983.
 Rothon R. Particulate-Filled Polymer Composites; Longman: Essex, 1995.

**3.** Sharma A.K., Hassan M.K., Tu J., Mauritz K.A., Wiggins J.S. Kinetic studies of POSS–DGEBA precursors derived from monoamine functional POSS using dynamic dielectric sensing and nuclear magnetic resonance. J. Appl. Polym. Sci. **2018**, 135 (13), 45994-46009.

**4.** Baggio D., Soudki K., Noël M. Strengthening of shear critical RC beams with various FRP systems. Constr. Build. Mater. **2014**, 66, 634–644.

**5.** Cabral-fonseca S., Correia J.R., Custódio J., Silva H.M., Machado A.M., Sousa J. Durability of FRP - concrete bonded joints in structural rehabilitation : A review. Int. J. of Adhesion and Adhesives **2018**, 83, 153–167.



# BURAK ŞAHİN<sup>1</sup>, NİHAT YILDIRIM<sup>1</sup>, ABDULLAH AKPOLAT<sup>1</sup>

<sup>1</sup> Gaziantep University, Faculty of Engineering, Department of Mechanical Engineering, Gaziantep, TURKEY.

#### Abstract

Gears used in aerospace applications and helicopter transmission units require special attention and specific design procedures owing to requirement for high load carrying capacity, lower level of noise and vibration, and less mass. During meshing, gears are mainly exposed to contact stress at tooth flank and bending stress at tooth root region. For aerospace applications, these stress levels are significantly higher because of higher load/torque transmission compared to general applications. This study mainly focuses on asymmetric tooth profile gears which can be an alternative to symmetric one especially used in helicopter transmission units because of their high load carrying capacity and low weight.

**Keyword:** Asymmetric gears, Spur gears, Aerospace gears, Aviation gears, Helicopter gears, Gear design, Analysis

#### **1. Introduction**

Gears are significant power transmission elements used for many different applications from very simple systems to the most complex one. Gears are also used for aviation industry and aerospace applications.

Gears especially used for aerospace applications need to have high load carrying capacity with low level of noise and vibration. In contrast to demand for high load carrying capacity, gears preferred for aerospace applications must have low weight. For this purpose, efforts to make gears lighter in weight with higher load carrying capacity continues to be relevant.

Gears under torque experience two different kinds of stress mainly; contact stress at gear tooth surface and bending stress at tooth root region. Gear tooth root strength and tooth surface durability relates to the ability to resist tooth breakage under bending stress and pitting and scuffing under contact stress respectively.

In the design of heavily loaded transmission gears as used in aerospace applications, it is often necessary to increase bending strength (in other words decrease bending stress) while maintaining load carrying capacity or increase the load carrying capacity while maintaining bending stress/strength [1].

In a standard gear which has symmetrical teeth, both left and right sides of a gear tooth profile have the same geometry/shape and hence similar bending and contact strength capacities. However, in most practical cases, both the forward and backward rotations are not always used for power transmission [2-3].

Technology



#### 2. Materials and Methods

In many gear transmissions, a tooth load on one flank is significantly higher and is applied for longer periods of time than for the opposite one; an asymmetric tooth shape reflects this functional difference. With asymmetric gears, the standard symmetric tooling gear rack is modified by altering the pressure angle of one of its flanks [4].

Two sides of the gear tooth are functionally different for most gears. Even if one side (drive side) is significantly loaded for longer periods, the opposite side (coast side) is unloaded or slightly loaded for short duration only. Thus asymmetric tooth (Figure 1) is well suited for cases where the torque is transmitted mainly, in one direction, [5] as in the case of most aerospace applications.

In this study, asymmetric spur gear has been investigated for tooth root bending stress by modeling asymmetric tooth as two comparable representative symmetric tooth (Figure 2).



Figure 1. Symmetric-asymmetric involute gear tooth profile



Figure 2. Modelling asymmetric gear as two symmetric gears

Root stress has been calculated by modifying formulae already presented for symmetric tooth involute spur gears [6].

$$\sigma_{F0} = \frac{F_t}{b m_n} Y_F Y_S Y_\beta Y_B Y_{DT}$$
(1)

Form factor ( $Y_F$ ) and stress concentration factor ( $Y_S$ ) are calculated by following formulae developed by ISO [6]:

Technology

The International Conference of Materials and Engineering



The International Conference of Materials and Engineering Technology  $Y_{F} = \frac{\frac{6h_{Fe}}{m_{n}} \cos \alpha_{Fen}}{\left(\frac{S_{Fn}}{m_{n}}\right)^{2} \cos \alpha_{n}}$ (2)  $Y_{s} = (1.2 + 0.13L)q_{s}^{\left[\frac{1}{1.21 + \frac{2.3}{L}}\right]}$ (3) s<sub>Fn</sub> L= (4) h<sub>Fe</sub>  $q_s \!\!=\! \frac{s_{Fn}}{2\rho_F}$ (5)

Symbol	Unit	Explanation
Ft	Newton	Nominal tangential load
b	mm	Face width
m <sub>n</sub>	mm	Normal module
Y <sub>F</sub>	-	Form factor
Y <sub>S</sub>	-	Stress concentration factor
Y <sub>β</sub>	-	Helix angle factor
Y <sub>B</sub>	-	Rim thickness factor
Y <sub>DT</sub>	-	Deep tooth factor
$\alpha_{\text{Fen}}$	Radian	Load direction angle, relevant to direction of
-		application of load at the outer point of single pair tooth
		contact of virtual spur gears
α <sub>n</sub>	Degree	Normal pressure angle
h <sub>Fe</sub>	mm	Bending moment arm for tooth root stress relevant to
		load application at the outer point of single pair tooth
		contact
s <sub>Fn</sub>	mm	Tooth root chord at the critical section
$ ho_F$	mm	Radius of root fillet
qs	-	Notch parameter; $q_s = s_{Fn} / 2\rho_F$

#### Table 1. Parameters required for equations of 1-5

#### 3. Results and Discussion

Symmetric tooth profile gears are general in use for aerospace applications. But asymmetric tooth profile can be an alternative to symmetric one for power transmission of aviation systems. Firstly, some case studies for symmetric and asymmetric spur gears are presented in this section to make comparison of the results of reference paper [7] and current work. General gear parameters are presented in Table 2 whereas results for tooth root bending stress are given in Table 3.

780

FI



Obtained stress results of both studies are very close to each other (Table 3). It is easy to conclude that stress developed in asymmetric gears is lower than symmetric one under same loading and operating conditions.

Parameter	Value	Unit
Module	1.0	mm
Teeth number (driver gear)	20	-
Teeth number (driven gear)	20	-
Addendum coefficient	1.00	-
Dedendum coefficient	1.25	-
Cutter tip radius coefficient	fully rounded	-

Table 2. General gear parameters of Case S1, A1 and A2

Table 3. Com	parison of	tooth root	stress of	Case S1, A1	and A2 [7]

Gear Pair	Notation	Tooth root stress (MPa)	
		Ref 7]	Current study
S1	D20C20	26.83	26.30
A1	D30C20	24.60	23.70
A2	D25C20	25.30	24.50

Asymmetrical tooth spur gears have some significant advantageous compared to symmetric gears. Less bending stress is produced at tooth root region of asymmetric gears under same load value. On the other side, higher load carrying capacity is an outstanding feature for asymmetric gears in the case of same stress level.

# **References:**

- 1. Francesco, G. D., Marini, S. Asymmetric Teeth Bending Stress Calculation. Gear Technology, 2007(March/April): 52–55.
- 2. Francesco, G. D., Marini, S. Structural Analysis of Asymmetric Teeth: Reductions of Size and Weight. Gear Technology. 1997 (September/October): 16-22.
- 3. Deng, G., Tsutomu, N. Enhancement of Bending Load Carrying Capacity of Gears Using an Asymmetric Involute Tooth. The International Conference on Motion and Transmissions, Fukuoka, Japan. November 2001: 513-517.
- 4. Kapelevich A. L. Asymmetric Gears: Parameter Selection Approach. Gear Technology. 2012(June/July): 48-51.
- 5. Singh, V., Senthilvelan, S. Computer aided design of asymmetric gear. 13th National Conference on Mechanisms and Machines. December 12-13 2007.
- 6. ISO. International Organization for Standardization. Calculation of Load Capacity of Spur and Helical Gears- Part 3: Calculation of Tooth Bending Strength. **2007.**
- 7. Sekar, R. P., Muhtuveerappan, G. Estimation of Tooth Form Factor for Normal Ratio Asymmetric Spur Gear Tooth. Mechanism and Machine Theory, **2015**, 90(2015): 187-218.



## NAIME OZDEMIR<sup>1</sup>, ABDULCABBAR YAVUZ<sup>2</sup>, PERIHAN YILMAZ ERDOGAN<sup>1</sup>, HUSEYIN ZENGIN<sup>1</sup>

<sup>1</sup>Gaziantep University, Faculty of Science and Literature Department of Chemistry, Sehitkamil, 27310 Gaziantep, TURKEY.

<sup>2</sup> Gaziantep University, Engineering Faculty, Department of Metallurgical and Materials Engineering, 27310, Sehitkamil, Gaziantep, TURKEY.

#### Abstract

Nickel film was electrodeposited in a single step by applying constant potential to the platinum flag surface in ionic liquid solution containing nickel chloride salts. Electrochemically synthesized nickel based electrodes were transferred to alkaline and ionic liquid electrolytes. Electrochemical properties were also investigated in alkaline electrolytes with different concentrations. Electrodeposited films showed better electrochemical performance in alkaline electrolyte. It is seen that Faradaic redox reactions occur exactly in the electrolyte with the highest concentration (5 M KOH) in the electrochemical properties of the aqueous electrolyte at different concentrations. Electrodeposited nickel film cannot be used in supercapacitor applications in ionic liquid electrolyte since no Faradaic reaction occurs. The electrochemical properties of Ni-based films produced in a ionic liquid electrolyte containing nickel chloride electrodeposited to the platinum flag working electrode was studied.

Keyword: Pseudocapacitor, Aqueous Electrolyte, Ionic Liquid, Platinum Electrode

#### **1. Introduction**

Energy production that does not create environmental pollution is an important issue [1]. One of the best ways to reduce non-renewable energy is to produce energy from sustainable sources and store these energy by electrochemical methods. Examples of devices that work with electrochemical energy conversion are fuel cells, batteries and supercapacitors. The supercapacitors operating on the principle of electrochemical energy conversion have many advantages over other energy storage devices due to their power densities, charge-discharge rates and specific capacitances [2]. Carbon-derived materials [3], transition metal oxide/hydroxides [4-6] and conductive polymers [7] can be used as electrode material for supercapacitor applications. The electrochemical stability and energy densities of metal oxide/hydroxides in these electrode materials are higher than other electrode materials (conductive polymers and carbon-derived materials) [8]. Electrode materials are separated into their contents as pseudocapacitor and electric double layer capacitor according to the electrical charge storage mechanisms. Electric double layer capacitor is the electrostatic energy storage

Technology

The International Conference of Materials and Engineering Technology process that stores the charge at the interface between the electrode surface and the electrolyte. In pseudocapacitors, Faradaic redox reactions occur and the process between the electrolyte and the electrode [9].

15

Cyclic voltammetry is the technique used to study the Redox reactions (reduction and oxidation steps) of molecules [10]. The components of an electrochemical reaction in a cyclic voltammogram consist of the working electrode, the counter electrode and the reference electrode. Cyclic voltammetry technique is an electrochemical technique that measures the current occurring in the electrochemical cell under conditions determined by applying voltage to the working electrode [11]. Voltage is scanned from  $E_1$  to  $E_2$  between the working electrode and the reference electrode and corresponding current is measured between the counter electrode and working electrode. Electrolyte selection is an important factor affecting the electrochemical performance of the electrodes [12]. It is necessary that all of the faradaic reactions occur within the voltaic range determined by the selected electrolyte. If only the reduction step or only the oxidation step takes place in the reaction, the electrochemical performance of the synthesized electrode cannot be mentioned [13].

Our first objective in this report was to obtain a nickel-based film on the platinum surface by electrochemical method. The second objective was to investigate the electrochemical performance of the electrode synthesized using cyclic voltammetry technique for the usability of the nickel-based electrode for supercapacitor applications. Finally, electrochemical performance of nickel-based films synthesized on platinum surface in alkaline electrolyte at different concentrations was investigated.

# 2. Experimental studies

Three-electrode configuration system (VersaSTAT3-AMETEK, TN, the USA) was used for the electrochemical properties of the nickel electrodes. Cyclic voltammetry technique was used to investigate the electrochemical properties of nickel films electrodeposited on platinum surface. The nickel films were electrodeposited on the platinum working electrode with constant potential application. Nickel films were prepared from ionic liquid solution containing choline chloride and urea (1:2 mole ratios). Electrodeposition solution was prepared by adding 0.2 M NiCl<sub>2</sub> to the ionic electrolyte and stirring at 50°C. The electrochemical working cell consists of a three-electrode system consisting of a platinum surface (working electrode), a titanium-coated platinum flag (counter electrode) and a platinum wire (reference electrode). The platinum surface was placed in a saturated nitric acid solution before electrodeposition. The electrode was immersed in the saturated nitric acid solution and washed with deionized water and ethyl alcohol. Nickel metal was electrodeposited on the platinum surface applying constant voltage of -0.8 (200 sec). The nickel modified electrodes covered on the platinum surface were transferred to alkaline (1 M KOH) and ionic liquid to determine the electrolyte solution for electrochemical properties. Nickel films were transferred to different alkaline concentration from 0.01 M to 5 M KOH and their electrochemical properties were investigated. In the electrochemical properties, the operating voltage range was between -0.3 V and +0.5 V by cyclic voltammetry technique.

# 3. Results and Discussion

# 3.1. Nickel electrodeposition on Pt flag working electrode

The nickel film was potentiostatically deposited on a Pt flag electrode by applying the voltage of -0.8 for 200 seconds in ionic liquid medium. Figure 1 shows the chronocoulometric data of the growth of nickel-based film on a Pt flag electrode by electrochemical deposition at 65 °C in Reline solution. Figure 1 shows that the nickel film deposited on the Pt flag has a 1.8 C. The surface image shows of bare Pt flag electrode and nickel plated Pt flag electrode. Bare Pt flag electrode surface was bright. It is clear that nickel film was coated on the Pt flag electrode surface.

The Internationa



**Figure 1.**Chronoamperometric curve (Charge-Time) of the Ni electrodeposition by applying -0.8 V potential for 200 seconds on the Pt flag electrode in Reline. Images of a) uncoated and b) nickel coated Pt flag electrodes.

# **3.2.** Electrochemical properties nickel electrode in aqueous electrolyte

Figure 32 shows the cyclic voltammetry curve of the nickel film deposited on the Pt flag in KOH electrolyte at different concentrations at a scan rate of 20 mV s<sup>-1</sup>. Figure 32 shows

Technology

784



voltammetric curves between -0.3 V and 0.5 V in the 0.01 M (black line), 0.1 M (red line), and 5 M KOH (green line) electrolyte of the nickel deposited Pt flag electrode. When the electrochemical activity of the nickel-based film in electrolytes of different concentration is compared, it is seen that charge (the area under current vs. potential graph) was higher in 5 M KOH electrolyte (Figure 32, green line). The oxidation and reduction peaks of the film in the 5 M KOH electrolyte are clearly visible. However, there are no oxidation and reduction peaks when film was scanned in 0.01 M (Figure 32, black line) and 0.1 M (Figure 32, red line) KOH electrolyte. Therefore, the effect of electrolyte concentration on the activity of the film was determined.



Figure 32.Cyclic voltammetry curve of nickel film on Pt flag in KOH electrolyte at different KOH concentrations (0.01, 0.1, 5 M) at a scan rate of 20 mV s<sup>-1</sup>.

Figure 33 shows a photograph of the bare Pt electrode, the nickel-based film electrodeposited on the Pt electrode, and the film in a different concentration of KOH electrolyte. Figure 33a shows the image of bare Pt flag surface. It had a bright and shiny surface. The nickel film obtained by applying the -0.8 V potential to the Pt flag working electrode for 200 seconds shows that the nickel was coated on Pt (Figure 33b). The image of the nickel-plated Pt flag electrode after polarized in 0.01 M KOH is shown in Figure 33c. Surface photograph of electrodeposited nickel-based film after cycled in 0.1 M KOH electrolyte (see Figure 33d). The surface image of the nickel-based Pt flag electrode in 5 M KOH electrolyte is presented in Figure 33e. After removal of 5 M KOH from the electrolyte, the film appears darker.





Figure 33. Images of bare Pt electrode and nickel based film deposited on Pt electrode and cycled in KOH electrolyte having different concentrations. Image of a) bare Pt flag, b) nickel deposited Pt flag, c) nickel deposited Pt flag cycled in 0.01 M KOH electrolyte, d) nickel deposited Pt flag cycled in 0.1 M KOH electrolyte and e) nickel deposited Pt flag cycled in 5 M KOH electrolyte.

#### 3.3. Electrochemical properties nickel electrode in a non-aqueous electrolyte

A nickel-based film was obtained by applying a -0.8 Volt to the Pt flag working electrode for 200 seconds (**Hata! Başvuru kaynağı bulunamadı.**). This film was scanned between -0.3 and 0.5 V potentials in the ionic liquid electrolyte. Figure 34 shows the cyclic voltammetry curves of the obtained nickel based film in the Reline electrolyte at a scan rate of 20 mV s<sup>-1</sup>. When the cyclic voltammetry curves of the nickel based electrode are examined, they had an oxidation but not apparent oxidation and reduction peaks throughout the selected potential range. This indicates that there is no capacitive response of the electrode and no Faradaic reactions at the electrode and electrolyte interface.





Figure 35 shows a surface picture of bare platinum flag surface (panel a), nickel deposited Pt flag electrode (panel b) and nickel based electrode after removal from the ionic liquid electrolyte (panel c). It is clearly seen the differences between the uncoated Pt flag and the nickel deposited on the Pt flag electrode in Figure 35a and Figure 35b, respectively. When the obtained nickel-based Pt flag electrode was scanned in ionic liquid, hollow structures were formed on the surface of Pt flag as shown in Figure 35c. This hollow structure may be caused by the gas release and the film being removed from the surface.



Figure 35.Photos of a) bare platinum surface, b) nickel electrodeposited Pt working electrode surface and c) nickel-based electrode surface after cycling in ionic liquid electrolyte.

**4. Conclusions:** In this study, nickel-based films were synthesized in a low-cost electrochemical method in ionic liquid electrolyte containing choline chloride and urea. The nickel film was electrodeposited from ionic liquid electrolyte having NiCl<sub>2</sub> salt by chronoamperometric method to the platinum flag working electrode with a voltage of -0.8 (200 sec). Electrodeposited Ni/Pt substrate electrodes were placed in alkaline and ionic liquid electrolytes to determine their electrochemical properties. Redox reactions of nickel-based modified films in ionic liquid (Reline) electrolytes have not been fully performed. This is due to the fact that nickel films do not have a significant oxidation and reduction peaks. Electrodeposited Ni films were put into alkaline electrolyte with different concentrations (0.01



ME

Acknowledgments: Naime Özdemir and Perihan Yılmaz Erdoğan thank to the YOK-Turkey for the 100/2000 PhD Program.

#### **References:**

- 1. González, A.; Goikolea, E.; Barrena, J. A.; Mysyk, R. Review on Supercapacitors: Technologies and Materials. Renew. Sustain. Energy Rev. 2016, 58, 1189–1206.
- 2. Pang, J.; Mendes, R. G.; Bachmatiuk, A.; Zhao, L.; Ta, H. Q.; Gemming, T.; Liu, H.; Liu, Z.; Rummeli, M. H. Applications of 2D MXenes in Energy Conversion and Storage Systems. Chem. Soc. Rev. 2019, 48 (1), 72–133.
- 3. Iro, Z. S.; Subramani, C.; Dash, S. S. A Brief Review on Electrode Materials for Supercapacitor. Int. J. Electrochem. Sci. 2016, 11 (12), 10628–10643.
- 4. Wang, G.; Huang, J.; Chen, S.; Gao, Y.; Cao, D. Preparation and Supercapacitance of CuO Nanosheet Arrays Grown on Nickel Foam. J. Power Sources 2011, 196 (13), 5756-5760.
- 5. Vijayakumar, S.; Nagamuthu, S.; Muralidharan, G. Supercapacitor Studies on NiO Nanoflakes Synthesized through a Microwave Route. ACS Appl. Mater. Interfaces 2013, 5 (6), 2188–2196.
- 6. Godillot, G.; Guerlou-Demourgues, L.; Taberna, P.-L.; Simon, P.; Delmas, C. Original Conductive Nano-Co3O4 Investigated as Electrode Material for Hybrid Supercapacitors. Electrochem. Solid-State Lett. 2011, 14 (10), A139.
- 7. Wang, K.; Wu, H.; Meng, Y.; Wei, Z. Conducting Polymer Nanowire Arrays for High Performance Supercapacitors. Small 2014, 10 (1), 14-31.
- 8. Sundrival, S.; Kaur, H.; Bhardwaj, S. K.; Mishra, S.; Kim, K.-H.; Deep, A. Metal-Organic Frameworks and Their Composites as Efficient Electrodes for Supercapacitor Applications. Coord. Chem. Rev. 2018, 369, 15–38.
- 9. Zhang, Y.; Feng, H.; Wu, X.; Wang, L.; Zhang, A.; Xia, T.; Dong, H.; Li, X.; Zhang, L. Progress of Electrochemical Capacitor Electrode Materials : A Review. Int. J. Hydrogen Energy 2009, 34 (11), 4889–4899.
- Molina, Á.; Serna, C.; López-Tenés, M.; Moreno, M. M. Theoretical Background for the 10. Behavior of Molecules Containing Multiple Interacting or Noninteracting Redox Centers in Any Multipotential Step Technique and Cyclic Voltammetry. J. Electroanal. Chem. 2005, 576 (1), 9–19.



- Nicholas, P.; Pittson, R.; Hart, J. P. Development of a Simple , Low Cost 11. Chronoamperometric Assay for Fructose Based on a Commercial Graphite-Nanoparticle Modi Fi Ed Screen-Printed Carbon Electrode. Food Chem. 2018, 241 (February 2017), 122–126.
- Hall, P. J.; Mirzaeian, M.; Fletcher, S. I.; Sillars, F. B.; Rennie, A. J. R.; Shitta-Bey, G. 12. O.; Wilson, G.; Cruden, A.; Carter, R. Energy Storage in Electrochemical Capacitors: Designing Functional Materials to Improve Performance. Energy Environ. Sci. 2010, 3 (9), 1238–1251.
- 13. Guo, S.; Wang, E. Synthesis and Electrochemical Applications of Gold Nanoparticles. Anal. Chim. Acta 2007, 598 (2), 181–192.

# ELECTROPOLYMERIZED POLYANILINE FILMS FOR ELECTROCHROMIC AND SUPERCAPACITOR APPLICATIONS

of Materials and Engineering Technology

# PERIHAN YILMAZ ERDOGAN<sup>1</sup>, ABDULCABBAR YAVUZ<sup>2</sup>, NAIME OZDEMIR<sup>1</sup>, HUSEYIN ZENGIN<sup>1</sup>

<sup>1</sup> Gaziantep University, Department of Chemistry, Sehitkamil, 27310 Gaziantep, TURKEY
<sup>2</sup> Gaziantep University, Department of Metallurgical and Materials Engineering, Sehitkamil, 27310 Gaziantep, TURKEY

#### Abstract

In the present study, PANI film was electrodeposited on graphite substrate at room temperature by electrochemical method which is simple and cost effective. Polyaniline was obtained in an acidic solution by applying 1 V potential on the graphite substrate. The deposited polyaniline and the bare graphite electrode was then transferred into the H<sub>2</sub>SO<sub>4</sub> electrolyte. PANI-Graphite composite electrode was scanned between -0.4 V and 1.2 V by cyclic voltammetry technique in 1 M H<sub>2</sub>SO<sub>4</sub> electrolyte and it was found to have excellent electrochemical activity. Morphological and electrochemical properties of the obtained electrode for supercapacitor and electrochromic applications has been investigated. The colour change of the obtained PANI-Graphite composite electrode in acid electrolyte at different voltages was recorded. PANI-Graphite composite electrode has been found to have black and green colour in positive and negative potentials, respectively. The obtained PANI-graphite composite electrode can be used as electrodes of electrochromic devices. Electrochemical capacitive properties of PANI film were evaluated by using cyclic voltammetry method. The composite film showed the maximum specific capacitance of 700 mF cm<sup>-2</sup> in 1.0 M aqueous H<sub>2</sub>SO<sub>4</sub> electrolyte at a scan rate of 5 mV s<sup>-1</sup>. Electrochemical activity of the composite electrode in different electrolyte solutions was investigated.

**Keyword:** Conducting polymer, electrodeposition, polyaniline, supercapacitor, electrochromic devices.

#### **1. Introduction**

Supercapacitors have higher power and energy density, compared to batteries and dielectric capacitors, respectively [1]. Supercapacitors can be divided into three groups according to their mechanisms: pseudocapacitors, electrical double layer capacitors (EDLC) and hybrid capacitors [2]. EDLCs use carbon-based materials and have a long cycle life [3]. However, they may have relatively low capacitance compared to pseudocapacitors. Pseudocapacitors have much higher capacitance due to their oxidation and reduction properties [4]. The important factors determining the performance and capacitance of supercapacitors are electrode and electrolyte. The electrolytes used in the supercapacitors may be aqueous or non-aqueous. The non-aqueous electrolyte consists of mixtures of ionic salts. Aqueous electrolytes may have some advantage as water is polarizable, conductive and has a relatively low viscosity. In supercapacitors, solutions such as KOH, H<sub>2</sub>SO<sub>4</sub> and Na<sub>2</sub>SO<sub>4</sub> are used as aqueous electrolytes (5). Metal oxides, carbon-derived materials, conductive polymers and their composites can be

used as electrodes in supercapacitors [6]. Conductive polymers are promising electrodes in pseudocapacitors. Because they are easy to synthesize, cheap and flexible [7]. Polyaniline (PANI), known as conductive polymer, is one of the most widely used polymers. PANI provides high specific capacitance of pseudocapacitors due to multiple oxidation and reduction states.

In electrochromic devices during electrochemical oxidation and reduction processes, the colour change or reflection properties can be changed [8]. Conductive polymers are widely used in electrochromic devices. Particularly, polyaniline is remarkable due to its environmental stability and optical properties [9]. PANI films have been widely used as electrode material for electrochromic devices because colour change can be observed between certain potentials during the electrochemical redox process [10].

The aim of this study was to potentiostatically electrodeposite PANI on the graphite electrode surface and to demonstrate its electrochemical stability. Since the PANI layer deposited on the graphite surface has a high surface area, an increase in the capacitance of the electrode is expected. The morphology of the PANI coated and uncoated graphite electrode is obtained. Colour change was observed with the change of potential of PANI-Graphite composite electrode. A green film at about -0.4 V was obtained while a black film at about 0.8 V was obtained. Conductive, economical and uniform PANI-graphite composite electrode can be used as electrodes of electrochromic devices. The PANI-graphite composite electrode exhibited much higher electrochemical stability in aqueous acidic electrolyte compared to ionic liquid electrolyte.

## 2. Experimental Section

Sulphuric acid (H<sub>2</sub>SO<sub>4</sub>, Merck, 95%), aniline (C<sub>6</sub>H<sub>5</sub>NH<sub>2</sub>, Merck, 99%) and sodium sulphate (Na<sub>2</sub>SO<sub>4</sub>, Merck, 99%) were used without further purification. 0.1 M aniline was prepared in 1 M sulphuric acid solution. The graphite sheet electrode was washed with deionized water only. Electrochemical measurements were made using a potentiostat device with a three-electrode system (VersaSTAT 3, the USA). Graphite sheet was used as working electrode, titanium coated platinum sheet was used as secondary electrode and Ag-AgCl (3.5 M KCl) electrodes were used as reference electrode in three electrode cells. Graphite sheet working electrode was immersed in aniline containing sulphuric acid solution and polyaniline electrodeposition was grown by applying 1 V potential. 1 M H<sub>2</sub>SO<sub>4</sub> electrolyte was used for electrochemical characterization of the obtained electrode. Cyclic voltammogram curves were recorded at different scan rates and their specific capacitances were calculated. The electrodedeposition and characterization of the resultant films were performed at room temperature ( $22 \pm 2$  ° C).

#### **3. Results and Discussion**

PANI film was potentiostatically electrodeposited on a graphite sheet electrode by applying 1 V potential for 100 seconds in acidic medium. Figure 36 shows the chronoamperometric data of the aniline growth in sulphuric acid solution at 25  $^{\circ}$ C.





The effects of potential on the PANI-graphite electrode surface were investigated. Figure 37 shows the PANI film deposited on a graphite sheet electrode showing the appearance of colour change in the sulphuric acid solution at different potentials. When the potential ranges from -0.6 to 1.2 V, it is clear that the PANI film on the graphite electrode surface provides visible electrochromism with colour changes from green to black. PANI film obtained by applying 1 V potential on the graphite plate electrode is green colored at about -0.4 V in H<sub>2</sub>SO<sub>4</sub> electrolyte, while a black film around +0.8 V is obtained.



**Figure 37.** Image of the PANI film electrodeposited on the graphite sheet electrode, colour change in sulphuric acid solution at different potentials.

Figure 38 illustrates the cyclic voltammetry curve of PANI in 1 M H2SO4 electrolyte to study the electrochemical behaviour of the PANI deposited graphite sheet electrode (red line) and a bare graphite sheet electrode (black line). In

Figure **38**, the electrochemical activity of the bare graphite sheet electrode (black line) in the 1 M H2SO4 electrolyte is very low compared to the PANI coated electrode (

Figure **38**, red line). Oxidation or reduction peaks of the bare graphite sheet electrode (

Figure **38** black line) were observed in the acidic medium even though it was low. A redox peak was observed in the acidic medium of the PANI coated graphite sheet electrode (

Figure 38, red line). The redox current of the PANI coated graphite sheet electrode (

Figure **38**, red line) is significantly increased compared to the bare graphite sheet electrode (

Figure **38**, black line) because the PANI film deposited on the surface of the bare graphite sheet electrode has a large and active surface area. The redox peaks in the acidic solution of the PANI coated graphite prove electroactivity of PANI. Thus, when the PANI coated graphite sheet electrode is placed in the H2SO4 solution and begins to scan between -0.4 V and 1.2 V as shown in

Figure **38**, the oxidation and reduction peaks of PANI can be observed.



**Figure 38.** Cyclic voltammetry curve of the bare graphite electrode (black line) and the PANI deposited graphite sheet electrode (red line) in the H2SO4 electrolyte (at 50 mV s-1 scan rate).

Figure 39 shows the cyclic voltammetry curves of a PANI-based film in a 1 M  $H_2SO_4$  electrolyte at a scan rate of 5, 10, 20, 50 and 100 mV s<sup>-1</sup>. It is seen that the curves have oxidation and reduction peaks at all scan rates. Considering the data presented in Figure 39, it can be considered that the oxidation occurs at about 0.3 V and the reduction occurs at about 0.7 V. As scan rate increases, current density increases. PANI film can be in various oxidation and reduction forms. These redox reactions may contribute to the change of potential peaks.



Figure 39. Cyclic voltamogram of PANI-Graphite electrode at different scan rates.

Figure 40 shows the cyclic voltamogram curve of the PANI film deposited on the graphite sheet in different electrolytes. The electrochemical activity of the PANI film deposited on the graphite sheet in the ionic liquid (reline) electrolyte (Figure 40 red line) is negligible compared to other solutions (Na<sub>2</sub>SO<sub>4</sub>, H<sub>2</sub>SO<sub>4</sub>). Oxidation and reduction peaks of composite films in Na<sub>2</sub>SO<sub>4</sub>



solution (Figure 40 black line) small. Oxidation and reduction peaks in  $H_2SO_4$  solution are obvious (Figure 40 blue line). Electrochemical activity in  $H_2SO_4$  electrolyte is quite high. Therefore,  $H_2SO_4$  was preferred as the electrolyte.



Figure 40. Cyclic voltammetry curve of PANI film deposited on graphite sheet in different electrolytes (at 50 mV s-1 scan rate).

Figure 41 shows the specific capacitance of the PANI deposited graphite sheet electrode at different scan rate (5, 10, 20, 50 and 100 mV s<sup>-1</sup>). High capacitance of energy storage materials is important in terms of power densities. As shown in Figure 41, the specific capacitance of the PANI deposited graphite sheet electrode varies inversely with the scan rate. The specific capacitance increases as the scan rate decreases. At low scan rate, the PANI film is in contact with the electrolyte. Therefore, more ion / electron transfer may occur at the electrode-electrolyte interface, resulting in higher capacitance at a low scan rate. The specific capacitance was found to be 700 mF cm<sup>-2</sup> at a scan rate of 5 mV s<sup>-1</sup>. At a scan rate of 100 mV s<sup>-1</sup>, it is 190 mF cm<sup>-2</sup>.



Figure 41. Variation of specific capacitance of PANI deposited graphite sheet electrode with respect to scan rate.

#### 4. Conclusions

It is important to obtain high surface area electrodes for supercapacitors and electrochromic devices. In this study, PANI was electropolymerised by applying 1 V potential to the graphite sheet electrode immersed in aniline solution in acidic medium for 100 seconds. The PANI deposited graphite electrode was then transferred to a 1 M H<sub>2</sub>SO<sub>4</sub> electrolyte. The PANI-graphite composite electrode was oxidized at +0.3 V in 1 M H<sub>2</sub>SO<sub>4</sub> at the forward direction and reduced at 0.7 V in the reverse direction. The PANI on graphite sheet electrode has a specific capacitance of 700 mF cm<sup>-2</sup> at a scan rate of 5 mV s<sup>-1</sup>. The colour of PANI-Graphite composite electrode has been found to have black and green colour in positive and negative potentials respectively. The obtained PANI-graphite composite electrode can be used as electrodes for electrochromic devices. The electrochemical activity of the composite electrode in different electrode at electrochemical activity of the composite electrode in different electrolyte solutions was investigated.

**Acknowledgments:** Perihan Yilmaz Erdogan and Naime Ozdemir wish to thank YOK-Turkey for 100/2000 scholarship program.

# **References:**

- 1. Burke, A. Ultracapacitors: Why, How, and Where Is the Technology. *J. Power Sources* **2000**, *91* (1), 37–50.
- 2. Marin S. Halper James C. Ellenbogen. Supercapacitors: A Brief Overview. *Biosens. Bioelectron.* **2010**, 25 (7), 1629–1634.
- Mensah-Darkwa, K.; Zequine, C.; Kahol, P. K.; Gupta, R. K. Supercapacitor Energy Storage Device Using Biowastes: A Sustainable Approach to Green Energy. *Sustain*. 2019, 11 (2).
- Fan, Z.; Chen, J.; Cui, K.; Sun, F.; Xu, Y.; Kuang, Y. Preparation and Capacitive Properties of Cobalt–nickel Oxides/Carbon Nanotube Composites. *Electrochim. Acta* 2007, 52 (9), 2959–2965.
- 5. Lota, K.; Sierczynska, A.; Acznik, I.; Lota, G. Effect of Aqueous Electrolytes on Electrochemical Capacitor Capacitance. *Chemik* **2013**, *67* (11), 1138–1145.



- Cai, J.; Niu, H.; Li, Z.; Du, Y.; Cizek, P.; Xie, Z.; Xiong, H.; Lin, T. High-Performance Supercapacitor Electrode Materials from Cellulose-Derived Carbon Nanofibers. ACS Appl. Mater. Interfaces 2015, 7 (27), 14946–14953.
- 7. Shown, I.; Ganguly, A.; Chen, L.; Chen, K. Conducting Polymer-based Flexible Supercapacitor. *Energy Sci. Eng.* **2015**, *3* (1), 2–26.
- 8. Mortimer, R. J.; Dyer, A. L.; Reynolds, J. R. Electrochromic Organic and Polymeric Materials for Display Applications. *Displays* **2006**, *27* (1), 2–18.
- Alam, M.; Ansari, A. A.; Shaik, M. R.; Alandis, N. M. Optical and Electrical Conducting Properties of Polyaniline/Tin Oxide Nanocomposite. *Arab. J. Chem.* 2013, 6 (3), 341– 345.
- 10. Le, T. H.; Kim, Y.; Yoon, H. Electrical and Electrochemical Properties of Conducting Polymers. *Polymers (Basel).* **2017**, *9* (4).

# ELECTROCHEMICAL BEHAVIOUR OF TIN BASED FILM CATHODICALLY DEPOSITED FROM NON-AQUEOUS MEDIA

aterials and Engineering Technology

# SITKI AKTAS<sup>1</sup>, ABDULCABBAR YAVUZ<sup>2</sup>, KAAN KAPLAN<sup>3</sup>, METIN BEDIR<sup>3</sup>

 <sup>1</sup> Department of Physics, University of York, York, YO10 5DD, UNITED KINGDOM
 <sup>2</sup> Gaziantep University, Engineering Faculty, Metallurgical and Materials Engineering Department, Sehitkamil, 27310 Gaziantep, TURKEY
 <sup>3</sup> Department of Engineering Physics, Faculty of Engineering, Gaziantep University, Sehitkamil, 27310 Gaziantep, TURKEY

## Abstract

Platinum electrode was coated with Sn in order to study electrochemical behaviour of Sn based modified electrode in acidic electrolyte. Sn was firstly electrodeposited potentiostatically on platinum in an ionic liquid containing  $Sn^{2+}$  salt. A deep eutectic solvent consisting of ethylene glycol and choline chloride (called Ethaline) was used as deposition electrolyte by applying different voltage and time at two different temperature (room temperature and 55 °C). Tin coated platinum electrode was immersed in Na<sub>2</sub>SO<sub>4</sub> and KOH solutions for the formation of tin oxide and tin hydroxide, respectively. Electrochemically treated films (tin oxide and tin hydroxide) were cycled in acidic electrolyte. SnO and Sn(OH)<sub>2</sub> coatings were stable in Na<sub>2</sub>SO<sub>4</sub> and KOH electrolytes, respectively. As oxidation and reduction peaks of tin oxide and tin hydroxide modified electrode in H<sub>2</sub>SO<sub>4</sub> was low both modified electrodes could not be electroactive in acidic media. Tin hydroxide and tin oxide films cycled in H<sub>2</sub>SO<sub>4</sub> were not electroactive and these films cannot be used in acidic media for electrochemical devices. Sn electrode electrodeposited at high temperature was more than 500 times thicker than that at room temperature. Thick tin modified electrode electrodeposited in Ethaline at high temperature had the same behaviour in neutral and alkaline solution as thin Sn film obtained at room temperature. Thick SnO and Sn(OH)<sub>2</sub> films were not electroactive in acidic media the same as thin ones. Sn electrode electrodeposited from deep eutectic solvent at 55 °C and treated with acidic solution was cycled in alkaline electrolyte. Reduction of the resulted film in KOH appeared. This electrode could be used for hydrogen evolution reaction in alkaline media.

Keyword: Electrodeposition. Ionic liquid. Tin, Tin oxide, Tin hydroxide.

#### **1. Introduction**

Electrodeposition can be used to coat metals and alloys. Electrolytes (changing additives) and deposition conditions (applied potential, time, and temperature) are critical for electrodeposition [1]. Different electrolyte and deposition conditions have been researched for the electrodeposition of tin and tin alloys [2]. Electrodeposition could be applied either constant potential (potentiostat) or constant current (galvanostat). Pulsed electrodeposition could also be used to obtain metals and alloys. As electrodeposition is a simple and inexpensive method, it is widely used in industry for metal plating. The thickness and surface morphology of coating could be tailored by changing deposition parameters [3]. It is well known that mass of metals/alloys (reduction of materials in electronics) is directly proportional to charge passed through electrode (Faraday's Law) [4]. Nucleation and growth of tin have been studied in detail depending on additives and deposition methods [5].

Sn and its alloys have been studied in the electronic industry instead of Pb which is highly toxic. Sn has been used as an alternative solders [6]. Tin coatings can protect a surface of a bulk materials from corrosion [7]. Tin and tin alloys have can be used as anode materials in lithium ion batteries instead of graphite [8]. Metals including steel, copper have been coated by tin and tin alloys in order to improve not only corrosion resistivity but also to the appearance of bulk materials.

Electrolytes used to electrodeposit tin and its alloys could be aqueous and non-aqueous solution. Aqueous electrolytes commonly used for tin growth are acidic or alkaline. Non-aqueous electrolytes can be ionic liquids which have studied commonly last decade [9]. Tin is electrodeposited from  $Sn^{2+}$  electrolyte by applying direct current. It is known that alkaline baths having hydroxide ions could be used without adding ligands. However, complexing agents are necessary to add acidic bath used for Sn deposition [10]. In this study an ionic liquid which has only ions have been used to obtain Sn. Generally current efficiency is low when aqueous bath is used. However, current efficiency for metal/alloy deposition in ionic liquid is high [11]. A deep eutectic solvent has been used as ionic liquid electrolyte for Sn electrodeposition in this work.

Deep eutectic solvent consist of a quaternary ammonium salts and hydrogen bond donor. Physical properties of ionic liquids and deep eutectic solvents are similar [12]. Tin and tin alloy (with zinc) were electrodeposited form choline chloride based deep eutectic solvent (with ethylene glycerol and urea) containing Sn salt [13]. Structure, morphology and deposition kinetic of tin obtained from ionic liquid were different than these of tin electrodeposited from aqueous solution [14]. However, electrochemical behaviour of resulting films were not studied in different media.

Hydroxides and oxides form of manganese, cobalt, nickel are commonly studied for energy storage devices (supercapacitor and batteries) [15]. However, the study of energy storage devices regarding tin based materials are limited. In this work, Sn was electrodeposited from a deep eutectic solvent and it was treated in aqueous electrolyte to obtain SnO and Sn(OH)<sub>2</sub>. After hydroxide and oxide form of Sn were obtained, they were transferred to acidic media in order to understand their

# 2. Materials and Methods

All experiments were carried out using Versastat 3 (AMATEK, Princeton Applied Research, USA). Pt flag and platinum coated mesh were used as working and counter electrode, respectively. Platinum working electrode was used because the potential window which is selected to study electrochemical behaviour of tin based coating is not overlapped by the reaction of Pt. Silver wire and silver-silver chloride reference electrodes were used in nonaqueous ionic liquid and aqueous electrolyte. All deposition potentials reported here is against the Ag wire reference electrode because ionic liquid was used for electrodeposition. Sn coated Pt electrodes electrodeposited from ionic liquid were transferred to aqueous solutions (alkaline, acidic and neutral) for polarisation treatment and cycling. Silver-silver chloride reference electrode having saturated potassium chloride was used in aqueous electrolytes. Before each electrodeposition of Sn, Pt electrodes were immersed in saturated HNO<sub>3</sub> in order to remove all coating from the surface. Then, the electrode was rinsed with deionized water and by ethanol. During the potentiostatic deposition, two constant potentials were applied at room temperature for 300 sec and at 55 °C for 500 sec in Ethaline. Ethaline was prepared by mixing one part choline chloride with two part ethylene glycol at 50 °C for 45 minutes. The Sn<sup>2+</sup> solutions were prepared by dissolving tin chloride in Ethaline. Chronoamperometric and chronocoulometric data of Sn growth were obtained. Polarization of electrodes was performed operating at a scan sweep of 20 mV s<sup>-1</sup> at room temperature.



# 3. Results and Discussion

Sn was electrodeposited from ethylene glycol and choline chloride mixture. This mixture is called Ethaline which is one of a deep eutectic solvent. As the electrochemical behaviour of tin coating was aimed to study, the substrate was platinum flag because platinum generally is electroinactive in different electrolyte at wide potential window [16]. It is known that deep eutectic solvent can be used for metal and alloy deposition generally at high temperate [17]. However, Sn was deposited on a platinum working electrode at room temperature. The deposition potential of -0.9 V was applied to platinum electrode immersed in Ethaline electrolyte containing 100 mM SnCl2 for 300 seconds. The deposition potential was against Ag wire reference electrode because water in Ag-AgCl reference electrode can contaminate ionic liquid. Chronoamperometric and choronocolumetric responses of platinum electrode in Ethaline ionic liquid are presented in Figure 1. Current density of electrode was around 55 µA  $cm^{-2}$  as soon as -0.9 V was applied and current density increased to 10  $\mu$ A cm<sup>-2</sup> after around 150 seconds. The response of current density of the electrode became linear after that point. Negative current can occur when a reduction reaction occurs. Negative current shown in Figure 1 indicates that tin was electrodeposited on the substrate. Ethaline was used as a deposition electrolyte because hydrogen evolution does not occur when deposition potential around -1 V was applied. Hydrogen evolution reaction during electrodeposition can cause the growth of metal hydroxide especially in aqueous solutions and decrease current efficiency. Charge density of tin deposition (blue line of Figure 1) was approximately 3.2 mC cm<sup>-2</sup> after 300 seconds. Charge was calculated from integration of current time graph. After Pt was polarised cathodically in Ethaline solution by application of -0.9 V, the surface became black indicated that Sn coated Pt surface. Tin coated platinum electrode was transferred to different aqueous electrolyte for electrochemical characterisation.

The International Confer



Figure 1: Chronoemperometry and chronocouloumetery of Sn growth on Pt working electrode from Ethaline containing 0.1 M SnCl2 by applying -0.9 V for 300 seconds at room temperature.

Sn modified Pt electrode was immersed firstly in Na<sub>2</sub>SO<sub>4</sub> solution in order to obtain a form of tin oxide. Cyclic voltammetry response of Sn coated Pt electrode in Na<sub>2</sub>SO<sub>4</sub> electrolyte is given in Figure 2a. In aqueous electrolyte the reference electrode was Ag-AgCl having saturated KCl solution as it is more stable than Ag wire which was used during electrodeposition of Sn in ionic liquid media. Oxidation of Sn started at around 0.25 V and reached to around 8  $\mu$ A cm<sup>-2</sup> at 0.7 V (see the first cycle of Figure 2a). Although an obvious oxidation of Sn was observed, a reduction related to that oxidation was not seen because tin film was oxidised but not reduced. The oxidised form of Sn was stable in Na<sub>2</sub>SO<sub>4</sub> electrolyte. Sn was cycled again in the same electrolyte to elucidate its electrochemical response and that time oxidation was also not observed. Tin oxide coated platinum electrode was taken out and washed with deionised water

Technology



and immersed in H<sub>2</sub>SO<sub>4</sub> for cycling. Cyclic voltamogram of SnO coated Pt is shown in Figure 2b. Oxidation and reduction reaction of the modified electrode in H<sub>2</sub>SO<sub>4</sub> was low (around 2  $\mu$ A cm<sup>-2</sup> at 0.7 V). It can be said that SnO modified electrode is not electroactive in acidic media. Tin was firstly electrodeposited on platinum and that electrode was treated in Na<sub>2</sub>SO<sub>4</sub> electrolyte to obtain tin oxide. As tin oxide film cycled in H<sub>2</sub>SO<sub>4</sub> was not electroactive, tin oxide films cannot be used in acidic media for electrochemical devices.



**Figure 2**: a) Cyclic voltamogram of Sn (electrodeposited on Pt from a deep eutectic solvent at room temperature) in 0.5 M Na<sub>2</sub>SO<sub>4</sub>. b) Cyclic voltamogram of SnO (Sn treated in Na<sub>2</sub>SO<sub>4</sub> given in panel a of this figure) was immersed in 0.1 M H<sub>2</sub>SO<sub>4</sub>. Sweeping speed for both cyclic voltammetry is 20 mV s<sup>-1</sup>.

A freshly prepared Sn modified Pt electrode (its typical growth response in Ethaline is presented in Figure 1) was transferred to a new aqueous KOH solution to oxidise tin to tin hydroxide by cathodic polarisation before cycling in acidic medium. It was shown in Figure 2 that tin oxide was not electroactive in acidic media. In order to observe if Sn(OH)2 is electroactive or not in acidic media, Sn coated Pt electrode was firstly polarised in alkaline media to obtain tin oxide based electrode. Cyclic voltammetry response of Sn modified electrode in KOH is presented in Figure 3a. As KOH was also an aqueous electrolyte, Ag-AgCl having saturated KCl was used as a reference electrode. Immediate application of 0 V to Sn electrode caused oxidation which could be the formation of tin hydroxide (around 0.03 mA cm<sup>-2</sup>) and had a plateau-like current response from 0.1 V to 0.5 V (approximately 0.02 mA cm<sup>-2</sup>) (shown in the first cycle of Figure 3a). An obvious oxidation of Sn was observed also here but a reduction peak was not seen as tin film was oxidised and tin hydroxide film was formed. The hydroxide based tin film became stable in KOH electrolyte. Sn coated Pt electrode was scanned again in alkaline electrolyte to understand its electrochemical behaviour. Oxidation of tin hydroxide film was again observed at around 0.45 V but this is not related to the transformation of tin to tin hydroxide. It is more related to oxygen evolution reaction because bubbles were formed on Sn based Pt electrode. Tin hydroxide based modified electrode was washed with deionised water after cycled in alkaline media and the treated film was immersed in H<sub>2</sub>SO<sub>4</sub> in order to understand its electrochemical properties in acidic media. Cyclic voltamogram data of Sn(OH)2 film is shown in Figure 3b. Oxidation and reduction peaks of tin hydroxide modified electrode in H<sub>2</sub>SO<sub>4</sub> was also low (around 2 µA cm<sup>-2</sup> at 0.7 V). Cyclic voltamogram of Sn(OH)<sub>2</sub> in H<sub>2</sub>SO<sub>4</sub> (Figure 3b) was the same as that of bare Pt in H<sub>2</sub>SO<sub>4</sub>. Therefore, It can be said that Sn(OH)<sub>2</sub> modified electrode was not electroactive in acidic media. Tin was firstly electrodeposited on platinum and that electrode was treated in KOH electrolyte to obtain tin hydroxide. As tin hydroxide film



cycled in  $H_2SO_4$  was not electroactive, tin hydroxide films cannot be used in acidic media for electrochemical devices.



**Figure 3:** a) Cyclic voltamogram of Sn (electrodeposited on Pt from a deep eutectic solvent at room temperature) in 1 M KOH. b) Cyclic voltamogram of Sn(OH)<sub>2</sub> (Sn treated in KOH given in panel a of this figure) was immersed in 0.1 M H<sub>2</sub>SO<sub>4</sub>. Sweeping speed for both cyclic voltammetry is 20 mV s<sup>-1</sup>.

Sn was electrodeposited from ethylene glycol and choline chloride mixture at room temperature (Figure 1) and oxidised in Na<sub>2</sub>SO<sub>4</sub> to form tin oxide (Figure 2a). Tin oxide was cycled in H<sub>2</sub>SO<sub>4</sub> (Figure 2b) and it was not electroactive in acidic media. Tin deposited platinum electrode was also immersed in KOH solution (Figure 3a) and tin hydroxide was formed and then tin hydroxide was cycled again in acidic electrolyte (Figure 3b). All these coatings electrodeposited from choline chloride and ethylene glycol were thin. Sn was electrodeposited from Ethaline solution at high temperature in order to obtain electroactive tin based film in acidic media. The substrate was again platinum the same as Figure 1. Deep eutectic solvents are usually used for metal and alloy deposition at high temperate (REF). The deposition potential of -0.6 V was again applied to platinum electrode which was immersed in Ethaline media having 0.1 M SnCl2 for 500 seconds. The time applied to electrodeposit Sn at high temperature was longer than that at room temperature (Figure 1) because it was aimed to obtain thicker film. Chronoamperometric and choronocolumetric responses of platinum electrode in choline chloride and ethylene glycol mixture (Ethaline) are shown in Figure 4. Current density of electrode was 5 mA cm<sup>-2</sup> immediately after the application of -0.6 V (see Figure 4) which was about 100 times greater than the current density of electrode obtained at room temperature. The current density reached to around 2 mA cm<sup>-2</sup> after approximately 50 seconds and cathodic current increased to about 10 mA cm<sup>-2</sup> after 500 seconds. There was not hydrogen evolution and current was directly related to the reduction of  $Sn^{2+}$  in Ethaline to tin metal. Cathodic current density (negative) shown in Figure 4 indicates that Sn was electrodeposited on Pt working electrode at high temperature. Charge density of tin deposition at room temperature (blue line of Figure 1) was about 3.2 mC cm<sup>-2</sup>. However, charge density of tin deposition at high temperature (blue line of Figure 4) was 1750 mC cm<sup>-2</sup>. It means that the Sn film electrodeposited in Ethaline at 55 °C was 500 times thicker than that at room temperature. After Pt was polarised cathodically in Ethaline solution by application of -0.6 V at 55 °C, the modified electrode was transferred to different aqueous electrolyte for electrochemical characterisation.



Figure 4: Chronoemperometry and chronocouloumetery of Sn growth on Pt working electrode from Ethaline containing 0.1 M SnCl2 by applying -0.6 V for 500 seconds at 55 °C. b) Cyclic voltamogram of Sn (electrodeposited on Pt from a deep eutectic solvent at room temperature) in 1 M KOH. c) Cyclic voltamogram of Sn(OH)<sub>2</sub> (Sn treated in KOH given in panel b of this figure) was immersed in 0.1 M H<sub>2</sub>SO<sub>4</sub>. Sweeping speed for panel b and panel c of this figure is 20 mV s<sup>-1</sup>.

Sn electrodeposited from Ethaline at high temperature (shown in Figure 4a) was cycled firstly in alkaline solution (1 M KOH) to transform thick Sn to Sn(OH)<sub>2</sub>. It was shown in Figure 3b that thin tin hydroxide was not electroactive in acidic media. In order to observe if thick Sn(OH)<sub>2</sub> could be electroactive or not in acidic media, thick Sn coated Pt electrode was firstly oxidised in alkaline media to obtain tin hydroxide. Cyclic voltammetry data of Sn modified electrode electrodeposited from hot (55 °C) Ethaline in KOH is given in Figure 4. Immediate application of -0.3 V to Sn electrode caused oxidation which could be the formation of tin hydroxide (around 1.4 mA cm<sup>-2</sup>) and had a plateau-like current response from -0.2 V to 0.5 V (approximately 6 mA cm<sup>-2</sup>) (shown in the first cycle of Figure 4b). The behaviour of thick Sn coating in KOH (Figure 4) was the same as that of thin Sn in KOH presented in Figure 3a but the only different is the high current density value of thick Sn. An oxidation of Sn with absence of reduction was again seen (Figure 4) similar to thin Sn film shown in Figure 3a. The hydroxide based thick tin film was stable in KOH electrolyte as repetitive cycles follow the same i-E route. Sn(OH)x coated Pt electrode was cycled in H<sub>2</sub>SO<sub>4</sub> electrolyte to compare its electrochemical behaviour with thin  $Sn(OH)_2$  in  $H_2SO_4$ . Before immersing the electrode in acidic media, it was washed with deionised water. Cyclic voltamogram data of Sn(OH)<sub>2</sub> film in acidic electrolyte is shown in Figure 4. Oxidation response of thick tin hydroxide film in H<sub>2</sub>SO<sub>4</sub> (around 6 µA cm<sup>-</sup>  $^{2}$  at 0.7 V) was greater than that of thin Sn(OH)<sub>2</sub> film in acidic media (around 2  $\mu$ A cm<sup>-2</sup> at 0.7 V) which could be related to oxygen evolution reaction (compare Figure 4c and Figure 3) because reduction of both Sn(OH)<sub>2</sub> electrode was similar. Sn(OH)<sub>2</sub> modified electrode was not



electroactive in acidic media. Tin was electrodeposited on platinum at room temperature and 55 °C. These electrodes were treated in KOH electrolyte to obtain tin hydroxide. As tin hydroxide film cycled in  $H_2SO_4$  was not electroactive, thick and thin tin hydroxide films cannot be used in acidic media for electrochemical devices.



**Figure 5:** a) Cyclic voltamogram of Sn (electrodeposited on Pt from a deep eutectic solvent at 55 °C) in 0.5 M Na<sub>2</sub>SO<sub>4</sub>. b) Cyclic voltamogram of SnO (Sn treated in Na<sub>2</sub>SO<sub>4</sub> given in panel a of this figure) was immersed in 0.1 H<sub>2</sub>SO<sub>4</sub>. Sweeping speed for both cyclic voltammetry is 20 mV s<sup>-1</sup>.

Thick tin modified Pt electrode was immersed again in Na<sub>2</sub>SO<sub>4</sub> solution for the formation of SnO. Cyclic voltammetry data of Sn coated Pt electrode in Na<sub>2</sub>SO<sub>4</sub> electrolyte is presented in Figure 5a. Oxidation current of thick Sn electrodeposited from hot Ethaline was around 300 µA cm<sup>-2</sup> at 0.7 V in Na<sub>2</sub>SO<sub>4</sub> (see the first cycle of Figure 5a). This current density was more than 35 times greater than current density of thin Sn in Na<sub>2</sub>SO<sub>4</sub> (8  $\mu$ A cm<sup>-2</sup> at 0.7 V shown in Figure 2a). Although an obvious oxidation of Sn was observed, a reduction related to that oxidation was not that high. The oxidised form of Sn was stable in Na<sub>2</sub>SO<sub>4</sub> electrolyte. Sn was cycled acidic electrolyte to elucidate its electrochemical response. Before cycling in H<sub>2</sub>SO<sub>4</sub>, thick tin oxide coating was washed with deionised water. Cyclic voltamogram of thick SnO coated Pt is shown in Figure 5b. Oxidation and reduction reaction of the modified electrode in H<sub>2</sub>SO<sub>4</sub> was low (around 0.1 mA cm<sup>-2</sup> at 0.8 V). The cycle voltamogram of SnO in  $H_2SO_4$  (Figure 5b) was similar to the 2nd and 3rd cycles of Sn in Na<sub>2</sub>SO<sub>4</sub> (Figure 5a). Thick or thin SnO modified electrode is not electroactive in acidic media. Thick and thin Sn electrodeposited on platinum from deep eutectic solvent at high (55 °C) and room temperature was treated in Na<sub>2</sub>SO<sub>4</sub> electrolyte to obtain tin oxide. As tin oxide film cycled in H<sub>2</sub>SO<sub>4</sub> was not electroactive, tin oxide films cannot be used in acidic media for electrochemical devices.


**Figure 6:** a) Cyclic voltamogram of Sn (electrodeposited on Pt from a deep eutectic solvent at 55 °C) in 0.5 M H<sub>2</sub>SO<sub>4</sub>. b) Cyclic voltamogram of tin based electrode (Sn treated in H<sub>2</sub>SO<sub>4</sub> given in panel a of this figure) was immersed in 1 M KOH. Sweeping speed for both cyclic voltammetry is 20 mV s<sup>-1</sup>.

Sn modified electrode was also treated with  $H_2SO_4$  and then cycled in KOH. Thick Sn film electrodeposited from a deep eutectic solvent at 55 °C (similar to the film shown in Figure 4) was cycled in acidic media (see Figure 6a). Cycling started at -0.2 V and quickly oxidation was observed at around 80 mA cm<sup>-2</sup>. This could be dissolution of Sn as tin could be dissolved in acidic media. As the main aim of this work is to characterise electrochemical behaviour of Sn based film in different electrolytes, the mechanism of Sn coated Pt in acidic media was not studied in detail. Instead Sn film which was treated in acidic media. Cyclic voltammetric response of Sn based film (after polarised in acidic media) in KOH electrolyte is shown in Figure 6b. Oxidation peak of Sn based film in KOH was not observed but strong reduction of the film in KOH appeared due to probably of hydrogen evolution. This kind of electrode could be used for hydrogen evolution reaction in alkaline media which is an important issue for fuel cell applications. However, Sn electrodeposited on Pt from deep eutectic solvent at high (55 °C) and treated in H<sub>2</sub>SO<sub>4</sub> electrolyte was not electroactive in KOH electrolyte.

#### 4. Conclusions

Sn was electrodeposited on platinum substrate from Ethaline ionic liquid (ethylene glycol and choline chloride mixture) containing 100 mM SnCl2 by applying -0.9 V for 300 seconds. Tin coated platinum electrode was immersed in different aqueous electrolyte to understand its electrochemical behaviour. As it is known that Sn can be dissolved in acidic media, Sn was firstly electrodeposited from a deep eutectic solvent and then treated with Na<sub>2</sub>SO<sub>4</sub> (neutral) and

alkaline solution. Sn coating was immersed firstly in Na<sub>2</sub>SO<sub>4</sub> solution to form tin oxide. An oxidation of Sn was observed but a reduction was not appeared as Sn was oxidised but not reduced and Sn was stable in Na<sub>2</sub>SO<sub>4</sub> electrolyte. SnO coated modified electrode was immersed in H<sub>2</sub>SO<sub>4</sub>. As oxidation and reduction reaction of the modified electrode in H<sub>2</sub>SO<sub>4</sub> was low, SnO modified electrode is not electroactive in acidic media.

of Materials and Engineering Technology

Sn was electrodeposited on platinum and was treated in KOH electrolyte to obtain tin hydroxide.  $Sn(OH)_2$  coating became stable in KOH electrolyte. As oxidation and reduction peaks of tin hydroxide modified electrode in  $H_2SO_4$  was low  $Sn(OH)_2$  modified electrode was not electroactive in acidic media. As tin hydroxide and tin oxide film cycled in  $H_2SO_4$  were not electroactive, tin oxide and hydroxide films cannot be used in acidic media for electrochemical devices.

Thick Sn was electrodeposited from Ethaline at 55 °C and hydrogen evolution did not occur. Mass of Sn film electrodeposited at high temperature was more than 500 times greater than that at room temperature. Sn coated Pt was oxidised in Na<sub>2</sub>SO<sub>4</sub> and tin oxide was formed. This thick tin oxide was cycled in H<sub>2</sub>SO<sub>4</sub> and it was not electroactive in acidic media. Thick tin modified Pt electrode was immersed in Na<sub>2</sub>SO<sub>4</sub> solution to form SnO. Sn film electrodeposited from hot Ethaline in Na<sub>2</sub>SO<sub>4</sub> electrolyte had higher oxidation current than Sn film electrodeposited from Ethaline at room temperature. Thick and thin SnO modified electrode are not electroactive in acidic media.

Sn modified electrode electrodeposited from deep eutectic solvent at 55 °C was treated with acidic solution and then cycled in alkaline electrolyte. Oxidation peak of Sn based film in KOH was not observed but strong reduction of the film in KOH appeared due to hydrogen evolution. This electrode can be used for hydrogen evolution reaction in alkaline media which is critical for fuel cell applications.

Acknowledgments: KK would like to thank to YOK for 100/2000 PhD scholarship. The work presented here was supported by Gaziantep University BAP (project number: MF.DT.19.16).

#### **References:**

- 1. Sano, H.; Sakaebe, H.; Matsumoto, H. Observation of Electrodeposited Lithium by Optical Microscope in Room Temperature Ionic Liquid-Based Electrolyte. J. Power Sources **2011**, 196 (16), 6663–6669.
- Low, C. T. J.; Walsh, F. C. Electrodeposition of Tin, Copper and Tin–copper Alloys from a Methanesulfonic Acid Electrolyte Containing a Perfluorinated Cationic Surfactant. Surf. Coatings Technol. 2008, 202 (8), 1339–1349.
- 3. Bicelli, L. P.; Bozzini, B.; Mele, C.; D'Urzo, L. A Review of Nanostructural Aspects of Metal Electrodeposition. Int. J. Electrochem. Sci. **2008**, 3 (4), 356–408.
- 4. Tang, A.; Li, Z.; Wang, F.; Dou, M.; Liu, J.; Ji, J.; Song, Y. Electrodeposition Mechanism of Quaternary Compounds Cu2ZnSnS4: Effect of the Additives. Appl. Surf. Sci. **2018**, 427, 267–275.
- 5. Arafat, Y.; Sultana, S. T.; Dutta, I.; Panat, R. Effect of Additives on the Microstructure of Electroplated Tin Films. J. Electrochem. Soc. **2018**, 165 (16), D816–D824.
- 6. Neveu, B.; Lallemand, F.; Poupon, G.; Mekhalif, Z. Electrodeposition of Pb-Free Sn Alloys in Pulsed Current. Appl. Surf. Sci. **2006**, 252 (10), 3561–3573.
- Collazo, A.; Figueroa, R.; Nóvoa, X. R.; Pérez, C. Corrosion of Electrodeposited Sn in 0.01 M NaCl Solution. A EQCM and EIS Study. Electrochim. Acta 2016, 202, 288– 298.



- Soulmi, N.; Porras-Gutierrez, A.-G.; Mordvinova, N. E.; Lebedev, O. I.; Rizzi, C.; Sirieix-Plénet, J.; Groult, H.; Dambournet, D.; Gaillon, L. Sn (TFSI) 2 as a Suitable Salt for the Electrodeposition of Nanostructured Cu 6 Sn 5–Sn Composites Obtained on a Cu Electrode in an Ionic Liquid. Inorg. Chem. Front. **2019**, 6 (1), 248–256.
- 10. Mahapatra, S. Das; Dutta, I. Co-Electrodeposition of Tin with 0.2–20% Indium: Implications on Tin Whisker Growth. Surf. Coatings Technol. **2018**, 337, 478–483.
- 11. Abbott, A. P.; Frisch, G.; Ryder, K. S. Electroplating Using Ionic Liquids. Annu. Rev. Mater. Res. **2013**, 43, 335–358.
- 12. Smith, E. L.; Abbott, A. P.; Ryder, K. S. Deep Eutectic Solvents (DESs) and Their Applications. Chem. Rev. 2014, 114 (21), 11060–11082.
- Abbott, A. P.; Capper, G.; McKenzie, K. J.; Ryder, K. S. Electrodeposition of Zinc-tin Alloys from Deep Eutectic Solvents Based on Choline Chloride. J. Electroanal. Chem. 2007, 599 (2), 288–294.
- 14. Srivastava, M.; Yoganandan, G.; William Grips, V. K. Electrodeposition of Ni and Co Coatings from Ionic Liquid. Surf. Eng. **2012**, 28 (6), 424–429.
- 15. Cao, F.; Zhao, M.; Yu, Y.; Chen, B.; Huang, Y.; Yang, J.; Cao, X.; Lu, Q.; Zhang, X.; Zhang, Z.; et al. Synthesis of Two-Dimensional CoS1.097/Nitrogen-Doped Carbon Nanocomposites Using Metal-Organic Framework Nanosheets as Precursors for Supercapacitor Application. J. Am. Chem. Soc. **2016**, 138 (22), 6924–6927.
- Silvester, D. S.; Aldous, L.; Hardacre, C.; Compton, R. G. An Electrochemical Study of the Oxidation of Hydrogen at Platinum Electrodes in Several Room Temperature Ionic Liquids. J. Phys. Chem. B 2007, 111 (18), 5000–5007.
- 17. Abbott, A. P.; Ballantyne, A.; Harris, R. C.; Juma, J. A.; Ryder, K. S.; Forrest, G. A Comparative Study of Nickel Electrodeposition Using Deep Eutectic Solvents and Aqueous Solutions. Electrochim. Acta **2015**, 176, 718–726.



# ZİRKONYA İLE GÜÇLENDİRİLMİŞ LITHIUM SILICATE BİO-SERAMİK MALZEMESİNİN FARKLI AŞINMA MEKANİZMALARINDAKİ TRİBOLOJİK DAVRANIŞININ İNCELENMESİ

# Efe Çetin YILMAZ<sup>1</sup>

<sup>1</sup> Kilis 7 Aralık Üniversitesi, Mühendislik-Mimarlık Fakültesi, Makine Mühendisliği Bölümü, Kilis, Türkiye

# Özet

Bu çalışmanın amacı, zirkonya ile güçlendirilmiş lithium silicate bio-seramik malzemesinin direkt temas ve aşındırıcı ortam aşıma mekanizmalarında tribolojik davranışının incelenmesidir. Bu çalışmada, (Vita Suprinity; zirconia-reinforced lithium silicate) bio-seramik malzemesinden 10 adet 12 mm genişliğinde 13 mm çapında silindirik biçimde test numuleri üretildi. Test numuneleri saf su ve üçüncü aşındırıcı (haşhaş tohumu) ortamlarında 50 N aşınma kuveti, 0.7 mm yanal hareket, 100.000 mekanik yükleme, 6mm çapında Al<sub>2</sub>O<sub>3</sub> aşındırıcı karşı bilye ve 37 °C ortam sıcaklığında 2 eksenli aşınma testlerine maruz bırakılmıştır. Aşınma testleri sonrasında numunelerin aşınma bölgeleri temassız 3D profilometre ile analiz edilmiş ve meydana gelen aşınma hacim kayıpları belirlenmiştir. Ayrıca her iki test grubunda rastgele numuneler seçilerek aşınma bölgesinin mikroyapıları taramalı elektron mikroskobu (SEM) ile incelenmiştir. Bu çalışma kapsamında elde edilen veriler aşınma mekanizmasının lithium silicate bio-seramik malzemesinin aşınma davranışını önemli ölçüde etkilemediğini göstermiştir. Ancak, lithium silicate bio-seramik malzemesinin aşınma bölgelerindeki mikro yapı incelendiğinde özellikle üçüncü aşındırıcı (haşhaş tohumu) ortam aşınma testlerinde malzeme yüzeylerinde derin aşınma çizgilerinin meydana geldiği gözlemlenmiştir. Bu aşınma çizgileri, lithium silicate bio-seramik malzemesinin yüzey altı mikro çatlaklardan meydana geldiği düşünülmüştür.

Keyword: Bio-seramik, Aşınma, Hacim Kaybı, Mikro yapı

#### Giriş

Son yıllarda, klinik çalışmaların estetik taleplerine göre çeşitli bilgisayar destekli tasarım / bilgisayar destekli üretim CAD / CAM seramik malzemeleri geliştirilmiştir [1]. Bu alandaki seramik malzemelerin geliştirilmesi, malzemenin daha iyi mekanik, kimyasal stabilite ve estetik özelliklere sahip olmasını hedeflemektedir [2]. Vücut içerine yerleştirilen biomalzemeler sürekli olarak aşınma ve yorulma mekanizmalarına maruz kalabilmektedir. Bu bozunma mekanizmaları vücut içerisine yerleştirilen biyomalzemelerin mekanik, kimyasal ve estetik davranışlarını önemli ölçüde etkileyebilmektedir. Vücut içerisindeki bu sürekli ve karmaşık yapının canlı doku üzerindeki testlerinin çok uzun zaman alması ve maliyetli olması sebebiyle araştırmacılar laboratuvar ortam deneylerine yönelmişlerdir [3, 4]. Literatürde birçok araştırmacı çeşitli aşınma mekanizmaları ile canlı doku içerisine yerleştirilen bio-malzemelerin

Technology

yorulma ve aşınma mekanizmalarını modellemişlerdir [5-8]. Geliştirilen bu test yöntemleri ile vücut içerisine yerleştirilen bio-malzemlerin zaman periyotlarındaki mekanik, kimyasal ve estetik davranışları tahmin edilebilecek ve daha tatminkâr bir tedavi sürecinin oluşmasına katkı sağlanacaktır.

of Materials and Engineering Technology

Aşınma mekanizmaları, sürekli eşzamanlı darbe ve yanal sürtünme mekanizması varlığından dolayı çiğneme döngüsü testleri sırasında en göze çarpan bozunma mekanizmalarından biridir. Çiğneme hareketi sırasında taban ile karşı (antagonist) malzeme arasında dört temel aşınma mekanizması olduğunu söylemek mümkündür. vücut. Bu aşınma mekanizmasında, seramik veya kompozit taban malzemesi ve karşı (antagonist) malzeme, çiğneme döngüleri sırasında yanal hareketle doğrudan temas halindedir. Üç gövdeli bir aşındırıcı, iki antagonist yüzey arasında hareket eden bir aşındırıcı üçüncü vücut partikülünün varlığına neden olur. Bu aşınma mekanizmasının en belirgin özelliği, iki aşınma yüzeyi arasında, gıda parçacıkları gibi üçüncü bir aşındırıcı yüzeyin varlığıdır. Aşınma mekanizması yorulma aşınması, çiğneme hareketi sırasında tekrarlanan yükler nedeniyle kompozit malzemenin aşınma yüzeyinde alt yüzey çatlakları olarak tanımlanabilir. Çiğneme hareketi sırasında gıda parçacıklarından kaynaklanan asit ortamı aşındırıcı aşınma mekanizmasına neden olabilir. Bu asit ortamı, seramik ve karşı (antagonist) malzeme arasında kolayca taşınabilir [6].

# Materyal ve Yöntem

Bu çalışmada, dikdörtgen biçimindeki lithium silicate bio-seramik test numuneleri, aşınma test mekanizmalarına göre su soğutmalı düşük hızlı elmas testere (Isomet Buehler GmbH, Düsseldorf, Almanya) kullanılarak küp biçiminde kesilmiştir. Kesilen seramik numuneler silisyum karbür aşındırıcı kağıtlar kullanılarak cilalandı. Daha sonra kesilmiş seramik parçaları 13 x 12 mm'lik silindirik akrilik bir reçineye gömülmüştür. Şekil 1 akrilik rezin içerisine gömülmüş lithium silicate bio-seramik test numune örneğini göstermektedir.



Şekil 1. Aşındırıcı bilye ve akrilik rezin içerisine gömülmüş lithium silicate bio-seramik test numunelerinin gösterimi

Tüm test numuneleri saf su ve üçüncü aşındırıcı (haşhaş tohumu) ortamlarında 50 N aşınma kuveti, 0.7 mm yanal hareket, 10.000 mekanik yükleme, 6mm çapında Al<sub>2</sub>O<sub>3</sub> aşındırıcı karşı bilye ve 37 °C ortam sıcaklığında 2 eksenli aşınma testlerine maruz bırakılmıştır. Aşınma test deneyleri için bilgisayar-kontrollü birbirinden bağımsız 2 eksenli aşınma test cihazı kullanılmıştır. Şekil 2 2 eksenli bilgisayar kontrollü aşınma test cihazını göstermektedir.



Şekil 2. Birbirinden Bağımsız 2 Eksenli Aşınma Test Mekanizmasının Sistematik Gösterimi

Aşınma testleri sonrasında numunelerin aşınma bölgeleri temassız 3D profilometre ile analiz edilmiş ve meydana gelen aşınma hacim kayıpları belirlenmiştir. Aşınma hacmi kaybı ve test numunelerinin derinliği, x ekseninde 8  $\mu$ m, y ekseninde 12  $\mu$ m ve temassız profilometre kullanarak aşınma yüzeyinde 1000  $\mu$ m / s ölçüm hızı taramasıyla belirlendi. (Bruker-Contour GT 3D Vision64 simülasyon yazılımı). Ayrıca her iki test grubunda rastgele numuneler seçilerek aşınma bölgesinin mikro yapıları taramalı elektron mikroskobu (SEM) ile incelenmiştir.

#### Sonuç ve Tartışma

Bu çalışmada, zirkonya ile güçlendirilmiş lithium silicate bio-seramik malzemesinin direkt temas aşınma mekanizmasında 0.15 mm<sup>3</sup> (0.03), aşındırıcı ortam aşınma mekanizmasında 0.17 mm<sup>3</sup> (0.05) hacim kaybına uğramıştır. Şekil 3 direkt temas aşınma mekanizması test prosedüründen sonra lithium silicate bio-seramik malzemesinin aşınma bölgesinin non-contact 3D ve 2D analizlerini göstermektedir. Şekil 3 incelendiğinde lithium silicate bio-seramik malzemesinin aşınma bölgesinin dışındaki bölgelerde belirli bir yüzey pürüzlülük değerlerine sahip olduğu görülecektir. Bu durum lithium silicate bio-seramik malzemesinin kesme işlemi sırasında malzeme yüzeyinde meydana geldiği düşünülmüştür. Şekil 4 aşınma testi prosedürü sonrasında lithium silicate bio-seramik malzemesinin aşınma bölgesinden alınan mikro-yapı analizini göstermektedir.



Şekil 3. Direkt Temas Aşınma Mekanizması Test Prosedüründen Sonra Lithium Silicate Bio-Seramik Malzemesinin Aşınma Bölgesinin Non-Contact 3D Ve 2D Analizlerinin Gösterimi



Şekil 4. Aşınma Testi Prosedürü Sonrasında Lithium Silicate Bio-Seramik Malzemesinin Aşınma Bölgesinden Alınan Mikro-Yapı Analizinin Gösterimi

Literatürde, birçok çiğneme simülatör cihazının, direct temas ve aşındırıcı ortam aşınma mekanizmalarını modelleyebildiği rapor edilmiştir [5, 9-12]. Laboratuvar ortamında oluşturulan bu test mekanizmaları vğcut içerisine yerleştirilen biyomalzemelerin zaman



periyotlarında mekanik ve kimyasal davranışı tahmin edilebilecektir. Oral tribolojideki aşınma süreci karmaşık ve sabit bir yapıya sahiptir. Çiğneme işleminde, oral ortam, ısırma kuvveti miktarı, üçüncü vücut partikülü, yanal hareket mekanizması, antagonist malzemenin mekanik özelliği, kompozit malzemenin aşınma davranışını pozitif veya negatif olarak etkileyebilir. Literatürde aşınma simülasyonları için hangi malzemenin antagonist bir malzeme olarak kullanılması gerektiği konusunda bir anlaşma yoktur. OHSU ve ZURICH çiğneme testi yöntemleri, antagonist malzeme olarak enamel önermiş, diğer test yöntemleri ise, antagonist malzemeler olarak seramik, alüminyum oksit ve paslanmaz çelik, alümina ve zirkonya seramik topunu önermiştir[13-15].

Şekil 4 incelendiğinde aşındırıcı ortam aşınma mekanizmasında lithium silicate bio-seramik malzemesinin aşınma bölgesinde aşındırıcı parçacıklarının kaldığı görülecektir. Bu parçacıklar aşınma testi boyunca taban test malzemesi ve karşı (antagonist) malzeme arasında üçüncü bir ortam olarak aşındırıcı davranış göstermişlerdir. Bu aşındırıcı parçacıklar ayrıca mekanik yükleme sırasında aşınma yük dağılımını değiştirerek malzeme aşınma yüzeyine homejen olmayan bir aşınma yük dağılımının oluşmasına katkı sağlamışlardır. Bu nedenle, lithium silicate bio-seramik malzemesinin aşındırıcı ortam test mekanizmasında daha fazla hacim kaybına uğramasına katkı sağladığı söylenebilir. Ayrıca bu parçacıkların aşınma sırasında parçalanması ile korozif etkinin oluşması kaçınılmaz bir durumdur.

# Değerlendirme

Bu çalışma kapsamında elde edilen veriler ile aşağıdaki değerlendirmeler yapılabilir.

- Lithium silicate bio-seramik malzemesi her iki aşınma test mekanizmasında benzer aşınma dirençleri göstermiştir.
- Aşındırıcı ortam aşınma test mekanizmasında Lithium silicate bio-seramik malzemesinin aşınma yüzeyinde aşındırıcı parçacıkların varlığı mikroyapı analizinde gözlemlenmiştir. Bu parçacıkların lithium silicate bio-seramik malzemesinin aşınma yüzeyinde mikro çatlakların oluşmasına katkı sağladığı söylenebilir.
- Daha sonraki çalışmalarda aşındırıcı parçacıkların korozif etkisinin incelenmesi canlı doku üzerinde yapılan testlere önemli derecede katkı sağlayacaktır.

# Kaynakça

- **1.** Elsaka, S.E. and A.M. Elnaghy, *Mechanical properties of zirconia reinforced lithium silicate glass-ceramic*. Dental Materials, 2016. **32**(7): p. 908-914.
- 2. Marocho, S.M.S., et al., *Mechanical strength and subcritical crack growth under wet cyclic loading of glass-infiltrated dental ceramics*. Dental Materials, 2010. **26**(5): p. 483-490.
- **3.** Yilmaz, E.Ç., *Effect of Sliding Movement Mechanism on Contact Wear Behavior of Composite Materials in Simulation of Oral Environment*. Journal of Bio- and Tribo-Corrosion, 2019. **5**(3): p. 63.

- 4. Traini, T., et al., *The zirconia-reinforced lithium silicate ceramic: lights and shadows of a new material.* Dental Materials Journal, 2016. **35**(5): p. 748-755.
- **5.** Yilmaz, E.C. and R. Sadeler, *Investigation of three-body wear of dental materials under different chewing cycles*. Science and Engineering of Composite Materials, 2018. **25**(4): p. 781-787.
- 6. Yilmaz, E.C. and R. Sadeler, *Investigation of Two- and Three-Body Wear Resistance on Flowable Bulk-Fill and Resin-Based Composites*. Mechanics of Composite Materials, 2018. **54**(3): p. 395-402.
- 7. Injeti, V.S.Y., et al., *A comparative study on the tribological behavior of Ti-6Al-4V and Ti-24Nb-4Zr-8Sn alloys in simulated body fluid.* Materials Technology, 2018: p. 1-15.
- **8.** Osiewicz, M.A., et al., *Contact- and contact-free wear between various resin composites.* Dental Materials, 2015. **31**(2): p. 134-140.
- **9.** Wimmer, T., et al., *Two-body wear rate of PEEK, CAD/CAM resin composite and PMMA: Effect of specimen geometries, antagonist materials and test set-up configuration.* Dental Materials, 2016. **32**(6): p. E127-E136.
- Tkachenko, S., et al., Wear and friction properties of experimental Ti-Si-Zr alloys for biomedical applications. Journal of the Mechanical Behavior of Biomedical Materials, 2014. 39: p. 61-72.
- **11.** Yilmaz, E.C., *Effects of thermal change and third-body media particle on wear behaviour of dental restorative composite materials.* Materials Technology, 2019.
- **12.** Finlay, N., et al., *The in vitro wear behavior of experimental resin-based composites derived from a commercial formulation.* Dental Materials, 2013. **29**(4): p. 365-374.
- **13.** Heintze, S.D., *How to qualify and validate wear simulation devices and methods.* Dental Materials, 2006. **22**(8): p. 712-734.
- 14. Hahnel, S., et al., *Two-body wear of artificial acrylic and composite resin teeth in relation to antagonist material.* J Prosthet Dent, 2009. **101**(4): p. 269-78.
- **15.** Wassell, R.W., J.F. McCabe, and A.W. Walls, *Wear characteristics in a two-body wear test.* Dent Mater, 1994. **10**(4): p. 269-74.



# Efe Çetin YILMAZ<sup>1</sup>

<sup>1</sup> Kilis 7 Aralık Üniversitesi, Mühendislik-Mimarlık Fakültesi, Makine Mühendisliği Bölümü, Kilis, Türkiye

# Özet

Titanyum ve titanyum alaşımları malzemelerinin yüksek osseintagrayon (kemik ile bütünleşme kabiliyeti)' den dolayı son yıllarda implant malzemesi olarak sıkça tercih edilmektedir. Ancak, insan vücudu içerisine yerleştirilen malzemeler sürekli olarak eş zamanlı çeşitli yorulma ve aşınma mekanizmalarına maruz kalabilmektedir. Bu durum insan vücudu içerisine yerleştirilen biyomalzemelerin osseintagrasyon (kemik ile bütünleşme kabiliyeti) dışında yeterli oranda mekanik, kimyasal ve estetik özelliklere de sahip olmasını gerektirmektedir. Saf titanium malzemesinin intra-oral tribolojik süreçte aşınma davranışı genel anlamda yetersizdir. Araştırmacılar bu alanda saf titanyum malzemesine çeşitli kaplama ve alaşım oluşturma teknikleri üzerinde çalışmalarını yoğunlaştırmaktadırlar. Çalışmalar neticesinde titanyuma daha yeterli oranda aşınma ve yorulma davranışları kazandırabilmişlerdir. Bu çalışmanın amacı literatürde saf titanyum ve çalışma kapsamında bahsedilen titanyum alaşımlarının aşınma davranışlarının analizi yapılacaktır.

Keyword: Titanyum, Titanyum Alaşımları, Aşınma, Biyo-malzeme

#### Giriş

Ticari olarak saf titanyum ve titanyum alaşımları, iyi yorulma direnci, nispeten düşük elastiklik modülü ve mükemmel korozyon direnci nedeniyle, havacılık ve biyomedikal uygulamalarda yaygın olarak tercih edilmektedir. Bununla birlikte, metalik malzemeler biyomedikal uygulamalar için antagonist malzeme olarak kullanıldığında yetersiz aşınma direnci yüksek sürtünme katsayısına sahiptirler<sup>1</sup>. Yapılan çalışmalarda, biyo-malzemenin aşınma direncinin, malzemenin istenilen şartlarda çalışmasında önemli bir rol oynadığını göstermektedir. Bu nedenle, bir biyomalzenin aşınma direnci klinik olarak önemlidir. Literatürde, biyomalzemelerin tribolojik özelliklerini incelemek için çeşitli çalışmalar yapılmıştır<sup>2</sup>.

Metalik biyomalzemelerin mekanik performansları açısından yüksek güvenilirlikleri, "yapay kalça eklemleri, kemik plakaları ve diş implantları gibi sert dokuların değiştirilmesine yönelik tıbbi cihazların imalatı için" kullanımları ile sonuçlanmıştır <sup>2-3</sup>. Tıp alanında, çeşitli özellik ve özelliklerinden dolayı, birçok malzeme türü ve alaşım araştırılmıştır <sup>2</sup>. Paslanmaz çelik, Co alaşımları ve Ti alaşımları da dahil olmak üzere tıp alanında kullanılmak üzere farklı alaşım sistemleri geliştirilmiştir. Aşağıda ASTM standardında kayıtlı olan ve biyomedikal uygulamalar için geliştirilmiş olan alaşımların kimyasal bileşimini özetlemektedir (şekil 1-3)<sup>4</sup>.

Technology

Alloy	Microstructure
1. Pure Ti	(ASTM F67-89)
2. Ti-6Al-4V ELI (ASTM F136-84, F620-87)	$\alpha + \beta$ type
3. Ti-6Al-4V (ASTM F1108-88)	$\alpha + \beta$ type
4. Ti-6Al-7Nb (ASTM F1295-92, ISO5832-11)	$\alpha + \beta$ type(Swiss)
5. Ti-5Al-2.5Fe (ISO5832-10)	$\alpha+\beta$ type (Germany)
6. Ti-5Al-3Mo-4Zr	$\alpha + \beta$ type (Japan)
7. Ti-15Sn-4Nb-2Ta-0.2Pd	$\alpha+\beta$ type (Japan)
8. Ti-15Zr-4Nb-2Ta-0.2Pd	$\alpha+\beta$ type (Japan)
9. Ti-13Nb-13Zr (ASTM F1713-96)	near $\beta$ type (U.S.A.), Low modulus
10. Ti-12Mo-6Zr-2Fe (ASTM F1813-97	β type (U.S.A.), Low modulus
11. Ti-15Mo	β type (U.S.A.), Low modulus
12. Ti-16Nb-10Hf	β type (U.S.A.), Low modulus
13. Ti-15Mo-5Zr-3A1	β type (Japan), Low modulus
14. Ti-15Mo-2.8Nb-0.2Si-0.26O	β type (U.S.A.), Low modulus
15. Ti-35Nb-7Zr-5Ta	β type (U.S.A.), Low modulus
16. Ti-29Nb-13Ta-4.6Zr	β type (Japan), Low modulus
17. Ti-40Ta, Ti-50Ta	β type (U.S.A), High corrosion resista

**Şekil 1.** Biyo-malzeme olarak kullanılan titanium alaşımları<sup>4</sup>

Titanyum ve Titanyum alaşımlarının yüksek biyouyumluluğu, tıp ve dişçilik alanlarında giderek artan bir kullanım alanı bulmasını sağlamıştır <sup>5-7</sup>. Biyomedikal alanında kullanılan titanyum alaşımları düşük elastiklik modülü ve TiO<sub>2</sub> tabakasının getirdiği mükemmel korozyon direnci ile önümüzdeki yıllarda da giderek artan bir kullanım sahası bulacaktır.

Laboratuvar ortamında uygun bir test metodolojisi kullanarak insan vücudunda kullanılan biyomalzemelerin aşınma mekanizmaları modellenebilir. Literatürde ball-on disc, pin-on disc ve block-on disc metalik biyomalzemelerin tribolojik davranışını incelemek için en yaygın kullanılan yöntemlerdendir. Şekil 2 de bu aşınma mekanizmaları sistematik olarak özetlenmiştir. Ancak son yıllarda özellikle insan ağız içerine yerleştirilen implant malzemeleri çiğneme test deneyleri ile modelleyebilen araştırmacılar çalışmalar yapmıştır <sup>8-10</sup>.



**Şekil 2.** a: ball-on disc, b: pin-on disc ve c: block-on disc aşınma mekanizmalarının sistematik gösterimi <sup>2</sup>

İnsan vücuduna yerleştirilen metalik biyomalzemelerin tribolojik davranışları değerlendirmek için farklı karakterizasyon teknikleri kullanılmıştır. Bunlardan, enerjiyi dağıtıcı X ışını spektroskopisi (EDXS / EDS) gibi kimyasal analizler, kompozisyonu belirlemek için seçilen bölgeler için kullanılabilir. Aşınma mekanizmasını belirlemek veya bir son test incelemesi yapmak için bir taramalı elektron mikroskobu / ışık mikroskobu (SEM / LM) kullanılabilir Ayrıca literatürde aşınma hacim kaybı ve aşınma derinliği analizlerinde 3D non-contact profilometre kullanılmıştır <sup>11-12</sup>. Yüzey kimyasını incelemek için X-ışını fotoelektron spektroskopisi (XPS) kullanılabilir. Sürtünme, yüzey pürüzlülüğü, çizilme ve yapışma gibi farklı tribolojik olayları incelemek için literatürde bir çalışma da atomik kuvvet mikroskobu (AFM) da kullanılmıştır <sup>13</sup>. Bununla birlikte, elastiklik modülü ve sertlik gibi diğer mekanik özellikler, derinlik algılayan bir girinti sistemine sahip AFM kullanılarak ölçülebilir.

Li ve ark. <sup>14</sup>, Nb içeriğinin, yüzey modifikasyonunun, karşı malzemenin malzemesinin ve ısıl işlemin Ti-Nb-Ta-Zr ve Ti-6Al-4V (TAV1) alaşımlarının aşınma özellikleri üzerindeki etkilerini incelemişlerdir <sup>14</sup>. Yapılan çalışmada, Nb içeriğinin arttırılmasının aşınma direncini arttırdığını bulmuşlardır. Ayrıca ısıl işlem, Nb205'ten gelen oksit parçacıklarının oluşumu nedeniyle Ti-29Nb-13Ta-4.6Zr'nin (TNZT1) yıpranmaya karşı direncini arttırdığı sonucuna varmışlardır. Bununla birlikte aşındırıcı karşı yüzey malzemesinin (antagonist malzeme) aşınma kaybı üzerinde önemli bir etkisi olduğu bildirilmiştir. Şekil 3, farklı aşındırıcı malzemeler (antagonist) ile yapılan aşınma testlerindeki farklı alaşımların mikro-yapı morfolojisini analiz etmektedir <sup>15</sup>.



Şekil 3. Wear morphology of (a) TNZT1, (b) TNZT3, and (c) TAV1 sliding on a stainless steel plate; (d) TNZT1, (e) TNZT3, and (f) TAV1 sliding on UHMWPE; (g) TNZT1, (h) TNZT3, and (i) TAV1 sliding on a pig bone in 0.9% NaCl<sup>15</sup>

Literatürde yapılan araştırmalar neticesinde elde edilen sonuçlar doğrultusunda aşağıdaki değerlendirmeler yapılabilir;

- Saf titanyum ve alaşımları üstün mekanik ve kimyasal davranışları nedeniyle önümüzdeki yıllarda sıkça biyomalzeme olarak kullanılacaktır.
- Titanyum alaşımları ile saf titanyumun yetersiz aşınma davranışı iyileştirilmeye çalışılmaktadır. Literatürde bu çalışmalar ile daha optimum titanyum alaşımları elde edilecektir.
- Laboratuvar ortam aşınma test yöntemlerinin gelişmesi ile canlı doku üzerinde çok uzun zamanlarda yapılan testlerin daha kısa sürede yapılması bu analizleri hızlandıracaktır. Ancak laboratuvar ortam testlerinin canlı dokuyu modelleyebilmesi üzerindeki parametreler daha fazla simüle edilebildikçe elde edilen sonuçların geçerliliği yükselecektir. Aşınma test mekanizmasında bilgisayar-kontrollü çiğneme simülasyon testlerinin gelişimi ile diş hekimliği alanda kullanılan biyomalzemelerin çalışma ortamının daha geçerli simüle edilebileceği açıktır.

# Kaynakça:

1. Cvijovic-Alagic, I.; Cvijovic, Z.; Mitrovic, S.; Panic, V.; Rakin, M., Wear and corrosion behaviour of Ti-13Nb-13Zr and Ti-6Al-4V alloys in simulated physiological solution. *Corros Sci* 2011, *53* (2), 796-808.

The International Conference of Materials and Engineering Technology

- 2. Hussein, M. A.; Mohammed, A. S.; Al-Aqeeli, N., Wear Characteristics of Metallic Biomaterials: A Review. *Materials* 2015, *8* (5), 2749-2768.
- 3. Niinomi, M., Metallic biomaterials. J Artif Organs 2008, 11 (3), 105-110.
- **4.** Niinomi, M., Recent metallic materials for biomedical applications. *Metall Mater Trans A* 2002, *33* (3), 477-486.
- **5.** Ribeiro, A. L. R.; Caram, R.; Cardoso, F. F.; Fernandes, R. B.; Vaz, L. G., Mechanical, physical, and chemical characterization of Ti-35Nb-5Zr and Ti-35Nb-10Zr casting alloys. *J Mater Sci-Mater M* 2009, *20* (8), 1629-1636.
- 6. Niespodziana, K.; Jurczyk, K.; Jurczyk, M., The synthesis of titanium alloys for biomedical applications. *Rev Adv Mater Sci* 2008, *18* (3), 236-240.
- 7. Henriques, V. A. R.; Galvani, E. T.; Petroni, S. L. G.; Paula, M. S. M.; Lemos, T. G., Production of Ti-13Nb-13Zr alloy for surgical implants by powder metallurgy. *J Mater Sci* 2010, *45* (21), 5844-5850.
- 8. Yilmaz, E. Ç., Effect of Sliding Movement Mechanism on Contact Wear Behavior of Composite Materials in Simulation of Oral Environment. *Journal of Bio- and Tribo-Corrosion* 2019, *5* (3), 63.
- **9.** Yilmaz, E. C.; Sadeler, R., Investigation of Two- and Three-Body Wear Resistance on Flowable Bulk-Fill and Resin-Based Composites. *Mech Compos Mater* 2018, *54* (3), 395-402.
- Lawson, N. C.; Cakir, D.; Beck, P.; Litaker, M. S.; Burgess, J. O., Characterization of thirdbody media particles and their effect on in vitro composite wear. *Dental Materials* 2012, 28 (8), E118-E126.
- **11.** Yilmaz, E. C.; Sadeler, R., Investigation of three-body wear of dental materials under different chewing cycles. *Science and Engineering of Composite Materials* 2018, *25* (4), 781-787.
- **12.** Yilmaz, E. C., Effects of thermal change and third-body media particle on wear behaviour of dental restorative composite materials. *Materials Technology* 2019.
- **13.** Bhushan, B., Nanotribology, nanomechanics and nanomaterials characterization. *Philos T R Soc A* 2008, *366* (1869), 1351-1381.
- 14. Li, S. J.; Yang, R.; Li, S.; Hao, Y. L.; Cui, Y. Y.; Niinomi, M.; Guo, Z. X., Wear characteristics of Ti-Nb-Ta-Zr and Ti-6Al-4V alloys for biomedical applications. *Wear* 2004, 257 (9-10), 869-876.
- **15.** Cvijovic-Alagic, I.; Cvijovic, Z.; Mitrovic, S.; Rakin, M.; Veljovic, D.; Babic, M., Tribological Behaviour of Orthopaedic Ti-13Nb-13Zr and Ti-6Al-4V Alloys. *Tribol Lett* 2010, *40* (1), 59-70.

# EFFECT OF SLIDING CONDITION ON WEAR BEHAVIOR OF COMPOSITE MATERIALS

# Efe Çetin YILMAZ<sup>1</sup>

<sup>1</sup>Kilis 7 Aralık University, Engineering Faculty, Mechanical Engineering, Kilis, Türkiye

#### Abstract

The purpose of this study was to effect of sliding condition on wear behavior of nano-filled composite material under two-body wear test mechanism. In this study, Supreme composite material with nano filler was subjected to abrasion test procedures in vitro conditions. In this study, the test samples were divided into two groups and subjected 50 N wear force, 1.6 Hz wear frequency, 240.000 wear cycles, constantly 37 °C temperature 0.3 mm and 0.6 mm sliding conditions through wear test procedures respectively. The volume loss in the wear area of the test samples after the wear test procedures was analyzed using non-contact 3D profilometer. In addition, microstructure analysis was performed by selecting random test samples from each group using scanning electron microscopy (SEM). According to in this study obtain data with the increase in the amount of lateral movement, the volume loss in the wear area of the composite material did not show a linear relationship with the amount of lateral movement.

Keyword: Wear, Bio-Composite, Sliding Wear, Volume Loss

#### **1.Introduction**

It is important to perform mechanical and static tests of biomaterials in the laboratory because of to be able to accurately predict various mechanic behavior of biomaterial in living tissue. It is widely used in the restoration of cutting and grinding teeth due to the similarity of natural tooth structure, easy processability and improved mechanical capabilities of light-curing dental composite materials <sup>1-3</sup>. Composite materials have been steadily improving in the field of dentistry since the 1960s and have gradually increased in use as dental biomaterials <sup>4</sup>. In this area studies of the mechanical behavior and properties of various dental restorative materials are interesting research topics for dentists and researchers <sup>5</sup>. There are several difficulties with the properties of dental restorative materials, and obtaining an ideal dental restorative material is an ongoing research topic. In particular, the properties of an ideal restorative material are classified as: (a) mechanical properties, (b) application, (c) biocompatibility, and (d) aesthetics. However, the development and application of new restorative dental materials depends on a comprehensive understanding of the response of existing dental materials to static and dynamic loads <sup>5</sup>.

When the development period of composite materials is analyzed; In the early 2000s, the development of an organic matrix based on only methacrylate chemistry to date, more specifically BisGMA (bisphenol A glycidyl dimethacrylate), TEGDMA (triethylene glycol dimethacrylate), BisEMA (ethoxylated bisphenol)<sup>4</sup>. It will be seen that a composite material placed in the mouth is subjected to complex and continuous mechanical loads during the chewing movement. These mechanical loads are focused on reducing undesirable stresses in

Technology



the matrix structure of the composite material and exhibiting similar elastic behavior on the counter material (human tooth). The novel monomers obtained by these improvements in the matrix structure of the composite material could be obtained either by ring-opening polymerizable fractions (Filtek LS, which is the only commercial example based on silorane chemistry) or higher molecular weight molecules that proved to be successful in reducing the molar shrinkage coefficient <sup>4</sup>. Although a variety of composite biomaterials have been developed and used as restorative dental materials, there is little data on the dynamic behavior of the materials. Therefore, in this study, was to effect of sliding condition on wear behavior of nano-filled composite material under two-body wear test mechanism.

# 2. Materials and Methods

In this study, the mechanical and chemical properties of the tested composite material are summarized in Table 1 (information provided by material manufacturers). In this study, five test samples of 2 mm weight X 7 mm diameter were prepared for each test group of composite material. Figure 1 shows the schematics of the wear simulation test device. Half of the specimens of each test group were loaded with a sliding movement of 0.3 mm, the other half loaded with a sliding movement of 0.6 mm through wear test procedures. The test samples were divided into two groups and subjected 50 N wear force, 1.6 Hz wear frequency, 240.000 wear cycles, constantly 37 °C temperature. Composite test specimens wear volume loss was determined using three-dimensional non-contact profilometer. In addition, microstructure analysis was performed by selecting random test samples from each group using scanning electron microscopy (SEM).

<b>Table 1.</b> Mechanical and Chemical Properties of the rested Composite Materia
--

Materials /	Manufacturer	Composition				
Code	/Туре	Monomer	Filler /Filler average size	Filler Weight% / Vol. %		
Filtek Supreme XT/ FIL	3M ESPE,St. Paul, MN,USA Nanofilled	Bis-GMA, UDMA, BisEMA, TEGDMA	Nanosilica Zirconia/silica Nanoclusters (nanoparticle size) (5-20nm)	78 / 60		



# **3. Results and Discussion**

In this study, nano-filled Supreme composite material was wear volume loss of about 2.13(0.75) mm<sup>3</sup> for 0.3 mm and about 3.19(0.81) mm<sup>3</sup> for 0.6 mm sliding condition after wear test procedures.

The International Con

Figure 2 shows Supreme composite material micro-structure for 0.6 mm sliding condition after wear test procedures. Figure 3 shows example of Supreme composite material wear area volume analyses using 3d non-contact profilometer Vision 64 program (for 0.6 mm sliding wear condition). When the Figure 2 and 3 are examined, it will be seen that protective organic matris layer is formed during the wear of the composite material, thereby preventing further loss of volume of the material after wear test procedures



Figure 2. Supreme Composite Material Micro-Structure for 0.6 Mm Sliding Condition After Wear Test Procedures.

Technology



Figure 3. Example of Supreme Composite Material Wear Area Volume Analyses

In this study, the wear test simulator was programmed to perform both vertical movement of 2 mm and lateral movement of 0.3 mm and 06 mm respectively. In literature, many studies have reported that lateral wear under load ranges from 0.3 mm to 1 mm  $^{6-10}$ . In wear tests, the change in the lateral movement amount of the abrasive ball will affect the wear area occurring in the material. Therefore, in this study, the amount of lateral movement is selected from the values mentioned in the literature.

In the intraoral tribological process, the mechanism of wear of dental composite materials can occur in two stages <sup>11</sup>. First, the wear of the structure of the organic matrix causes surface roughness and loss of volume in the structures of the inorganic structures <sup>12</sup>. Then, particles that separate from the inorganic matrix structure are compressed in a lateral movement during chewing movement, thereby protecting the organic matrix structure. For this reason, it can be said that the structure of the monomer in the composite material will affect the wear behavior and surface roughness. According to in this study obtain data with the increase in the amount of lateral movement, the volume loss in the composite material increased after wear test procedures. However, the increase in volume loss in the wear area of the composite material did not show a linear relationship with the amount of lateral movement.

# **References:**

1. Rodriguez, H. A.; Kriven, W. M.; Casanova, H., Development of mechanical properties in dental resin composite: Effect of filler size and filler aggregation state. *Mat Sci Eng C-Mater* **2019**, *101*, 274-282.

The International Conference of Materials and Engineering Technology

- 2. Alsharif, S. O.; Akil, H. B.; Abd El-Aziz, N. A.; Bin Ahmad, Z. A., Effect of alumina particles loading on the mechanical properties of light-cured dental resin composites. *Mater Design* 2014, *54*, 430-435.
- **3.** Angeletaki, F.; Gkogkos, A.; Papazoglou, E.; Kloukos, D., Direct versus indirect inlay/onlay composite restorations in posterior teeth. A systematic review and meta-analysis. *J Dent* **2016**, *53*, 12-21.
- **4.** Fugolin, A. P. P.; Pfeifer, C. S., New Resins for Dental Composites. *J Dent Res* **2017**, *96* (10), 1085-1091.
- 5. Khosravani, M. R., Mechanical behavior of restorative dental composites under various loading conditions. *J Mech Behav Biomed* **2019**, *93*, 151-157.
- 6. Hahnel, S.; Schultz, S.; Trempler, C.; Ach, B.; Handel, G.; Rosentritt, M., Two-body wear of dental restorative materials. *J Mech Behav Biomed Mater* **2011**, *4* (3), 237-44.
- 7. Lazaridou, D.; Belli, R.; Petschelt, A.; Lohbauer, U., Are resin composites suitable replacements for amalgam? A study of two-body wear. *Clin Oral Investig* **2015**, *19* (6), 1485-92.
- Wimmer, T.; Huffmann, A. M. S.; Eichberger, M.; Schmidlin, P. R.; Stawarczyk, B., Twobody wear rate of PEEK, CAD/CAM resin composite and PMMA: Effect of specimen geometries, antagonist materials and test set-up configuration. *Dental Materials* 2016, *32* (6), E127-E136.
- 9. Mehl, C.; Scheibner, S.; Ludwig, K.; Kern, M., Wear of composite resin veneering materials and enamel in a chewing simulator. *Dent Mater* 2007, 23 (11), 1382-9.
- **10.** Hahnel, S.; Behr, M.; Handel, G.; Rosentritt, M., Two-body wear of artificial acrylic and composite resin teeth in relation to antagonist material. *J Prosthet Dent* **2009**, *101* (4), 269-78.
- **11.** Yilmaz, E. C.; Sadeler, R., Investigation of Two- and Three-Body Wear Resistance on Flowable Bulk-Fill and Resin-Based Composites. *Mech Compos Mater* **2018**, *54* (3), 395-402.
- 12. Jorgensen, K. D., Restorative Resins Abrasion Vs Mechanical-Properties. *Scand J Dent Res* 1980, 88 (6), 557-568.

# EFFECT OF NANO SLICA WITH EPOXY RESIN BONDED SINGLE-STRAP REPAIRS

ference of Materials and Engineering Technology

# HARUN KARAOĞLAN<sup>1</sup>, AHMET ERKLİĞ<sup>2</sup>, NURETTİN FURKAN DOĞAN<sup>3</sup>

<sup>1</sup>Gaziantep University, Faculty of Engineering, Mechanical Engineering Department, Gaziantep 27310, Turkey. <sup>2</sup>Gaziantep University, Faculty of Engineering, Mechanical Engineering Department, Gaziantep 27310, Turkey. <sup>3</sup>Gaziantep University, Faculty of Engineering, Mechanical Engineering Department, Gaziantep 27310, Turkey.

# Abstract

In this study, effects of tensile properties of epoxy adhesive with nano silica on the glass epoxy laminates adhesively bonded single strap repairs is investigated. Nano slica particles were added to the epoxy resin with an amount of 1,2,3 wt%. Single strap repairs were used as different patch ratio (D/d=2, and D/d=3). Patchs were opened by CNC machine having 10 mm diameter. Tensile tests were carried out to the specimens and their load carrying capacities were measured. Experiments show that load carry capacity increases with increasing nano slica in epoxy.

Keywords: adhesively bonded, silica, and single-strap repairs.

# **1. Introduction**

In recent years, composite materials have been thoroughly used in engineering application areas such as electronics, aerospace, aeronautics, automotive, and to traditional industries such as sports, packaging , and construction. Since the 1970s, application of composites have widely increased due to development of new fibers such as carbon, boron, and aramids, and new composite systems with matrices made of metals and ceramics. Adhesively bonded repairs are rising options to mechanical repairs in engineering applications areas and ensure many advantages over conventional mechanical connectors. These advantages are lower edge stress concentration factors, more uniform distribution of stress, lighter weight, water tightness and better fatigue properties, lower fabrication cost, etc. [1-4].

The most used methods to adhesively bond damaged structures consist of single or doublelap/strap, scarf and step configurations. (Schematic representation of single-strap, double-strap, and scarf repairs are shown in figure 1). Single and double-strap repairs put forward the advantages of easy application and low costs. However they may not be viable for high responsibility structures, because these geometries lead to moderate peel and shear stress concentrations at the bond edges, appearing from the different straining effects on the structure and patches, which obstruct a high efficiency of these repairs.[5-6]



Scarf repair

Figure 1. Schematic view of single-strap, double-strap, and scarf repairs.

Many authors have studied the mechanical properties of composite materials on the adhesivelybonded repair techniques. Moreira et.al [7] have studied high-cycle fatigue analys is of singlestrap repairs of carbon-epoxy composite laminates. It was concluded that cohesive failure inside the adhesive revealed higher fatigue life relative to inter laminar failure of the adherend and applied load is the most important parameter influencing the fatigue life. Campilho et. al [8] have studied an experimental and finite element parametric on the behaviour of single and double-strap repairs of carbon-epoxy laminates under buckling unrestrained compression. It was concluded that the finite element method can be a valuable predictive tool and an option for the reduction of costs due to experimentation, provided that suitable criteria are employed for the simulation of the different types of fracture. De Moura [9] reaserched application of cohesive zone modeling to composite bonded repairs. It was concluded that the best combination of the geometrical effects analyzed consists of a combination of patch inner chamfering and 45° straight fillet. Bulut et. al [10] studied the tensile properties of epoxy adhesive with the inclusion of micro-scale perlite and sewage sludge ash particles for glass-epoxy laminates adhesively bonded single-strap repairs. It was found that joining performance of composite laminates can be improved by the mixing of perlite or SSA filler with an adhesive epoxy at low weight percentage of both fillers contributing to resolve an important engineering problem during the service. Citil [11] investigated effects of patch material on the adhesive in the repair of damaged pipes. Pattanaik et. al [12] investigated the effect of mixing time on the mechanical properties of fly ash filled epoxy based composites, showed that the importance of suitable mixing between adhesive and filling particles. Daraker, and Chandore [13] has studied effect of surface roughness on single lap adhesive joint strength. It was found the surface roughness parameter must be considered during the design stage of adhesively bonded joints, as the bond strength varied significantly by 30-35 %, between the different surface roughness values. Cheng et. al [14] investigated analysis of an adhesively bonded single-strap joint integrated with shape memory alloy (SMA) reinforced layers. It was concluded from the numerical analyses that the SMA provides an effective way of enhancing the load carrying capacity of composite joints. Tsai et.al[15] investigatied into the stresses in doublelap adhesive joints with laminated composite adherends. Experimental and finite element show that the displacement fields are obtained from both analysis, respectively, for both joints, except for a fringe waviness, due to the three-dimensional phenomena.

There are no studies related to this literature study. The purpose of this study was to investigate the load carrying capacity of single strap joint of S-glass/epoxy composite reinforced with nano silica particles. Nano slica particles were added to the epoxy resin with an amount of 1, 2, 3 wt%. The load carrying capacity of samples was explored in terms of force- displacement relation and results were compared with each other for different patch ratios.

# 2. Materials and Procedures

#### **2.1. Production of test examples**

The composite plates used in the experiments were density of glass fiber is 200 g m<sup>-2</sup>. An epoxy resin (MOMENTIVE-MGS L285) with hardener (MOMENTIVE-MGS H285) at a stoichiometric ratio of 100:40 was used as the matrix. The main mechanical properties of both epoxy and S-glass fiber are listed in table 1 and 2, respectively.

The International Conference of Materials and Engineering Technology

The composite plates were cut to using a CNC machine. Size of the composites plates were 100 x 50 mm.Centre of the composite plates were drilled with 10 mm diameter in CNC machine, which yielded the patch repair ratio D/d=2, and D/d=3 (patch diameter(D), and hole diamater(d)). Patch diameters used for patch ratios were 20 and 30 mm, The average thickness of the samples was measured as  $2 \pm 0.2$  mm, and the adhesive thickness of the adhesive layer is measured as 0.2 mm.

Mechanical data of epoxy resin					
Density	1,18 -1,20 [g/cm <sup>3</sup> ]				
Flexural strength	110-120 [N/mm <sup>2</sup> ]				
Modulus of elasticty	3,0-3,3 [N/mm <sup>2</sup> ]				
Tensile strength	70-80 [N/mm <sup>2</sup> ]				

<b>Table 1</b> Mechanical properties of both epoxy [16]	•
---	---

E12 (GPa)	19.6	ט12	0.14	G12 (GPa)	3.8
E21 (GPa)	19.6	υ21	0.08	G21 (GPa)	3.8
E23 (GPa)	11.7	υ23	0.08	G23 (GPa)	3.8

**Table 2** Mechanical properties of S-glass fiber [17].

Sandpaper was used to smooth surface of the composite plates and patches then cleaned with acetone before the bonding. Nano silica, hardener, and epoxy resin were stirred in different ratio at 12000 rpm during 5 minutes by the mixer. Chemical compositions of the silica is shown in table 4. Plates and patches were bonded with mixture of nano slica, hardener, and epoxy resin. Mixing ratio of nano silica is given in table 3.

#### Table 3 Weight ratio of nano silica

silica (wt %)	Epoxy and hardener (wt%)	Plates and patch (D/d=2)	D/d=3
%0	%100	5	5
%1	%99	5	5
%2	%98	5	5
%3	%97	5	5



 Table 4 Chemical composition and physical properties of the silica [18]

Chemical Composition					
Content	Nano Slica				
SiO2	99.05				
A12O3	0.05				
Na2O	0.48				
LOI	0.1				
Physical Properties					
Specific Gravity	1.37				
Specific surface area(BET) m <sup>2</sup> /g	85				

# 2.2. Tensile test

Tensile tests were performed on the controlled tensile test machine Shimadzu AG-X . The machine worked with 300 kN at cross head speed of 1mm.min  $^{-1}$  as shown in figure 2. Five repeating test samples were used for each group, and their average result was taken.



Figure 2. Shimadzu AG-X Machine

# 3. Results and discussions

# 3.1 Effect of Nano Silica

Patch repairing performances of the samples were examined with two different patch repair ratios (D/d = 2 and D/d = 3). Tensile results were measured for four different mass ratios of nano silica particle by mixing adhesive epoxy. The results of the tensile tests are listed in table 5.



	The Internet
Table 5	Tensile test results

Force-displacement relations and their maximum values are shown in figure 3 and 4. As can be seen from the result, although the amount of silica in the epoxy increases the tensile strength, the tensile strength values of the samples with the patch ratio D/d = 2 were lower than those without silica.But tensile strength was higher in tests with patch ratio D/d = 3. As it is understood from the results, the effect of silica increases as the patch diameter increases and also tensile strength increased with increasing amount of silica in epoxy. Maximum tensile force was increased up to 3% wt content of silica. According to test results, the maximum single strap repair performance in tensile strength (3% wt content of silica particles) was recorded as 3.29% for D/d = 3 compared with the pure epoxy adhesive.



(a)

Technology



Figure 3 (a) Force-deformation relations and (b) maximum values of tensile test result for D/d=3





Figure 4 (a) Force-deformation relations and (b) maximum values of tensile test result for D/d=2

The particles remaining on the plates after the test are more visible when the patch diameter is large. This shows that the increase in the amount of silica added to the epoxy and the increase in patch diameter increases the tensile strength. This was also attributed to the distribution and weight percentages of particles in the epoxy, resulting in the higher strength between particle-matrix interfaces (shown in figure 5). However, opening a central hole resulted in the reduction of tensile strength by about 47.61%.



Figure 5. Fractured samples after tensile tests.

830

# 4. Conclusions

The effect of silica particles inclusions on patch repairing performance of S-glass reinforced composite laminates was investigated by mixing with different contents in the adhesive epoxy resin. The following conclusions can be written from this study:

The International Conference of Materials and Engineering Technology

- A higher amount of nano silica particles inclusion increases tensile strength of composite material.
- Silica content at 3 wt%, tensile strength reaches its maximum value due to the improved load transfer between particle-matrix interfaces.
- Patch repairing performance of the samples with D/d = 3 is greater than samples with D/d = 2.
- The maximum single strap repair performance in tensile strength (3% wt content of silica particles) was recorded as 3.29% for D/d = 3 compared with the pure epoxy adhesive.
- The particles remaining on the plates after the test are more visible when the patch diameter is large. This shows that the increase in the amount of silica added to the epoxy and the increase in patch diameter increases the tensile strength.
- As a result, repairing performance of composite laminates can be improved by the mixing of silica particles with an adhesive epoxy at high weight percentage of particles, contributing to resolve an important engineering problem during the service.

#### References

1. Adams, R.D., Wake, W.C., 1984. Structural Adhesive Joints in Engineering. Elsevier, London.

**2.** Chandraprakash Giri 1 Abhishek Jha 2 Omesh Chandrakar3 . Overlap Shear Strength Determination In Composite Materials Using Finite Element Approach For Single Strap Butt Joint (International Journal of Engineering Research & Technology (IJERT) Vol. 2 Issue 5, May - 2013 ISSN: 2278-0181)

**3.** De Moura, M. F. S. F. (2015). Application of cohesive zone modeling to composite bonded repairs. *The Journal of Adhesion*, *91*(1-2), 71-94.

4. Kaw, Autar K. Mechanics of composite materials / Autar K. Kaw.--2nd ed. [1-457].

**5.** R.D.S.G. Campilho a, M.F.S.F. de Moura a,\*, J.J.M.S. Domingues b. Using a cohesive damage model to predict the tensile behaviour of CFRP single-strap repairs.(Received 16 April 2007; received in revised form 3 September 2007 Available online 16 October 2007).

**6.** Campilho, R. D. S. G., De Moura, M. F. S. F., Ramantani, D. A., Morais, J. J. L., & Domingues, J. J. M. S. (2009). Tensile behaviour of three-dimensional carbon-epoxy adhesively bonded single-and double-strap repairs. *International Journal of Adhesion and Adhesives*, 29(6), 678-686.

**7.** R.D.F.Moreira a, M.F.S.F.deMoura b,n, M.A.V.Figueiredo b, R.L.Fernandes a, J.P.M.Gonçalves. High-cycle fatigue analys is of single-strap repairs of carbon– epoxy composite laminates was performed.(International JournalofMechanicalSciences103(2015)22–29).

**8.** R.D.S.G. Campilho a,b,\*, M.F.S.F. de Moura b, D.A. Ramantani b, J.J.L. Morais c, J.J.M.S. Domingues d. Buckling strength of adhesively-bonded single and double-strap repairs on carbon-epoxy structures.( Composites Science and Technology 70 (2010) 371–379).

**9.** De Moura, M. F. S. F. (2015). Application of cohesive zone modeling to composite bonded repairs. *The Journal of Adhesion*, *91*(1-2), 71-94.

**10.** Mehmet Bulut1, Ahmet Erkliğ2 and Nurettin Furkan Doğan2. On adhesive properties of perlite and sewage sludge ash with epoxy resin bonded single-strap repairs (Mehmet Bulut *et al* 2017 *Mater. Res. Express* **4** 085302).

11. Şerif Çitil Pamukkale Univ Muh Bilim Derg, 23(4), 422-426, 2017

**12.** Pattanaik A, Mohanty M K and Sathpathy M P 2015 Effect of mixing time on mechanical properties of epoxy-fly ash composite. (*J. Mater. Metall. Eng.* **5** 11–7)

**13.** Darekar, D. H. & Chandore, R. N. Effect of Surface Roughness on Single Lap Adhesive Joint Strength. 6th International Conference on Recent Trends in Engineering & Technology (ICRTET - 2018)

**14.** Cheng, J., Wu, X., Li, G., Pang, S. S., & Taheri, F. (2007). Analysis of an adhesively bonded single-strap joint integrated with shape memory alloy (SMA) reinforced layers. *International journal of solids and structures*, *44*(10), 3557-3574.

**15.** M.Y. Tsai  $a, \hat{n}, J$ . Morton b An investigation into the stresses in double-lap adhesive joints with laminated composite adherends.(International Journal of Solids and Structures 47 (2010) 3317–3325)

16. Hexion Specialty Chemicals www.hexionchem.com

**17.** Bulut M, Erklig A and Yeter E 2016 Experimental investigation on influence of Kevlar fiber hybridization on tensile and damping response of Kevlar/glass/epoxy resin composite laminates *J. Compos. Mater.* **50** 1875–86

**18.** https://www.researchgate.net/figure/Chemical-composition-and-physical-properties-of-cement-fly-ash-and-nano-silica-used-in\_tbl1\_319173825



# ALI YASAR<sup>1</sup>, ISMAIL SARITAS<sup>2</sup>

<sup>1</sup>Selcuk, University, Technology Faculty, Department of Mechatronics Engineering, Konya, TURKEY <sup>2</sup>Selcuk, University, Technology Faculty, *Department of Electrical and Electronics Engineering, Konya, TURKEY* 

#### Abstract

Existing methods used to provide instant solutions to classical methods in real estate appraisal are insufficient to create housing value maps. Therefore, the usability of fuzzy logic methodologies supported by computer technology in the application process was investigated. In the study, Taiwan New Taipei City Sindian Dist. 100 real estate data obtained from the real estate appraisal using fuzzy logic approach.

With the application of the methodology, housing criteria were established and the network structure was divided into three criteria: age of residence, proximity to MRT station, and number of stores accessible from residences. The application of fuzzy logic is evaluated with 3 main criteria of the structure and these main criteria are modeled. Matlab program was used for modeling with Fuzzy Logic.

Keyword: Real Estate Validation, Fuzzy Logic, House Validation, UCI Database

#### **1. Introduction**

Real estate appraisal business, which has an important place in developing economies, is one of the areas requiring specialization [1]. The values obtained through real estate appraisal are taken into consideration in various fields such as taxation, banking transactions, expropriation and trading. The methods and criteria used in the valuation area vary [2]. The valuation of immovables and their reflections on taxation constitute one of the important economic resources of developed societies. As a matter of fact, 56% of the capital resources in the world is based on immovables. In the world, real estate appraisal was initially needed for the taxation of agricultural land. It has been expanded with valuations for purchase, sale, leasing, capital market, crediting, insurance, expropriation, nationalization and privatization [3]. Supply and demand changes are also directly reflected in the values of real estates under increasing competition conditions. Especially supply and demand changes of houses have a serious impact on economic growth. It is possible to monitor this change in real estate values and determine the economic factors causing the change through objective valuation studies. However, the fact that each real estate has a different and unique characteristic makes valuation a time-consuming and costly process [4]. In regional or large scale real estate valuation processes, artificial intelligence techniques, regression analysis, Principal component analysis (PCA), statistical methods, multiple regression analysis, artificial neural networks, [5-6] and Fuzzy Logic [7] are used [8]. In our study, fuzzy logic method has been developed by considering certain criteria that can be used in housing valuation. The case study was carried out in New Taipei City Sindian Dist. According to the 100 real estate data from.

# 2. Materials and Methods

# 2.1. Fuzzy logic approach

The first serious step towards the concept of fuzzy logic was developed as a mathematical modeling in an article published by the Azerbaijani scientist Lütfü Askerzade (Zadeh) in 1965. Zadeh listed the general features of fuzzy logic with this model he developed [9].

e International Conference

aterials and Engineering Technology

 $\Box$  In fuzzy logic, information is in the form of linguistic expressions such as large, small, very few.

 $\Box$  In fuzzy logic, everything is represented by a certain degree in the range of (0.1).

- □ Fuzzy inference is done with rules defined between linguistic expressions.
- □ Fuzzy logic uses approximate thinking instead of thinking based on absolute values.
- $\Box$  Each logical system can be expressed as fuzzy.
- $\Box$  The mathematical model of fuzzy logic is very suitable for systems that are difficult to obtain.

# 2.2. Application

The data set is shown in figure1, which is taken into account in the Google map in x and y coordinates, with a total of 100 values from the New Taipei City Sindian District in Taiwan.



Figure 42. Tayvanda New Taipei City Sindian Dist.(Google Maps)

In our study, a set was formed with criteria including 100 data consisting of 3 values (input) and market value (output). General information about the 3 inputs and 1 outputs used in the data set are as follows; The age of the building, the distance to the MRT station and the number of Commercial Areas that can be reached were taken as inputs and a fuzzy system was estimated to estimate the appraisal value of this residence (Figure 2).



Figure 43. Fuzzy System Flow Chart For the Housing Valuation

# **3. Results and Discussion**

In the distribution between market values and FIS values, it is seen that FIS values are successful in estimating the market values in the system created by using uncertain logic approach in real estate valuation by criterion classification (Figure 3).



An accuracy of 74.89 percent was obtained. This shows that fuzzy logic can be used in valuation processes.

**4. Conclusions:** It is expected that the accuracy of the classification process will be increased with the better adjustment of the classification process on the data used in the study.

**Acknowledgments:** All data in the data set were studied with ANN and presented in ICAT'19 as "USING ARTIFICIAL NEURAL NETWORK APPROACH FOR VALUATION OF THE HOUSE".

#### **References:**

1. Saraç, E. (2012). Yapay sinir ağları metodu ile gayrimenkul değerleme (Doctoral dissertation, İstanbul Kültür Üniversitesi/Fen Bilimleri Enstitüsü/İnşaat Mühendisliği Anabilim Dalı).

The International Conference of Materials and Engineering Technology

- Yaşar, A., & Yalpır, Ş. To Use Fuzzy Logic Approach For Valuation Of The Parcel. In International Symposium on Intelligent System Engineering (ISISE 2013)(Abu Dhabi/UAE).
- 3. Yomralıoğlu, T., Nişancı, R., Çete, M., & Candaş, E. (2011). DÜNYA'DA VE TÜRKİYE'DE TAŞINMAZ DEĞERLEMESİ.
- 4. Hayrullahoğlu, G., Aliefendioğlu, Y., Tanrıvermiş, H., & Hayrullahoğlu, A. C. (2017, September). Konut Piyasalarında Hedonik Değerleme Modeli Tahmini: Ankara İli Çankaya İlçesi Çukurambar Bölgesi Örneği. In Proceedings of 2 nd International Conference on Scientific Cooperation for the Future in the Economics and Administrative Sciences (p. 25).
- 5. Hamzaoui, Y. and Perez, J. A. H. Centro de Investigación en Ingeniaría y Ciencias Aplicadas (CIICAp), UAEM. Av. Universidad No. 1001, Col CHAMILPA, C.P. 62209, Cuernavaca, Morelos, México. 2011 10th Mexican International Conference on Artificial Intelligence Application of artificial neural networks to predict the selling price in the real estate valuation process. 2011,
- 6. Cechin, A.L., Souto, A., González, M.A.: Real Estate Value At Porto Alegre City Using Artificial Neural Networks. SBRN 2000: 237-242. 2000,
- 7. Bagnoli. C., Smith, H. C., (1998) "The Theory of Fuzzy Logic and its Application to Real Estate Valuation" Journal Of Real Estate Research
- 8. Pagourtzi E., Assimakopoulos V., "Development of Real Estate Evaluation System with The Use of G.I.S. Technology", Kti Welcomes You To The 10th European Real Estate Society Conference in Helsinki ERES 2003, Finland. 2003,
- 9. Dinçer, H., Hacıoğlu, Ü., & Yüksel, S. (2017). Türk Bankacılık Sektöründe Dengeli Skorkart Temelli Yeni Hizmet Geliştirme Yetkinliğinin Bulanık Mantık Çerçevesinde Hibrit Çok Kriterli Karar Verme Yöntemiyle Karşılaştırmalı Analizi.

# EFFECT OF TRANSITION TEMPERATURE ON MECHANICAL PROPERTIES OF TIG WELDED AISI 304 PIPES

of Materials and Engineering Technology

# Necip Fazil YILMAZ<sup>1</sup>, Mahmut Furkan KALKAN<sup>1</sup>, Musa YILMAZ<sup>1</sup>

<sup>1</sup>Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, Turkey

#### Abstract

AISI 304 is one of the most widely used steel types in the stainless steel family used industry. AISI 304, which has very important properties in many ways, during the solidification stage temperature transition between 800°C and 500°C which is the sensitivity range during welding, chromium and carbon elements precipitates at grain boundaries. The longer the heat affect zone remains in this critical temperature range, the more precipitation will occur in this zone. As a result of this precipitation, the mechanical properties of AISI 304 change negatively due to intergranular corrosion. AISI 304 pipes are provided with the high temperature conductivity at the welding of copper cylinder with water ducts with high level of conductivity and with the help of water as a coolant, the sensitive temperature range is passed quickly. In this study, it has been shown that the mechanical properties can change in a good way by passing the sensitization temperature range quickly. Mechanical properties were investigated using hoop tensile test and hardness test.

Keywords: AISI 304, Intergranular corrosion, Weld decay, TIG Welding

#### **1.Introduction**

The most widely used material among the steel types on the industry is austenitic stainless steels, which have superior properties such as excellent high temperature and corrosion resistance. These properties of austenitic stainless steel come from the rich amount of chromium and nickel they contain. They have also relatively high tensile strength and good ductility to withstand different operating contidions and temperatures. These types of steels are the most widely used steels on the industry. AISI 304 pipes are used as when high temperature and pressure are required and a cooling system pipes in nuclear power plants [1-2].

Tungsten Inert Gas (TIG) welding method is widely used especially in welding of thin metals. TIG welding is a commonly preferred method in austenitic stainless steels compared to other joining methodsAlso according to American Welding Society (AWS) specification TIG is very popular welding type of austenitic stainless steel products. The word Tungsten stands for non-melting electrode, which provides the electric current to the arc. Because of the Inert gas is used in this method, Inert gas does not chemically active for bonding with other elements. Finally, the word gas refers to the material that covers the molten weld bath and arc, also removes the air surrounding the welding zone. As shielding gas, argon, helium or mixtures thereof are generally used [3-5].

Although AISI 304 pipes are widely used which are welded with TIG welding method, there are several problems that occur during welding operation. One of the important problem in welded AISI 304 pipes is weld decay [6]. This problem occurs at the solidification stage temperature range of 800°C and 500°C during cooling of the pipe welding. Weld decay is caused in the austenitic grain boundaries within the Heat Affected Zone (HAZ) near the welding fusion line in this solidification stage temperature range. This deterioration is reduced by the



laterials and Engineering Technology

In this study, the mechanical properties of the solidification temperature range under different conditions were investigated during the welding of AISI 304 pipes by TIG welding method. . The parameters and applied tests are explained in this article. The aim of this study is to observe the effect of this situation on mechanical values while welding deformation is eliminated by different cooling methods.

# 2. Material Method

3 mm thick commercial AISI 304 plates were tubed with 48 mm outer diameter and 100 mm length. The chemical compositions of AISI 304 samples and filler material (ER308L, 2.4 mm in diameter) given in table 1. 45 degree V-type welding mouth was prepared for all AISI 304 specimens according to the configuration shown in Fig. 1.



Figure 1. The butt single V groove joint used to manufacture the pipes. All dimensions are in mm.

Material	С%	Si%	Cr%	Ni%	Mn%	<b>S%</b>	Mo%	P%	V%	Cu%	Fe%
AISI 304	0.08	1	18-20	8-10	2	0,03	0,070	0,045	0,030	-	Bal.
ER308L	0.03	0.3-0.65	19.5-22	9-11	1-2.5	0.03	0.75	0.03	-	0.75	Bal.

Table. 1 Chemical Composition of AISI 304 and Filler Material

INVERTEC V160-TP model TIG welding device was applied with double pass-technique for manufacturing pipes at workshop of mechanical engineering department at Gaziantep University. Specifications of welding procedure were used in this study, shown in Table 2. Table. 3. lists information about the parameters of experiments.

Table 2. Welding Procedure Specifications						
Welding	Filler	Electrode	Electrode			
Process	Materials	Gas Type	Current	Size	Туре	
TIG	ER 308-L	Argon	Dc- 76A	2 mm	2% throated	

Experimental Number	Copper	Passing Fluid	Flowrate	Temperature Of Water				
1	No	No	-	-				
2	Yes	Water	45 l/min	5° C				

Table 3. Experimental Conditi
-------------------------------



Welded specimens were examined by hoop tensile test and micro hardness test to obtain effect of different cooling conditions to mechanical properties of HAZ. The hoop tensile tests, as detailed in Figure 2, were applied by 300 kN capacity Shimadzu AG-X series universal testing machine with D-blocks in the experiments to investigate. The speed of loading rate was 5 mm/min. Each sample under different conditions was subjected to tensile tested at least five times and averaged tensile test results.

Hardness values of TIG welded AISI 304 samples were measured by using LHV-1 Microvickers test device. Vickers diamond pyramid was used as a indenter and 0.5 kg load was used. Hardness test values were measured by calculating the average of 3 uniform measurements for each sample under different conditions.



Figure 2. Hoop Tensile Test of Weld Specimen and D-blocks assembly.

# **3. Result and Discussion**

# **Hoop Tensile Test**

Hoop tensile strength of the specimens were investigate by ring hoop tensile test method. Figure 3. gives the ultimate hoop tensile stress in each situation. The ultimate hoop tensile strength observed in the sample without any cooling process was observed as the lowest value of 566,52 MPa. The highest value is 659,2 MPa, which is cooled by ice water which is passed through copper inside AISI 304 pipe.



Figure 3. Hoop tensile stress, stroke graph for Sample 1

Figures 4 and 5 shows hoop tensile stress-stroke diagrams of 2 samples. The toughness of the sample with ice water passing through the copper inside the welded specimen during TIG welding operation appears to be quite high compared to the sample being welded normally.



Figure 4. Hoop tensile stress-stroke graph for Sample 1



Figure 5. Hoop tensile stress-stroke graph for Sample 2


Figure 6 shows the results of the micro-Vickers tests for samples which prepared different parameters. Hardness tests were applied on the cross-section of the TIG welded AISI 304 pipes. Both of the two different samples shows the HAZ is softest region in the welded joint. The comparison of the two microhardness samples showed that the hardness was slightly higher in the cooled sample.



Figure 6. Micro-hardness profile through the welding zone

#### 4. Conclusions

In this study, two samples produced with different parameters were examined. Hoop tensile and micro-hardness tests were applied. With the applied test, the effect of cooling method on weld decay during the manufacturing of AISI 304 pipes by welding method was examined by mechanical tests. As a result of hardness tests, it was showed that the welding zone and HAZ were softer in the sample 1 which did not apply any cooling process during welding operation. In addition, when the results of the hoop tensile test were examined, it was found to be higher in the ice-cooled sample during the welding method.

#### References

**1.** Kumar, M. V., & Balasubramanian, V. (2014). Microstructure and tensile properties of friction welded SUS 304HCu austenitic stainless steel tubes. International Journal of Pressure Vessels and Piping, 113, 25-31.

**2.** Sun, H., Wu, X., & Han, E. H. (2009). Effects of temperature on the oxide film properties of 304 stainless steel in high temperature lithium borate buffer solution. Corrosion Science, 51(12), 2840-2847.

**3.** Rossi, B. E. (1954). Welding Engineering. McGraw-Hill Interamericana.

**4.** R. Winston Revie and Herbert H. Uhlig. (2008). Corrosion and Corrosion Contrl, 4th edition. John Wiley & Sons, Inc, Canada.

**5.** E. Gözütok: The influence of argon-hydrogen gas mixture on mechanical and microstructural properties of the joints in TIG welding of stainless steels, M.SC. Thesis in Medical Engineering, University of Karabük, Türkiye (2009)

**6.** Tomoyuki Fujii, Keiichiro Togho, Yota Mori, YoshinobuShimamura, "Crystallography of intergranular corrosion in sensitized austenitic stainless steel" Materials Characterization, 144 (2018), pp. 219-226

**7.** Welding Handbook (1972). Metals and Their Weldability. 6th Edition. The American Welding Society.

**8.** S. Saha, M. Mukherjee, T.K Pal "Microstructure, Texture, and Mechanical Property Analysis of Gas Metal Arc Welded AISI 304 Austenitic Stainless Steel" Journal of Materials Engineering and Performance, vol 24(3), pp. 1125-1139, March 2015

841

# A STUDY ON PROCESS PARAMETER IN ADDITIVE MANUFACTURING

The International Conference of Materials and Engineering Technology

# Musa YILMAZ<sup>1\*</sup>, Necip Fazil YILMAZ<sup>2</sup>, Omer EYERCIOGLU<sup>2</sup>

<sup>1\*</sup>Gaziantep University, Naci Topçuoğlu Vocational School, Department of Machinery and Metal Technologies, Turkey <u>\*msyilmaz@gantep.edu.tr</u>
<sup>2</sup>Gaziantep University, Faculty of Engineering, Department of Mechanical Engineering, Turkey nfyilmaz@gantep.edu.tr, eyercioglu@gantep.edu.tr

#### Abstract

Thermoplastics produced quickly and easily in 3D printers are essential for automotive, defense and aerospace applications due to its ability of producing diverse and complex geometric parts. The aim of this study is to examine mechanical behavior of polycarbonate (PC) samples fabricated by fused deposition modeling (FDM) technology. Fused deposition modelling is rapidly growing 3D-printing technology. Samples with raster angles of 30 and 60 degrees were built using PC polymer material in the 3D-printing system. The effect of process parameter of raster angle and mechanical properties such as tensile and bending results were investigated. The results of this study were compared with the results of the previous study.

Keyword: Raster Angle, PC, Additive Manufacturing

#### **1. Introduction**

Additive manufacturing technology is one of the popular methods in rapid prototyping (RP) technology to producing three dimensional (3D) components. Additive manufacturing applications are emerging in various fields such as automotive, biomedical industry, agriculture, food production and aerospace [1-5]. Fused deposition modeling (FDM) is widely used for thermoplastic printing in the modern manufacturing due to its ability of producing complex and diverse geometric parts [6-8]. 3D parts are produced layer-by-layer directly from CAD (computer aided design) data in this technology [9]. The process starts with taking the raw material in the form of a filament and heating it to the semi-molten form. The semi-molten filament is then extruded through the nozzle which moves over the build table to create a layer of a three-dimensional model onto the platform [10, 11]. The deposited material bonds to the layer beneath it, cools and hardens. When one layer is finished, the platform drops a layer thickness height and the nozzle continues filling up to the all model is filled completely, obtaining a 3D model [12].

The advantages of this technology are easy material chance, compact size, low working temperature, low maintenance costs and supervision-free operation [13-14]. However, the main disadvantage of FDM is that it has a narrow range of materials [15] and limited information about the mechanical properties of FDM products, especially strength.



The International Conference of Materials and Engineering Technology Parts manufactured by FDM technique have many promising applications even if they may be restricted due to relatively lower strength than the injection-molded parts. Nowadays, in addition to the use of FDM technique for rapid fabrication, parts manufactured in this technology can be used directly as the final product. The mechanical properties of produced part in FDM technology must be sufficient so that it can meet the functional requirements. Therefore, in order to better understand the behavior of the FDM parts, the relevant mechanical properties need to be understood in detail. The mechanical property of the FDM product can be affected by many factors. Printing parameters such as raster angle, raster width, air gap, build orientation, layer thickness, infill density and pattern, and feed rate, among others, have a significant impact on the quality and performance of FDM printed parts [16].

Polycarbonate which is an engineering thermoplastic, is widely used in aerospace, medicine, automotive and other sectors. Products fabricated with polycarbonate filaments offers accuracy, stability and durability creating strong parts that withstand functional testing. It also has excellent mechanical properties compared to Polylactic Acid (PLA), Acrylonitrile Butadiene Styrene (ABS) and other thermoplastics [17]. When combined with a 3D-printer, PC offers strong parts for production parts and functional prototyping [18].

In this study, polycarbonate samples manufactured by FDM in two different raster angle such as 30 and 60° were used and these were compared with the samples produced with 3 different raster angles (0, 45 and 90°) in the previous study. Their mechanical properties were investigated to expose the effect of raster angle manufactured by 3D printing method.

#### **2. Experimental Procedure**

Polycarbonate filaments were used as the fabrication material for the thermoplastic samples in this work. The tensile strength was 63.7 MPa and the density of the PC was 1.20 g/cm<sup>3</sup> [19]. The working properties of polycarbonate filament are showed in Table 1. Stratasys Fortus 450mc printer was used for sample production. Stratasys Fortus 450mc printer machine and schematic representation of FDM process presented Figure 1.

Table 1. Sample specification					
Tensile Strength63.7 M					
Print Temperature	365°C				
Platform Temperature	145 °C				
Layer Thickness	0,254 μm				

Samples for tensile and bending test were prepared according to ASTM D638 and ASTM D790 standard, respectively. The detail and dimensions of the samples are illustrated in Figure 2. The printing process performed considering two different parameters as variables which are the raster angles ( $30^{\circ}$  and  $60^{\circ}$ ). The other printing parameters were kept the same in all samples.



Figure 1. a) Schematic representation of FDM process [18] and b) The photograph of 3D Printer machine used in this work

A Shimadzu AGX universal testing device was used for tensile and bending testing according to ASTM D638 and ASTM D790 standard, respectively. At room temperature, the load was applied at a rate of 5 mm/min during tensile test. In order to obtain reliable tensile and bending performance results, each test was repeated for five different samples. A symmetric 3-point bending test on a span of 30 mm were performed with a speed of 1.56 mm/sec.



Figure 2. Shape and dimensions of the test specimen for

- a) Tensile test samples
- b) Bending test samples
- c) The building direction
- d) Representation raster anglee) Raster angle used in this work



### 2. Experimental Result

Tensile test results of this study (30 and 60°) and the previous study [20] (0, 45 and 90°) are presented in Table 2. Sample built at 30° raster angle has the bigger strength than sample built at 60° raster angle (see Table 2). When all the results are compared, there is a relatively small increase in the test results as it rises from a sample having a raster angle of 0° to a sample having a raster angle of 45°. However, when the raster angle was increased from 45 degrees to 90 degrees, a sudden decrease in test results was observed. Sample built at 45° raster angle had the greatest strength of 48.8 MPa.

he International

Table 2. Tensile test results of samples				
Raster Angle	Stress	Strain		
(°)	(MPa)	(%)		
0	46,1	3,8		
30	48,5	4,9		
45	48,8	5,5		
60	39,6	3,5		
90	32,5	2,8		

When we look at Table 2, it is clearly seen that strain values show the same trend as stress. The strain value showed a trend increasing between 0°-45° and decreasing between 45°-90°. While the strain value of the samples produced at 90° raster angle was 2.8, the strain value of samples produced at 45° raster angle increased by 96% and reached the maximum value of 5.5. Stress and strain curves for 3D-printed PC samples are demonstrated in Figure 3. Since the individual raster layers of samples produced at 90° raster angle are vertical to the tensile load, the fracture of the sample is observed at the bonding between adjacent raster layers [21].



Figure 3. Engineering stress-strain curves of samples

Technology



Table 3 presents the bending test results. When the bending test results are examined, it is seen that the higher the raster angle, the lower the bending load bearing capacity of the material. Sample produced at 0° degree raster angle (202.36 N) reached the maximum bending force, but the displacement of the sample produced at 0° was relatively smaller than that of the sample produced at 45° raster angle. The lowest bending force value belongs to the sample produced at 90° raster angle which is similar to the tensile test results.

Table 3. Bending test results						
<b>Raster Angle</b>	Raster Angle Force					
<b>(</b> <sup>0</sup> <b>)</b>	(N)	( <b>mm</b> )				
0	202,4	9,5				
30	172,2	7,1				
45	160,1	10,6				
60	133,9	6,3				
90	98,3	4,9				

Mean values of bending force and displacement graph are presented in Figure 4. Image of a representative sample during 3-point bending test is shown in Figure 5. Bending properties of 3D-printed PC sample are influenced due to the weak interlayer bonding [22].



Figure 4. Force-Displacement curves of samples



Figure 5. Images of sample during bending test experiment

#### 4. Conclusion

In this paper, effect of process parameter (raster angle) on the mechanical property of polycarbonate samples produced with 3D-printer has been studied. It can be concluded that the raster angle remarkably affects the mechanical properties of 3D-printed samples in terms of tensile and bending test. Mechanical properties of the FDM part is significantly affected by the selection of raster angle. It has also been found that, the main reason for the poor strength property is the weak interlayer bonding.

#### References

- 1. Hager, I., A. Golonka, Putanowicz, R. 3D Printing of Buildings and building components as the future of sustainable construction. Procedia Eng, **2016**, 151:292-299
- 2. Farina, I., Fabbrocino, F. G., Carpentieri, et al, On the reinforcement of cement mortars through 3D printed polymeric and metallic fibers. Compos Part B, **2016**, 90:76-85
- Tran, P., Ngo, T.D., Ghazlan, A., Hui, D., Bimaterial 3D printing and numerical analysis of bio-inspired composite structures under in-plane and transverse loadings. Compos Part B, 2017, 108:210-223
- 4. Chimento, J., Jason Highsmith, M., Crane, N. 3D printed tooling for thermoforming of medical devices. Rapid Prototyp J, **2011**, 17(5): 387-392
- 5. Bose, S., Bose, D., Ke, H., Sahasrabudhe, A., Bandyopadhyay. Additive manufacturing of biomaterials. Prog Mater Sci, **2018**, 93:45-111,
- 6. Sun, Q., Rizvi, G.M., Bellehumeur, C.T., Gu, P., Effect of processing conditions on the bonding quality of FDM polymer filaments. Rapid Prototyp J, **2008**, 14(2):72-80
- 7. Kalsoom, U., Nesterenko, P.N., Paull, B., Recent developments in 3D printable composite materials. RSC Adv, **2016**, 6(65):60355-60371
- 8. Gurrala, P.K., Regalla, S. P., Part strength evolution with bonding between filaments in fused deposition modelling: this paper studies how coalescence of filaments contributes to the strength of final FDM part. Virtual Phys Prototyp, **2014**, 9(3):141-149
- Mohamed, O., Ahmed, S.H., Masood, Bhowmik, J. L., Optimization of fused deposition modeling process parameters for dimensional accuracy using I-optimality criterion. Measurement 2016, 81, 174-196
- 10. Andreas, G., Understanding Additive Manufacturin. Rapid Prototyping, Rapid Tooling, Rapid Manufacturing, 2011



- 12. Li, H., Wang, T., Sun, J., Yu, Z., The effect of process parameters in fused deposition modelling on bonding degree and mechanical properties. Rapid Prototyping Journal, **2018**, 24(1), 80-92
- Anitha, R., Arunachalam, S., Radhakrishnan, P., Critical parameters influencing the quality of prototypes in fused deposition modelling. Journal of Materials Processing Technology, 2001 118(1-3), 385-388
- 14. Bernard, A., Fischer, A., New trends in rapid product development. CIRP Annals, **2002**, 51(2), 635-652
- Thrimurthulu, K. P. P. M. P., Pandey, M., Reddy, N.V., Optimum part deposition orientation in fused deposition modeling. International Journal of Machine Tools and Manufacture, 2004, 44(6), 585-594
- 16. Chacón, J. M., Caminero, M. A., García-Plaza, E., Núñez, P.J., Additive manufacturing of PLA structures using fused deposition modelling: Effect of process parameters on mechanical properties and their optimal selection. Materials & Design, 2017, 124, 143-157
- 17. Cantrell, J. T., Rohde, S., Damiani, D., Gurnani, R., DiSandro, L., Anton, J., Ifju, P.G., Experimental characterization of the mechanical properties of 3D-printed ABS and polycarbonate parts. Rapid Prototyping Journal, **2017**, 23(4), 811-824
- 18. Information of https://www.stratasys.com/materials/search/pc, retrieved on 16.07.2019
- Jang, M.G., Ryu, S.C., Juhn, K.J., Kim, S.K., Kim, W.N., Effects of carbon fiber modification with multiwall CNT on the electrical conductivity and EMI shielding effectiveness of polycarbonate/carbon fiber/CNT composites. Journal of Applied Polymer Science, 2019, 136(14), 47302
- Yilmaz, N.F., Yilmaz, M., Eyercioglu, E., Effect of Raster Angle on Mechanical Properties of 3D Printed Polycarbonate Polymers. 8 th International Conference, ICAT'19 Sarajevo, Bosnia and Herzegovina, August 26-30, 2019
- 21. Zhang, X., Chen, L., Mulholland, T., Osswald, T.A., Effects of raster angle on the mechanical properties of PLA and Al/PLA composite part produced by fused deposition modeling. Polymers for Advanced Technologies, **2019**
- 22. Wu, W., Geng, P., Li, G., Zhao, D., Zhang, H., Zhao, J., Influence of layer thickness and raster angle on the mechanical properties of 3D-printed PEEK and a comparative mechanical study between PEEK and ABS. Materials, **2015**, 8(9), 5834-5846



# SOĞUKTA SERTLEŞEN KOMPÖZİT PELET ÜRETİMİNDE BAĞLAYICI OLARAK MELAS VE CMC'NİN BİRLİKTE KULLANIMININ PELET ÖZELLİKLERİNE ETKİSİ

of Materials and Engineering Technology

Elif ARANCI ÖZTÜRK<sup>1</sup>, Fırat ELİBOL<sup>1</sup>, Edip ERCEK<sup>1</sup>, Ali ABO<sup>1</sup>, Ezgi GÜLER<sup>1</sup>, Yağmur ADSIZ<sup>1</sup>, Nadir ŞENGÜL<sup>\*1</sup>, Aydın KARATEPE<sup>2</sup>

<sup>1</sup>Fırat Üniversitesi, Mühendislik Fakültesi, Metalurji ve Malzeme Mühendisliği Bölümü, 23119, ELAZIĞ <sup>2</sup>Erdemir Madencilik San. ve Tic. A.Ş., Divriği/SİVAS

## Özet

Alternatif demir çelik üretim yöntemlerinde aglomerasyon işlemleri önemli bir aşamayı oluşturmaktadır. Sünger demir ya da demir tanesi üretimi gibi nispeten yeni teknolojilerde, üretimden önce gerçekleştirilen aglomerasyon işlemlerinde genellikle empürite içermeyen organik bağlayıcılar tercih edilmektedir.

Bu çalışmada, daha önce melas ve CMC'nin ayrı ayrı kullanıldığı çalışmalar temel alınarak, bu iki bağlayıcı kombinasyonunun pelet mukavemetine etkisinin incelenmesi amaçlanmıştır. Deneysel çalışmalarda, Divriği A kafa manyetit cevheri konsantresi (pelet keki), redükleyici olarak kok ve bağlayıcı olarak melas - CMC karışımı kullanılarak soğukta sertleşen kompozit pelet üretimi gerçekleştirilmiştir. %0,5, 1, 1,5, 2, 3 ve 4 oranlarında CMC katı olarak pelet kekinin içine ilave edilmiş, %30'luk melas çözeltisinden, %12 kadar alınıp pelet yapım aşamasında püskürtülerek peletler üretilmiştir. Daha sonra, üretilen peletler farklı sıcaklık ve sürelerde kurutulmuştur. Mukavemet bakımından optimal sonuçlar, %2 CMC kullanılarak üretilen peletlerin 150 °C sıcaklıkta 60 dakika kurutulması sonucunda 286,7 N/Pelet'lik mukavemet değeri olarak elde edilmiştir. Elde edilen mukavemet sonuçlarının soğukta sertleşen pelet standartlarına uygun olduğu belirlenmiştir.

Anahtar Kelimeler: Peletleme, Soğukta sertleşebilen kompozit pelet, Bağlayıcı, Melas, CMC.

# 1. GİRİŞ

Geleneksel demir çelik üretimi yüksek fırın olarak adlandırılan fırınlarda gerçekleştirilir ve üretilen ürün pik demir veya sıcak metal olarak adlandırılır. Yüksek fırınlar, kok fabrikası, sinter fabrikası ve hava ısıtma sobaları gibi yan tesislere ihtiyaç duyduğundan yüksek yatırım ve işletme maliyeti gerektirmektedir. Elde edilen pik demir ya çelik üretimi için yüksek fırınlarla entegre olarak çalışan bazik oksijen fırınlarına beslenir ya da döküm sektörüne pik olarak satılır.

Çelik üretiminde dünyada kullanılan en yaygın yöntemlerden biri de elektrik ark ocaklı tesislerde yapılan üretimdir. Elektrik ark ocaklı tesislerde ana hammadde hurdadır. Ancak hurdanın yanı sıra sünger demir ve demir tanesi gibi ürünlerin kullanılması üretilecek çeliğin kalitesini arttırmaktadır. Sünger demir ve demir tanesi üretimi gibi nispeten yeni teknolojilerde yapılan üretimde kullanılan cevherler, üretim yöntemine göre toz, parça ya da pelet halinde kullanılabilmektedir. Burada üretilen peletler, gerek kullanılan bağlayıcı türü, gerekse pişirme sıcaklıkları bakımından yüksek fırın teknolojisinde kullanılan peletlerden çok farklıdır. Kullanılan bağlayıcının bentonit gibi kirlilik arz etmeyen bağlayıcı olması ve daha düşük sıcaklıklarda sertleştirilmesi, bu peletlerin karakteristik özelliklerindendir.



Kompozit peletlerin kullanımı, oksit ve karbonun iyi karışmasına bağlı olarak yüksek reaksiyon hızına sahip olması, koklaşmamış kömür ve odun kömürü gibi tozların kullanılabilmesi gibi avantajları vardır. Bu şarj maddeleri yüksek fırın dışında alternatif demir üretim teknolojileri sunmaktadır [2]. Reaksiyon hızlarından dolayı kompozit peletlerin kullanılabilmesi için iki temel özelliğe dikkat etmek gerekmektedir. Bunlardan ilki, pelete olan ısı transferine bağlıdır. İkincisi de karbonlu peletlerin pişirilememesinden dolayı soğuk bağlı olarak kullanılması ve redüksiyon prosesi esnasında peletlerin üzerine hiçbir yükün binmemesinin sağlanmasıdır [2].

Döner hazneli fırınlarda düşük yükseklikteki pelet katmanları kullanıldığı için peletin üzerine minimum yük binmektedir. Bu yüzden peletlerin yüksek dayanımda olmalarına gerek yoktur. Ancak ticari boyuttaki bir üretim tesisinde taşıma gibi işlemlerde nispeten düşük bir basma mukavemeti gerektirmektedir. İstenen bu mukavemetin de yaklaşık olarak 250-300 N/Pelet olduğu belirlenmiştir [2, 3]. Şekil 1'de kompozit pelet üretim akım şeması görülmektedir.



Şekil 1. Kompozit pelet üretimi akım şeması [3].

Bentonit, demir cevheri peletlenmesinde çok yaygın olarak kullanılan bir bağlayıcıdır. Ancak bentonitin kullanılmasında bazı sakıncalar vardır. Ürünü gang mineralleri ile kirletir ve ürünün demir tenörünü düşürür. Eğer bentonit direk redüksiyon için peletlerin hazırlanmasında kullanılırsa ham peletler direk redüksiyon dayanımının gereklerinin yerine getirmek için oksidasyon kavurması veya ön ısıtmaya tabi tutulmak zorundadırlar [4].

Buna karşılık bentonite göre pişme süresi ihmal edilebilecek organik bağlayıcıların kendilerine has avantajları mevcuttur. Organik bağlayıcılar ürünü kirletmezler. Bu belirgin özellik organik bağlayıcıların geliştirilmesini teşvik etmektedir [4].

851



Selülozik yapıya sahip olan CMC, krem renginde tozdur. Suda kolayca çözünür, organik sıvılarda çözünmez. Ağır metal tuzları ile reaksiyona girdiğinde suda çözünmeyen, şeffaf, dayanıklı ve organik maddelerden etkilenmeyen bir film oluşturur. Yarı sentetik, suda çözünebilir bir polimerdir [5].

Literatürde organik bağlayıcılardan bazıları üzerinde daha önce yapılmış çalışmalarda, bağlayıcı olarak melas ve CMC ayrı ayrı kullanılarak olumlu sonuçların elde edildiği belirtilmektedir [6, 7]. Sodyum karboksil metil selülozün (CMC) bağlayıcı olarak kullanıldığı deneylerde, %2, 4, 8, 12, 16 ve 20 oranlarında CMC ve %30 oranında kok ilavesi ile kompozit pelet üretilmiş, bu peletler daha sonra oda sıcaklığından başlamak üzere, farklı sıcaklık ve sürelerde kurutma işlemine tabi tutulmuşlar, oda sıcaklığında 2 gün bekletilen numunelerin, 2. gün sonunda suyunu tamamen kaybettiği, yeniden kristalleşme ile yüksek dayanımların elde edildiği vurgulanmıştır [6].

Melas, şeker üretiminde, teknik ve ekonomik şartlar altında şuruplardan maksimum kristal şeker alındıktan sonra geriye kalan ana şuruptur. Şeker fabrikalarında şeker pancarından, kristal şeker elde edilirken, hasat şekli, depolama şartları ve şeker elde etme yöntemine bağlı olarak kristallendirilemeyen en son surup seklinde yaklasık %4 oranında melas kalır [8].

Daha önce melas ile yapılmış bir çalışmada ise %30, 40, 50 ve 60'lık oranlarda melas su içerisinde çözündürülerek bu çözeltilerin %10'u peletleme esnasında pelet keki karışımının üzerine sıvı halde püskürtülerek beslenmiştir. Elde edilen küresel peletlerin yaş düşme ve basma mukavemeti testleri yapılarak bir kurutma fırınında 100, 150, 200 ve 250 °C sıcaklıklarda 1, 2, 3 ve 4 saat bekletilerek kurutulmuş, kurutma süresinin pelet dayanımı ile ilişkisi incelenmiştir. Aynı çalışmada, 150 °C'de 3 saat kurutulan numunelerde pelet mukavemetinin arttığı 3 saatten sonra düştüğü, 200 ve 250 °C'de yapılmış deneylerde ise 1 saat kurutma süresinden sonra pelet dayanımının düştüğü belirtilmiştir [7].

Bu çalışmada, literatürde melas ve CMC' nin ayrı ayrı kullanılarak elde edilen mukavemet değerlerinden yola çıkılarak, bu iki bağlayıcının bir arada ve farklı oranlarda kullanılmasının pelet mukavemeti üzerine yapacağı etkinin araştırılması planlanmıştır.

# **2. MATERYAL METOD**

#### 2.1.Kullanılan Hammaddeler

Deneylerde kullanılan Divriği A Kafa manyetit konsantresi Erdemir Maden Ticaret A.Ş.'den temin edilmiştir. Konsantrenin kimyasal bileşimi Tablo 1'de verilmiştir.

Bileşen	%	Bileşen	%
Fe	68,03	K <sub>2</sub> O	0,084
Fe <sub>3</sub> O <sub>4</sub>	90	Na <sub>2</sub> O	0,046
SiO <sub>2</sub>	1,66	Mn	0,06
CaO	0,55	Р	0,022
$Al_2O_3$	0,41	TiO <sub>2</sub>	0,059
MgO	0,03		

Tablo 1. Erdemir Maden Ticaret A.Ş. Divriği tesislerinden temin edilen pelet kekinin kimyasal bileşimi



	Sabit Karbon	Uçucu Madde	Kükürt	Brüt Kal Değer (kcal	lorifik Kül /kg)
%Ağırlık	88,83	0,43	0,75	7077	10,74

Tablo 2. Deneylerde kullanılan metalürjik kok kömürünün içeriği

Deneylerde bağlayıcı olarak kullanılan melas, Elazığ Şeker Fabrikası'ndan, CMC ise piyasadan satın alma yoluyla temin edilmiştir. CMC'nin elementel analizinde %26,1 C içerdiği ısıl değerinin ise 1917,4 cal/g olduğu belirlenmiştir. %33,12 C içeren melasın ısıl değerinin ise 2428,3 cal/g olduğu bulunmuştur.

#### 2.2. Yöntem

Deneylerde 75 mikronluk elekten geçirilen pelet keki, önce bağlayıcı ilave edilmeksizin, bir karıştırıcı içine konularak yaklaşık 5 dakika kadar karıştırılıp, topaklaşmaların oluşması önlenmiş; ardından, ilave edilen çeşitli miktarlardaki CMC, kok ve CaCO<sub>3</sub> ile birlikte 10 dakika karıştırılmıştır. Karıştırılan malzeme, laboratuvar ölçekli yatayla 45°'lik açı yapan kendi ekseni etrafında 30 devir/dakika hızla dönen küresel pelet tamburuna beslenmiş, üzerine karışımın %12'si olacak şekilde %30'luk melas çözeltisinden püskürtülerek öncelikle pelet çekirdekleri oluşturulmuştur. Kartopunun büyüme mekanizmasına benzer şekilde büyüyen peletler 12-14 mm boyutuna ulaşıncaya kadar malzeme ilavesine devam edilmiştir. Deneylerde kullanılan küresel peletleme cihazı Şekil 2'de gösterilmektedir.



Şekil 2. Küresel Peletleme Cihazı

Pelet üretimi aşamasında ilave edilen melas çözeltisinin pelet içindeki taneciklerin bağlanma mekanizmalarına etkisi büyüktür. Pelet özellikleri, peletlenen partiküllerin fiziksel karakteristiklerine, sıvı fazın yüzey gerilimine, viskozitesine ve bağlayıcının bağ kuvvetine bağlıdır. Aşırı miktarda sıvı her bir peletin birbirine yapışmasına sebep olacak ve pelet makinesinden uzaklaştırılması zorlaşacaktır. Elde edilen peletlerin çekirdek hali, çekirdeklerin büyümesi ve ürün peletlerin görünümü ve boyutları Şekil 3'te gösterilmektedir.



Çekirdek oluşumu

Çekirdek peletlerin büyümesi

Oluşan peletler

of Materials and Engineering Technology

Şekil 3. Pelet oluşum aşamaları ve elde edilen peletlerin görünümü

Soğuk bağlı peletlerin hazırlanmasında organik bağlayıcı içeren yaş peletler kurutulmaları esnasında sertleştirilerek, kurutma ve pişirme safhaları bir arada yapılmaktadır. Yapılan bu çalışmada peletlerin kurutma işlemleri 250 °C'ye kadar çıkabilen bir etüvde yapılmıştır. Peletler üzerinde yaş pelet düşme ve kuru mukavemet testleri standartlara uygun bir şekilde yapılmış olup, tüm testler en az 10 pelet üzerinde tekrarlanmış ve ortalama değerler alınmıştır. Şekillendirilen peletlerin, yaş pelet düşme sayıları, 45 cm'den çelik bir plaka üzerine düşürülerek kırıldığı ana kadar tekrarlanmış, düşme sayısının bağlayıcı miktarıyla orantılı bir şekilde 9'dan 25'e kadar arttığı gözlenmiştir. Yaş pelet mukavemet değerleri ise 8-10 N/Pelet arasında değişmektedir.

Farklı sıcaklıklarda kurutulan peletlerin kuru basma dayanımları Şekil 4'te gösterilen mukavemet ölçüm cihazında, iki çelik plaka arasına konulan peletin kırılıncaya kadar yük uygulanması şeklinde yapılmıştır.



Şekil 4. Pelet mukavemeti ölçüm cihazı

Basma dayanımlarının tespitinde her bir grup pelet türünden yaklaşık 12-13 mm çapa sahip olan rastgele 10 numune seçilmiş ve bunların basma dayanımları ölçülerek ortalamaları alınmıştır.



Soğukta sertleşen kompozit peletler için literatürde tavsiye edilen basma dayanımı değeri 250-300 N/Pelet'dir. Deneysel çalışmalarda kullanılan bağlayıcı oranları bu standartlara ulaşabilecek şekilde ilave edilmiştir. Deneylerde bağlayıcı olarak önce vüksek konsantrasyonlardaki melas çözeltisi tek başına kullanılmış ancak, yüksek viskozitesinden dolayı, peletleme esnasında püskürtme isleminde güçlükler yaşanmıştır. Literatürde yapılan bazı çalışmalarda farklı dozajlardaki melas çözeltisi ilaveli peletler için değişik sıcaklıklarda kurutma süresindeki artışın pelet dayanımında azalmaya sebep olduğu belirtilmiştir [7]. Aynı çalışmada %30'luk melas çözeltisi püskürtülerek hazırlanan peletlerin soğukta sertleşen pelet standartlarına yakın mukavemet değerine sahip olabilmeleri için 150 °C'de 3 saate kadar kurutulmaları gerektiği vurgulanmıştır. Önceki çalışmalar da dikkate alınarak, bu çalışmada %30'luk melas çözeltisinin püskürtülmesinde bir problem yaşanmadığı için bu orandaki melas çözeltisinden peletlenecek malzemenin %6, 8, 10 ve 12'si kadar püskürtülerek hazırlanan peletlerin farklı sıcaklıklarda kurutulmaları sonucu %12'lik oranda standartlara yakın mukavemet değerlerinin elde edildiği görülmüş ancak standartlar arasında bir değer elde edilmemiştir. Soğukta sertleşen pelet mukavemeti standartlarına ulaşmak için, melas oranı sabit tutulup, peletlenecek toz malzemeye (flaks + kok + konsantre) %0,5, 1, 1,5, 2, 3 ve 4 oranında CMC ilave edilerek karıştırılmış ve pelet yapım aşamasında %30'luk melas çözeltisinden toplam karışımın %12'si kadar püskürtülerek üretilen peletler 150, 200 ve 250 °C sıcaklıklarda kurutulmuştur.

#### **3. BULGULAR VE TARTIŞMA**

Yapılan deneylerde peletlerin 150, 200 ve 250 °C'deki kurutma işlemine tabi tutulmaları sonucu, ilk 60 dakikada kurutma süresinin artması, pelet mukavemetini düşürmüştür.

İlave edilen melas oranı sabit tutulup, farklı oranlarda ilave edilen CMC ile hazırlanan peletlerin farklı sıcaklıklarda kurutulmaları sonucu, %3 CMC ilaveli peletler hariç, tüm peletlerde mukavemet değerleri 150 °C'lik kurutma işleminin özellikle ilk 60 dakikalık kısmında mukavemet değerlerinde belirgin bir artışa neden olduğu görüldü (Şekil 5). Ancak daha uzun kurutma sürelerinde mukavemet değerlerinde azalma olduğu görülmüştür.



Şekil 5. Değişen CMC miktarının pelet basma dayanımına etkisi

düşük yüksek CMC molekülleri konsantrasyonlarda, düz bir yapı sergilerken, konsantrasyonlarda moleküller üst üste binerek sarmal bir yapıya dönüşür ve sıcaklığa bağlı olarak geri dönüsümlü jel haline gelir. Selüloz ergimez ancak 180 °C sıcaklıkta bozunur. 200 °C'nin üzerindeki sıcaklıklara ise, Na-CMC'nin bozunma tehlikesi nedeniyle çıkılmaması gerektiği literatürde belirtilmektedir [9,10]. Şekil 5'te verilen grafiklere bakıldığında, 200 °C ve üzerindeki sıcaklıklarda hemen hemen tüm CMC oranlarında mukavemet değerleri bakımından bir düşüşün olduğu görülmektedir. %2 CMC ilaveli peletlerin mukavemet değerlerinde 288 N/Pelet'lik değerler elde edildiği görülmektedir. Bu peletlerin %20-24 arasında poroziteye sahip olduğu görülmüştür. Bu oranın altında ve bu oranın üzerindeki oranlarda CMC ilavesi mukavemet değerlerinde bir artışa neden olmadığı gibi, standartlar altında değerler elde edilmiştir. Bu durum, sıcaklık artışının hem melas hem de CMC'nin bozunmasına dayandırılabilir. Hem CMC hem de melasın sıcaklık artışıyla göstereceği değişimi görebilmek için termogravimetrik analizine bakılmış ve Şekil 6 ve 7'de bu analiz sonuçları verilmiştir.



CMC belirli bir sıcaklığın üzerinde jelleşmektedir. Bütün organik bağlayıcıların 200 °C sıcaklıklar üzerinde bozunmaya uğrayıp, bağlayıcılık vasfını yitirdikleri, daha önce yapılan çalışmalarda belirtilmektedir. Literatürde CMC'nin alevlenme noktasının 290 °C'nin üzerinde olduğu belirtilmektedir [10]. Bu çalışmada CMC'nin termogravimetrik analizine bakıldığında 293 °C' de ani bir kütle kaybının olduğu gözlenmiştir (Şekil 6).



Şekil 6. CMC' nin termogravimetrik analizi

%33,12 C içeren melasın termogravimetrik analizinde 216 °C sıcaklığa kadar %3'lük bir kütle kaybı gözlenmiş, ancak melasın kabarıp sişmesi ve krozenin dışına taşması nedeniyle 216 °C'den daha yüksek sıcaklıklar için termogravimetrik analizi yapılamamıştır (Şekil 7).



Şekil 7. Melasın termogravimetrik analizi



# 4. SONUÇ

Erdemir Maden Ticaret A.Ş. Divriği tesislerinden temin edilen Divriği A kafa manyetit cevheri konsantresi (pelet keki) ve kok ile bağlayıcı olarak melas ve CMC karışımı kullanılarak soğukta sertleşen kompozit pelet üretilmesine çalışılmıştır.

Soğuk bağlı kompozit peletlerin kuru basma dayanımlarının literatürde 250-300 N/Pelet arasında olması gerektiği belirtilmiştir. Daha önce yapılan çalışmalarda %2 oranında CMC tek başına kullanıldığında basma dayanımının yaklaşık 353 N/Pelet olarak elde edildiği bildirilmiştir. Burada yapılan çalışmada melas-CMC birlikte kullanılmış, ancak literatürde bahsedilen değerlere ulaşılamadığı görülmüştür. Melasın CMC ile birlikte bağlayıcı olarak ilave edilmesinin farklı organik fazların oluşmasına neden olduğu ve bunun sonucunda da daha düşük sıcaklıklarda bozunmaya uğradıkları anlaşılmaktadır. Yapılan TG analizlerden de anlaşılacağı üzere melasın CMC'ye nazaran daha düşük sıcaklıklarda bozunduğu, bozunma sıcaklığı nispeten daha yüksek olan CMC'nin de daha düşük sıcaklıklarda bozunmasına neden olduğu düşünülmektedir. Bu durum göz önünde bulundurularak, peletlerin oda sıcaklıklarında kurutulması düşünülmüş, ancak oda sıcaklığında yapılan kurutma işlemlerinde yeterli mukavemet değerlerine ulaşılamamıştır.

Soğukta sertleşen küresel pelet üretimi için CMC-melas kombinasyonu üzerine yapılan bu çalışmada, %30 kok ve %1,13 CaCO<sub>3</sub> ilavesinin yanı sıra, bağlayıcı olarak %2 CMC'nin katı olarak harmanın içine ilave edilmesi, pelet yapımı sırasında su yerine %30'luk melas çözeltisinden karışımın %12'si kadar püskürtülmesi, üretilen peletlerin 150 °C sıcaklıkta 60 dakika bekletilmesi sonucu 288 N/Pelet'lik mukavemet değerinin elde edildiği görülmüştür.

#### KAYNAKLAR

[1] Ghosh, A., Mungolge, M., Gupta, N., ve Tiwari, S., 1999, "A Preliminary Study of Influence of Atmosphere on Reduction Behavior of Iron Ore-Coal Composite Pellets", ISIJ International, Vol. 39. No. 8, pp, 829-831

[2] Mourao, M., ve Takano, C., 2003, "Self-Reducing Pellets for Ironmaking Mechanical Behavior", Mineral Processing & Extractive Metall. Rev., 24: 233-252.

[3] Agrawal, B., B., Prasad, K., K., Sarkar, S., B., ve Ray, H.S., 2000, "Cold Bonded Ore-Coal Composite Pellets For Sponge Ironmaking Part 1 Laboratory Scale Development" Ironmaking and Steelmaking, Volume 27, Number 6, December 2000, pp. 421-425 (5).

[4] Qiu, G., Jiang, T., Huang, Z., Zhu, D., ve Fan, X., 2003, "Characterization of Preparing Cold Bonded Pellets for Direct Reduction Using an Organic Binder", ISIJ International, Vol. 43 No. 1, pp. 20–25

[5] http://www.prosisgida.com.tr/Kivam-Vericiler\_Karboksimetil-seluloz---E-466\_11.html

[6] Birol B., Benkli Y. E., Boyrazlı M., Sarıdede M. N., 2009, "Soğukta Sertleşen Kompozit Peletlerin Mukavemetine Bağlayıcı Türünün Etkisinin İncelenmesi", 5. Uluslararası İleri Teknolojiler Sempozyumu (IATS'09), 13-15 Mayıs 2009, Karabük, Türkiye

Technology

The International Conference of Materials and Engineering



[7] Benkli Y.E, Boyrazlı M., Artır R., Çizmecioğlu Z., 2012, "Soğuk Bağlı Kompozit Demir Peletlerde Farklı Bağlayıcıların Basma Dayanımı Üzerine Etkilerinin Araştırılması" Makine Teknolojileri Elektronik Dergisi 2012,(9)15-26

[8] Türkiye Şeker Fabrikaları A.Ş., 2015, "2014 Sektör Raporu", Ankara.

[9] http://www.biokimkimya.com/content.asp?id=14&v=c&d=p&pid=1065&l=tr

[10] [Kanematu, T., 1985. Process for Producing Sodium Carboxymethylcellulose,United States Patent, No:4,421, 594 dated 04.06.1985.

# EXPERIMENTAL, ANALYTICAL AND NUMERICAL EXAMINATION OF ACTIVE FAILURE SURFACES OCCURRING BEHIND THE INVERTED T CANTILEVER RETAINING WALL

The International Conference

aterials and Engineering Technology

# HAKAN ALPER KAMİLOĞLU<sup>1</sup>, EROL ŞADOĞLU<sup>2</sup>, FATİH YILMAZ<sup>1</sup>

<sup>1</sup> Bayburt University, Civil Engineering Department, Bayburt, TURKEY. <sup>2</sup> Karadeniz Technical University, Civil Engineering Department, Trabzon, TURKEY.

#### Abstract

Determination of lateral earth pressure plays vital role in retaining wall design. Failure surfaces are very effective in active lateral earth pressure acting on cantilever retaining walls. Calculations of lateral earth thrusts vary for two different cases, namely short heel or long heel, based on the intersection of T type cantilever wall and failure surface. In this study, effect of heel length on active failure mechanism was examined with numerical simulation based on FEM. The results of the numerical analyses were compared with the results of small-scale model tests and an analytical method. In comparison, inclination angle of active failure surfaces were taken into account. An earth thrust maximization code suggested in the literature was used to determine failure surface inclination angles analytically. In order to determine failure inclination experimentally, results of small scale tests were used. In the tests failure surfaces were determined using particle image velocimetry technique (PIV). Numerical analysis of failure surfaces were performed using commercially available finite element program Plaxis 2D. The same material properties is used in all numerical models. As a result of the study short heel-long heel cases and effective parameters on the inclination angles of the failure surfaces are explained elaborately.

**Keyword:** T Type cantilever retaining wall, Failure surface, Short heel, Long heel.

#### **1. Introduction**

Retaining walls are civil engineering structures that have been used since ancient times. The cantilever retaining wall is one of the most commonly used retaining structures in geotechnical engineering and this type of retaining walls have been used since World War II. Different techniques can be used to determine lateral earth thrusts acting on retaining structures, such as the limit equilibrium method [1]–[4] slip line method, limit analysis method [5], [6], stress characteristics method [7], zero extension line (ZEL) method [8], numerical methods [9]–[11], and graphical methods [12]. Nonetheless, Coulomb's [13] and Rankine's [14] methods are still used to calculate lateral earth pressure. While evaluating these methods, it can be seen that failure surface geometry plays an important role in magnitude of lateral earth thrusts.

One of the parameters effecting lateral earth pressure is heel length of the inverted T type cantilever retaining walls. There are a number of studies considering the heel length in lateral earth pressure calculations [15], [16]. The retaining walls can be classified as long heel or short heel based on heel length. In active state, if heel length of the wall is not long enough, active failure surface intersects with the wall. This type of wall heel is termed as 'short heel'. On the other hand, it is supposed that active failure surface reaches to the backfill surface without meeting any obstacle if

the wall heel is long enough. As a result of the studies, it can be seen that active failure surface mechanisms occurring behind the wall with long heel and short heel are totally different[15], [17]. In addition, various failure mechanisms occur based on wall geometry [2]. Despite there are many studies considering earth pressure distribution, there are limited number of studies considering effect of heel length on lateral earth pressure distribution.

The International Conference of Materials and Engineering

In this study, it is intended to examine short and long heel phenomena with experimental, analytical and numerical methods. Inverted T cantilever walls can be classified as short heel or long heel based on the intersection of cantilever wall and failure surface. Thus, failure surface inclinations should be taken into account in the analyses. Small scale test results performed by Kamiloğlu and Şadoğlu [2] is presented in the experimental part of the study. In the study, active failure surfaces were determined using PIV method. An algorithm suggested by Kamiloğlu and Şadoğlu [2] was used to evaluate active failure surfaces analytically. The algorithm was coded to determine active earth thrust coefficient and failure surface inclination angles occurring behind inverted T cantilever walls. Analytically derived active earth thrust formulas by Kamiloglu and Şadoğlu [2] was used in the algorithm. Commercially available finite element program Plaxis 2D was used to examine active failure surfaces numerically. Within scope of the analysis, inverted T cantilever retaining wall with 3m height was considered. In all analyses, effect of density, heel length and foundation thickness parameters on failure surface inclinations were examined.

#### 2. Materials and Methods

In this part of the study, examination methods of active failure surfaces are clarified elaborately. Same soil properties and wall geometries are taken into account in all methods. Within this scope, heel length (b) and foundation thickness (H<sub>3</sub>) parameters are expressed in terms of wall height (H) as follows;

$$b = \beta H \tag{1}$$

$$H_3 = aH \tag{2}$$

Same heel length coefficients ( $\beta$ ) and foundation thickness coefficients ( $\alpha$ ) are considered in experimental, analytical and numerical studies.

#### 2. 1. Experimental Examination of Active Failure Surfaces

Results of small-scale tests performed by Kamiloglu and Şadoğlu [2] are taken into account. Poorly graded sand (SP) was used as granular backfill. The failure surface geometries were observed for various heel lengths, foundation thicknesses and densities in the experiments. The model cantilever retaining walls used in the study had a height of 0.30 m and were made of hard wood. Five model walls with different heel lengths (0.03, 0.06, 0.09, 0.12, and 0.15 m) were used to investigate the effect of heel length on failure surfaces in active state. With the aim of examining the relation between failure surface and foundation thickness, model retaining walls with different foundation thickness, model retaining walls with different foundation thickness, model retaining walls with different foundation thickness, model retaining walls with different foundation thickness (0.03m, 0.06m, and 0.09m) were used for each heel length (0.03m, 0.06m, 0.09m, 0.12m, and 0.15m). Failure surface observations were performed for different backfill densities ( $\rho_1$ =1.45 Mg/m<sup>3</sup>,  $\rho_2$ =1.58 Mg/m<sup>3</sup>,  $\rho_3$ =1.65 Mg/m<sup>3</sup>) in the active state. The model retaining wall

Technology

with a heel length of 0.06m and foundation thickness of 0.03m was used to determine the effect of backfill density on failure surfaces.

he International Conference of Materials and Engineering Technology

In order to determine active failure surfaces the model wall was subjected to translation during the tests and active translation recorded with digital camera. PIV analysis was performed using captured images. The image sequences obtained from the small-scale tests were analyzed with PIVlab, and, the displacement vector fields and the simple strain-rate fields were determined. Inclination angle of the failure surfaces were determined using results of the PIV analyses. Internal friction angle of the backfill, friction angle between the model wall and the backfill, and some relevant geotechnical properties of the backfill were determined to compare experimental results with analytical and numerical results. Material properties of the experimental study are shown in Table 1.

Table 1. Some basic properties of backfill soil				
Property	Value			
D <sub>10</sub>	0.55 mm			
D <sub>30</sub>	0.75 mm			
D <sub>50</sub>	0.95 mm			
$D_{60}$	1.20 mm			
Coefficient of uniformity (Cu)	2.18			
Coefficient of gradation (Cc)	0.85			
Average specific gravity (Gs)	2.50			
Maximum density ( $\rho_{max}$ )	1.67 Mg/m <sup>3</sup>			
Minimum density ( $\rho_{min}$ )	1.41 Mg/m <sup>3</sup>			
Internal friction angle [ $\rho_1$ = 1.45 Mg/m <sup>3</sup> ]	φ=36°			
Internal friction angle $[\rho_2 = 1.58 \text{ Mg/m}^3]$	φ=38°			
Internal friction angle $[\rho_3 = 1.65 \text{ Mg/m}^3]$	φ=40°			
Modulus of elasticity $[\rho_1 = 1.45 \text{ Mg/m}^3]$	25.6 MPa			
Modulus of elasticity $[\rho_2 = 1.58 \text{ Mg/m}^3]$	36.6 MPa			
Modulus of elasticity $[\rho_3 = 1.65 \text{ Mg/m}^3]$	47.5 MPa			
Friction angle [wall backfill; $\rho_1 = 1.45 \text{ Mg/m}^3$ ]	δ=32°			
Friction angle [wall backfill; $\rho_2 = 1.58 \text{ Mg/m}^3$ ]	$\delta = 35^{\circ}$			
Friction angle [wall backfill; $\rho_3 = 1.65 \text{ Mg/m}^3$ ]	δ=36°			

#### 2.2. Analytical Examination of Active Failure Surfaces

Earth thrust determination method suggested by Kamiloğlu and Şadoğlu [2] for the T type cantilever retaining walls is taken into account in analytical part of the study. Thus, an algorithm suggested by Kamiloglu and Şadoğlu [2] (Fig.1) is used to predict inclination angles of the active failure surfaces. In order to compare analytical results with results of the other methods, same heel lenght and foundation thickness coefficient, internal friction angle, density parameters with experimental study is used.

862



Figure 1. Flowchart of thrust-maximization algorithm [2]

## 2.3. Numerical Examination of Active Failure Surfaces

Numerical analyses were performed using Plaxis 2D 8.6 commercially available finite element software. In numerical model retaining walls with 3 m height were considered. In order to create compatible wall geometries with analytical and experimental studies the heel lengths are determined as b= 0.3m, 0.6m, 0.9m, 1.2m, 1.5m and foundation thicknesses are determined as 0.3m, 0.6m and 0.9m. Beside, same backfill properties with experimental study is considered in the FE analyses.

In order to prevent intersection between the active failure surface and the vertical boundaries, horizontal and vertical dimensions of the model were chosen as 8m and 6m respectively. Displacements of the vertical boundaries of the model were constrained in horizontal directions and the bottom boundary of the model was constrained horizontally and vertically. The retaining wall was translated horizontally away from the backfill (0.1% of the wall height) to create active state

Mohr-Coulomb material model and fifteen noded triangular element with three Gauss point were used in numerical analyses. In contact surfaces (wall-backfill), interface elements were used to simulate the contact behaviour. Backfill properties shown in Table 1 is taken into account in the FE analyses. Additionally, other parameters considered in the FE analyses are presented in Table 2. Strength reduction factors of the interface elements are determined as  $\delta/\phi$  value. By taking into consideration suggestions of Kamiloğlu et.al. [18] average element size of the model is determined as 70.16 x10<sup>-3</sup> m (Fig.2b). The mesh size of the element is refined locally around backfill and the wall, particularly near the contact surfaces between the wall and backfill. As a result of the FE analyses strain fields and failure surface inclinations are determined.

Table 2. Parameters considered in the FE analyses								
ф	Propert	Value	ø	Propert	Value	ø	Propert	Value
	У			У			У	
	E	25.6 MPa		E	36.6 MPa		Е	47.5 MPa
	υ	0.3	38°	υ	0.3	ι γu	υ	0.3
	$\gamma_{unsat}$	14.5 kN/m <sup>3</sup>		γunsat	15.8 kN/m <sup>3</sup>		γunsat	16.5kN/m <sup>3</sup>
36°	γsat	18.7 kN/m <sup>3</sup>		γsat	19.5 kN/m <sup>3</sup>	40°	γsat	19.96kN/m 3
	δ	32°		δ	35°		δ	36°
	Rinter	0.888		Rinter	0.92		Rinter	0.9
	с	0.1 kN/m <sup>2</sup>		с	0.1 kN/m <sup>2</sup>		с	0.1 kN/m <sup>2</sup>

From the FE analyses the failure surfaces occurring behind the cantilever walls with 0.3 m of foundation thickness and different heel lengths (0.3m, 0.6m, 0.9m, 1.2m, and 1.5m) are shown in Fig.2.

Technology

e International Conference of Materials and Engineering



Figure 2. Failure surfaces obtained with FE analyses (  $\alpha = 0.3 \text{ m}$ ,  $\gamma = 1.45 \text{ Mg/m}^3$ ,  $\phi = 36^\circ$ ): (a)  $\beta = 0.3 \text{m}$ ; (b)  $\beta = 0.6 \text{m}$ ; (c)  $\beta = 0.9 \text{m}$ ; (d)  $\beta = 1.2 \text{m}$ ; (e)  $\beta = 1.5 \text{m}$ .

The FE results for the wall with 0,6 m foundation thickness and various heel lengths (0.3m, 0.6m, 0.9m, 1.2m, and 1.5m) are present in Fig.3



(a)  $\beta$ =0.3m; (b)  $\beta$ =0.6m; (c)  $\beta$ =0.9m; (d)  $\beta$ =1.2m; (e)  $\beta$ =1.5m.

¢.



In Figure 4, results of the FE analyses for the wall with 0.9m foundation thickness and different heel lengths (0.3m, 0.6m, 0.9m, 1.2m, and 1.5m) are shown.



Figure 4. Failure surfaces obtained with FE analyses (  $\alpha = 0.9 \text{ m}$ ,  $\gamma = 1.45 \text{ Mg/m}^3$ ,  $\phi = 36^\circ$ ): (a)  $\beta = 0.3 \text{m}$ ; (b)  $\beta = 0.6 \text{m}$ ; (c)  $\beta = 0.9 \text{m}$ ; (d)  $\beta = 1.2 \text{m}$ ; (e)  $\beta = 1.5 \text{m}$ .

The internal friction angles of the backfill used in the analyses are  $\phi = 36^{\circ}$ ,  $\phi = 38^{\circ}$ , and  $\phi = 40^{\circ}$  from the experimental  $\rho - \phi$  curve of the backfill, corresponding to densities of 1.45, 1.53, and 1.65 Mg/m<sup>3</sup>, respectively. The cantilever retaining wall with 0.06 m of heel length and 0.03 m of foundation thickness are used in the analyses. The effect of backfill density on active failure surface geometry is presented in Fig.5.





#### **3. Results and Discussion**

Results of the analytical, numerical and experimental studies are shown in Table 3, Table 4 and Table 5. After horizontal translation of the wall, two active failure surfaces occur behind the wall. Inclination angle of the failure surface occurring close to the wall is termed as angle  $\psi$  and inclination angle of the failure surface occurring at the far point of the wall is termed as angle  $\theta$ . Effect of heel length coefficient and foundation thickness coefficient on angle  $\psi$  and angle  $\theta$  is shown in Table 3 and Table 4 respectively. In addition, results of the FE analyses are compared with results of the analytical [2] and experimental [2] studies and Rankine's method [14].

The International Conference of Materials and Engineering Technology

From the Table 3 it is seen that results of the numerical study are compatible with the results of experimental and analytical studies. Angle  $\psi$  obtained with experimental, analytical and numerical methods are effected from the heel length and foundation thickness coefficients. On the other hand, inclination angle of the failure surface found by using Rankine's method is not effected from the heel length or foundation thickness parameters.

Table 3.	$\psi$ angles	determined	with exp	perimental,	analytical,	numerical	and Rankin	e's method

		Heel Length Coefficient					
Foundation thickness coefficient	Method	β=0.1	β=0.2	β=0.3	β=0.4	β=0.5	
	Experimental Study [2]	72°	66°	64°	61°	DFS	
α=0.1	Analytical Study [2]	74°	70°	67°	64°	63°(LH )	
	Numerical Study	70°	63°	64°	61°	57°	
	Rankine $(45+\phi/2)$	63°	63°	63°	63°	63°	
	Experimental Study [2]	73°	$70^{\circ}$	59°	DFS	DFS	
α=0.2	Analytical Study [2]	73°	69°	66°	63°(LH )	63°(LH )	
	Numerical Study	70°	64°	64°	59°	57°	
	Rankine $(45+\phi/2)$	63°	63°	63°	63°	63°	
	Experimental Study [2]	73°	67°	DFS	DFS	DFS	
α=0.3	Analytical Study [2]	73°	68°	65°	63°(LH )	63°(LH )	
	Numerical Study	71°	$66^{\circ}$	64°	61°	57°	
	Rankine $(45+\phi/2)$	63°	63°	63°	63°	63°	

#### DFS=dispersed failure surface, LH=longheel.

The angle  $\theta$  determined with various methods are presented in Table 4. It is seen from the table that wall dimensions have negligible effects on the angle  $\theta$ . In addition, long heel or short heel cases can be defined with analytical and experimental studies. However, this cases cannot be defined using numerical methods and Rankine's methods.



**Table 4.**  $\theta$  angles determined with experimental, analytical, numerical and Rankine's method

the International Conference

of Materials and Engineering Technology

#### DFS=dispersed failure surface, LH=longheel.

Inclination angles of the active failure surfaces (angles  $\psi$  and  $\theta$ ) determined using different methods are seen in Table 5. The failure surface inclination angles are examined for various backfill densities. As it is seen from the table, backfill density (or internal friction angle) has considerable effects on failure surface inclination angles (angle  $\psi$  and  $\theta$ ).

Table 5. Life	<b>Table 5.</b> Effect of density on angle $\psi$ and angle $\phi$						
Density (Mg/m <sup>3</sup> )	ρ= 1.45		ρ=1	.58	ρ=1	ρ=1.65	
Method	Ψ	θ	ψ	θ	ψ	θ	
Experimental Study [2]	66°	59°	66°	71°	67°	77°	
Analytical Study [2]	$70^{\circ}$	61°	$70^{\circ}$	62°	71°	63°	
Numerical Study	64°	55°	$66^{\circ}$	57°	68°	58°	
Rankine $(45+\phi/2)$	63°	63°	64°	64°	65°	$65^{\circ}$	

**Table 5.** Effect of density on angle  $\psi$  and angle  $\theta$ 

#### 4. Conclusions:

In this study, active failure surfaces ocurring behind inverted T cantilever retaining walls are examined considering various methods. In examination of the failure surfaces, inclination angles are taken into account. The FE analyses were performed for the wall with different heel lengths, foundation thicknesses and backfill densities. The numerical results are compared with results of the small-scale tests [2], an algorithm coded to determine active failure surface inclination angles [2] and Rankine's method [14]. From the study the following conclusions may be drawn:

• As a result of the numerical study it is seen that heel length and foundation thickness have considerable effects on the inclination angle of the active failure surface occurring at the close point of the wall (angle  $\psi$ ). On the other hand the inclination angle of the active

failure surface occurring at the far point of the wall (angle  $\theta$ ) is not effected from the heel length or foundation thickness.

The International Conference of Materials and Engineering Technology

- Results of the numerical analyses are compatible with the results of analytical and experimental studies.
- Short heel or long heel cases can be examined considering results of the experimental and analytical studies. However, these cases cannot be recognized clearly using the FE analysis referred in the study.
- Effect of heel length and foundation thickness parameters on the inclination angles of the active failure surfaces cannot be determined with Rankine's method.
- Backfill density and internal friction of the backfill have vital effects on active failure surfaces.

#### **References:**

- 1. Y. Cai, Q. Chen, Y. Zhou, S. Nimbalkar, and J. Yu, Estimation of Passive Earth Pressure against Rigid Retaining Wall Considering Arching Effect in Cohesive-Frictional Backfill under Translation Mode, Int. J. Geomech., vol. 17, no. 4, p. 04016093, Apr. 2017.
- 2. H. A. Kamiloğlu and E. Şadoğlu, Experimental and Theoretical Investigation of Short and Long Heel Cases of Cantilever RetainingWalls in Active State, Int. J. Geomech., 2019.
- **3.** A. Pain, Q. Chen, S. Nimbalkar, and Y. Zhou, Evaluation of Seismic Passive Earth Pressure of Inclined Rigid Retaining Wall Considering Soil Arching Effect, Soil Dyn. Earthq. Eng., vol. 100, pp. 286–295, Sep. **2017**.
- Y. Zhou, Q. Chen, F. Chen, X. Xue, and S. Basack, Active Earth Pressure on Translating Rigid Retaining Structures Considering Soil Arching Effect, Eur. J. Environ. Civ. Eng., vol. 22, no. 8, pp. 910–926, Aug. 2018.
- **5.** A. H. Soubra and P. Regenass, Three-Dimensional Passive Earth Pressures by Kinematical Approach, J. Geotech. Geoenvironmental Eng., vol. 126, no. 11, pp. 969–978, **2000**.
- X. L. Yang, Upper Bound Limit Analysis of Active Earth Pressure with Different Fracture Surface and Nonlinear Yield Criterion, Theor. Appl. Fract. Mech., vol. 47, no. 1, pp. 46–56, 2007.
- **7.** A. Keshavarz and Z. Pooresmaeil, Static and Seismic Active Lateral Earth Pressure Coefficients for C- $\phi$  Soils, Geomech. Eng., vol. 10, no. 5, pp. 657–676, **2016**.
- **8.** A. Keshavarz and Z. Pooresmaeil, Evaluation of the Static and Seismic Active Lateral Earth Pressure for c-φ Soils by the ZEL Method," Sci. Iran., no. March, **2016**.
- 9. D. Benmeddour, M. Mellas, R. Frank, and A. Mabrouki, Numerical Study of Passive and Active Earth Pressures of Sands, Comput. Geotech., vol. 40, pp. 34–44, 2012.
- **10.** J. S. Lee, H. G. Chae, D. S. Kim, S. B. Jo, and H. J. Park, Numerical Analysis of Inverted T-type Wall Under Seismic Loading, Comput. Geotech., vol. 66, pp. 85–95, **2015**.
- **11.** J. S. Shiau, C. E. Augarde, A. V. Lyamin, and S. W. Sloan, Finite Element Limit Analysis of Passive Earth Resistance in Cohesionless Soils, Soils Found., vol. 48, no. 6, pp. 843–850, **2009**.
- **12.** H. Lu and B. Yuan, Calculation of Passive Earth Pressure of Cohesive Soil Based on Culmann 's Method, Water Sci. Eng., vol. 4, no. 50539110, pp. 101–109, **2011**.
- **13.** C. A. Coulomb, Essai Sur Une Application Des Règles de Maximis & Minimis à Quelques Problèmes de Statique, Relatifs à L'architecture, des Sci. Mem. MATh. Phys. Par Divers Savants, vol. 7, pp. 343–382, **1776**.
- 14. W. J. M. Rankine, On The Stability of Loose Earth, 1857.
- 15. V. R. Greco, Active Earth Thrust on Cantilever Walls with Short Heel, Can. Geotech. J., vol.



38, pp. 401–409, **2001**.

- **16.** A. T. C. Goh, Behavior of Cantilever Retaining Walls, J. Geotech. Eng., vol. 119, no. 11, pp. 1751–1770, **1993**.
- **17.** Y. M. Cheng, Seismic Lateral Earth Pressure Coefficients for c-φ Soils by Slip Line Method, Comput. Geotech., vol. 30, no. 8, pp. 661–670, **2003**.
- **18.** H. A. Kamiloğlu, E. Şadoğlu, and F. Yılmaz, Numerical Analysis of Active Earth Pressures on Inverted T Type and Semi-Gravity Walls, in 3<sup>rd</sup> International Conference on Advanced Engineering Technologies, **2019**.



The International Conference of Materials and Engineering Technology

## FATİH YILMAZ<sup>1</sup>, HAKAN ALPER KAMİLOĞLU<sup>1</sup>

<sup>1</sup> Bayburt University, Engineering Faculty, Civil Engineering Department, Bayburt, TURKEY.

# Özet

Zemin stabilizasyonu, problemli zeminlerin mühendislik özelliklerini iyileştirmede kullanılan en yaygın yöntemdir. Kimyasal stabilizasyon, zayıf zeminlerin tahrip edici etkilerini en aza indirmek için alternatif bir yöntemdir. Bu çalışmada, otoklavlanmış gaz beton ve uçucu külün zemin stabilizasyonunda kullanılabilirliği araştırılmıştır. Elek analizi, likit limit, plastik limit, kompaksiyon ve serbest basınç dayanımı testleri yapılmıştır. Ek olarak, hazırlanan karışımların donma-çözülme döngüsünün etkisi altında davranışları incelenmiştir. Elde edilen veriler ışığında, zemin stabilizasyonu çalışmalarında uçucu kül ve otoklavlanmış gaz beton atıkları kullanımının dayanma değerlerini arttırdığı tespit edilmiştir. Mukavemet değerlerindeki artışa ek olarak, donma-çözülme etkisi altında daha kararlı davranışlar ortaya çıkmıştır.

Anahtar Kelimeler: Zemin stabilizasyonu, otoklavlanmış gaz beton, uçucu kül, serbest basınç dayanımı, donma-çözülme etkisi

#### 1. Giriş

Geoteknik mühendisliği açısından elverişsiz olarak kabul edilen fiziksel, kimyasal, mekanik ve hidrolik özelikleri zayıf olan zeminlerin bu özelliklerinin amaçlanan bir mühendislik uygulamasında kullanılmak üzere iyileştirilmesine zemin stabilizasyonu denilmektedir. Zemin stabilizasyonu genel manasıyla, zeminlerin sahip olduğu özelliklerin iyileştirilmesidir. Stabilizasyon tekniklerindeki ana gaye, mühendislik özellikleri zayıf olan zeminlerin hacimsel stabilite, mukavemet, permeabilite, dayanım ve durabilite parametrelerinin zeminin kullanılacağı projedeki amacına uygun hale getirilmesidir. Zeminin mühendislik özellikleri istenilen düzeyde olmadığında karşılaşılan problemleri çözmek için farklı metodlar uygulanmaktadır. Bunlar; zeminin kaldırılıp yeni zeminle ikame edilmesi, üstyapıda boyut ve proje değişikliklerine gidilmesi veya zeminin stabilize edilmesi olarak sıralanabilir.

Zemin stabilizasyonu çalışmaları genellikle mekanik ve kimyasal stabilizasyondan oluşur. Mekanik stabilizasyon, katkı maddeleri kullanılmaksızın yapılan iyileştirme yöntemidir. Kimyasal stabilizasyon ise katkı maddelerinin farklı oranlarda kullanılmasıyla yapılan iyileştirme yöntemidir. Zeminin geoteknik özellikleri belirlendikten sonra kullanılacak stabilizasyon tekniğine karar verilmelidir. Bu geoteknik özelliklerin belirlenmesinin ardından zemin için en uygun stabilizasyon yöntemi veya yöntemleri seçilerek iyileştirme çalışmaları yapılmalıdır [1].

Puzolanlar kimyasal bileşenleri bakımından silikat ve alüminat (SiO<sub>2</sub> ve Al<sub>2</sub>O<sub>3</sub>) esaslı maddelerdir. Bu çalışmada gaz beton atıkları puzolanik özellikteki madde olarak kullanılmıştır. Puzolanlar kendi başlarına bağlayıcı değildirler. Birincil bağlayıcıya ihtiyaç duyarlar. Çalışma kapsamında birincil bağlayıcı olarak uçucu kül kullanılmıştır. Uçucu kül zemine katıldığında bünyesindeki kalsiyum oksit, silikat ve alüminatın hidratasyona uğramasıyla kuvvetli bağlar oluşturarak zemin danelerini bir arada tutmaktadırlar [2]. Uçucu küllerin inceliğinin artması mekanik dayanım artışlarını meydana getirir. Uçucu küller, kimyasal kompozisyonlarına göre C ve F sınıfı olarak adlandırılırlar. CaO miktarı % 10'dan fazla olan grup C sınıfı uçucu külleri oluştururlar [3].

The International Conference of Materials and Engineering Technology

Kumar ve Sharma uçucu külü %0, %5, %10, %15 ve %20 oranlarında kullanarak zeminin serbest şişme yüzdesi ve basıncı, plastisite, kompaksiyon, dayanım ve geçirimlilik değerlerini incelemişlerdir ve uçucu kül miktarının artmasıyla hazırlanan karışımlarda serbest basınç dayanımı değerlerinde artış görüldüğü sonucuna varmışlardır [4]. Kumar vd. tarafından yapılan başka bir çalışmada ise polyester fiber, kireç ve uçucu külün birlikte kullanımının şişen zeminlerin stabilizasyonunda uygun olduğu belirlenmiştir [5]. Brooks tarafından yapılan çalışmada %12 pirinç kabuğu külü ve %25 uçucu kül ile birlikte, bu katkılar için en iyi dayanımlar elde edilmiştir [6].

Yılmaz tarafından yapılan çalışmada killi bir zemine %0, %5, %10, %15, %20, %25, %30 oranlarındaki uçucu kül ilave edilerek hazırlanan karışımlarda en iyi dayanım değerleri %25 uçucu kül içeren karışımlardan elde edilmiştir [7].

Bu çalışma kapsamında birincil bağlayıcı olarak kullanılan uçucu külün ve ikincil bağlayıcı olarak değerlendirilen gaz beton atıklarının birlikte kullanılabilirliği irdelenmiştir. Stabilizasyon çalışmasında doğal malzemeye öncelikle %20 oranında uçucu kül katılmıştır. Bunun yanı sıra gaz beton atıklarının etkisinin irdelenebilmesi amacıyla %0, %5, %10, %15 ve %20 oranlarında gaz beton atıkları ilave edilerek deney numuneleri elde edilmiştir. Hazırlanan karışımların dayanım parametreleri incelenmiştir.

#### 2. Materyal ve Metod

Deneylerde kullanılan zemin Bayburt ili Demirözü ilçesindeki bir araştırma çukurundan alınmıştır. Birleşik zemin sınıflama sistemi ASTM D 2487 standardına göre çalışmada kullanılan zeminin sınıfı CL olarak belirlenmiştir. Uçucu kül Afşin-Elbistan Termik Santralleri'nden temin edilen C sınıfı uçucu küldür. Gaz beton atıkları ise Bayburt çevresindeki inşaatlardan laboratuvara getirilmiş ve öğütme işlemi neticesinde stabilizasyon çalışmalarında kullanılmıştır. Doğal zemine ait temel özellikler ve deney numuneleriyle ilgili adlandırılmalar Tablo 1 ve Tablo 2'de sırasıyla sunulmuştur.

USCS sınıflandırma	CL
Likit limit, LL (%)	46
Plastik limit, PL (%)	28
Plastisite indisi, PI (%)	18
Optimum su içeriği, w <sub>opt</sub> (%)	21
Maksimum kuru yoğunluk,	1.56

## Tablo 1. Zemin özellikleri



#### Tablo 2. Deney numuneleriyle ilgili notasyonlar

Deney numunelerinin karışım oranları	Notasyon
Doğal zemin	S
Zemin ve uçucu kül	SFA
Zemin, uçucu kül ve gaz beton atığı	SFAAC
Zemin, uçucu kül ve %5 gaz beton atığı	SFAAC/5
Zemin, uçucu kül ve %10 gaz beton atığı	SFAAC/10
Zemin, uçucu kül ve %15 gaz beton atığı	SFAAC/15
Zemin, uçucu kül ve %20 gaz beton atığı	SFAAC/20

# 3. Sonuçlar ve Öneriler

Karışımların indeks özelliklerini belirlemek amacıyla yapılan kıvam limitleri deney sonuçları Tablo 3'te sunulmuştur. Çalışma kapsamında belirlenen 7, 28 ve 84 günlük kür sürelerinin ardından gerçekleştirilen serbest basınç testleri sonucunda elde edilen veriler Tablo 4 ve Şekil 1 yardımıyla gösterilmiştir.

Tablo 3. Kıvam limitleri				
Numune	Likit Limit	Plastik Limit	Plastisite İndisi	
SFA	45	23.7	21.3	
SFAAC/5	36.8	27.3	9.5	
SFAAC/10	34.6	27.9	6.7	
SFAAC/15	33.4	28.2	5.2	
SFAAC/20	32.8	28.6	4.2	

<b>Tablo 4.</b> Serbest basing dayanını sonaçıan				
Numune	7 gün (kPa)	28 gün (kPa)	84 gün (kPa)	
S	175.3	178.7	179.4	
SFA	754.4	942.8	1020.1	
SFAAC/5	944.9	1159.6	1298.5	
SFAAC/10	819.4	1152.9	1168.9	
SFAAC/15	751.5	1026.6	1030.1	
SFAAC/20	526.3	910.9	968.2	

Tablo 4. Serbest basınç dayanımı sonuçları



Şekil 1. Dayanım sonuçları

Tablo 3'te sunulan kıvam limitleri incelendiğinde uçucu kül ve gaz betonun beraber katkı maddesi olarak kullanılmasının likit limit değerlerini azalttığı, plastik limit değerlerinde kayda değer değişimler meydana getirmediği, plastisite indisi değerlerini azalttığı görülmektedir. Tablo 4 ve Şekil 1'e göre sadece zemin içeren karışımların 7, 28 ve 84 günlük kür süreleri sonucunda dayanım değişimine uğramadıkları, sadece uçucu kül içeren karışımlarda ise doğal zemine oranla çok ciddi dayanım artışlarının meydana geldiği görülmektedir. Bu sonuçlar literatür ile uyumlu ve beklenen verilerdir. ÇAlışmasının ana gayesi olan iki katkı maddesinin beraber kullanılabililiği incelendiğinde ise özellikle SFAAC/5 ve SFAAC/10 numunelerinde gaz beton atıklarınının dayanıma etkisi görülmektedir.

Elde edilen veriler ışığında gaz beton atıklarının uçucu kül ile beraber zemin stabilizasyonu çalışmalarında kullanılmasının mukavemet değerlerini sadece uçucu kül kullanımına göre %10-20 daha fazla arttırdığı sonucuna varılmıştır. Donma-çözülme çevrimi sonucunda uçucu kül ve gaz beton atığı içeren karışımlarda sadece uçucu kül içeren karışımlara göre daha durabil davranışlar tespit edilmiştir. Bu sonuçlar geoteknik açıdan önemli olduğu kadar çevre mühendisliği açısından atıkların bertarafı için de ayrı bir önem teşkil etmektedir. Çalışmayı arazi deneyleriyle veya ıslanma-kuruma deneyi, geçirimlilik deneyi gibi deneylerle genişletmek mümkündür. Uçucu kül yerine farklı birincil bağlayıcılar kullanarak elde edilen sonuçlar irdelenebilir.

# Kaynaklar

- [1] Yılmaz, F. Tüfit Taşların Zemin Stabilizasyonunda Kireçle Birlikte Kullanılabilirliğinin Standart Deneyler ve Bilgisayarlı Tomografi Tekniği ile Araştırılması, Doktora Tezi, Karadeniz Teknik Üniversitesi Fen Bilimleri Enstitüsü, 2015.
- [2] Çokça, E., İpek, T., Kireç, Çimento, C ve F-sınıfı Uçucu Kül Katkısının Şişen Bir Zeminin Şişme Basıncına Etkisi, Zemin Mekaniği ve Temel Mühendisliği Sekizinci Ulusal Kongresi, (10), 2, 1998.
- [3] Erdinç, M., "Uçucu Küllü Betonlarda Dayanım ve Klor Geçirimsizliği", İstanbul Teknik Üniversitesi Fen Bilimleri Enstitüsü, 100 s., İstanbul, 1995.



- [4] Kumar, B., R., P. ve Sharma, R., S., Effect of Fly Ash on Engineering Properties of Expansive Soils, Journal of Geotecnical and Geoenvironmental Engineering, 130, 7, 764-767, 2004.
- [5] Kumar, A., Walia, B. S. ve Bajaj, A., Influence of Fly Ash, Lime and Polyester Fibers on Compaction and Strength Properties of Expansive Soils, Journal of Materials in Civil Engineering, 19, 3, 242-248, 2007.
- [6] Brooks, R., M., Soil Stabilization with Fly Ash and Rice Husk Ash, Int. Journal of Research and Reviews in Applied Sciences, 1, 3, 209-217, 2009.
- [7] Yılmaz, F., Zemin Stabilizasyonunda Uçucu Kül Kullanımı, ISEM2016, 3rd International Symposium on Environment and Morality, 4-6 November 2016, Alanya Turkey, 2016.

# HİDROKSİAPATİT TOZ KATKILI ELEKTRİKSEL KIVILCIMLA İŞLENMİŞ TI6AI4V ALAŞIMININ MİKROSERTLİK DEĞİŞİMLERİ

The International Conference

of Materials and Engineering Technology

# NİHAL EKMEKCİ<sup>1</sup>, YASİN EFE<sup>2</sup>

<sup>1</sup>Zonguldak Bülent Ecevit Üniversitesi, Makina Mühendisliği Bölümü, Zonguldak, TÜRKİYE. <sup>2</sup>Zonguldak Bülent Ecevit Üniversitesi, Fen Bilimleri Enstitüsü, Makina Müh. Anabilim Dalı, Zonguldak, TÜRKİYE.

#### Özet

Toz Katkılı Elektriksel Kıvılcımla İşleme (TKEKİ) yönteminde, mikrosaniyeler bazında çok yüksek noktasal sıcaklar işlenen yüzeyde ve kesitte birçok metalürjik karakteristiğin değişimine sebep olmaktadır. Yöntemle işlenen numune kesitlerinde oluşan ve kalınlıkları birkaç mikrondan başlayan katmanlar üzerinden sertlik ölçümleri almak hayli güçleşmektedir. Bu çalışma kapsamında, Hidroksiapatit (HA) toz katkılı elektriksel kıvılcımla işlenmiş Ti6Al4V alaşımının yüzey özellikleri Taramalı Elektron Mikroskopu (SEM) ve Enerji Dağılım Spektrometresi (EDS) ile, kesit mikroyapısı ise optik mikroskop ile incelenmiştir. Kesitte, oluşan katmanlar üzerinde değişen sertlik değerleri mikro sertlik cihazı vasıtasıyla ölçülmüştür. Ölçümlerde Vickers uç ile, HV 0.025 kg (245.175 mN) kuvvetinde yük 25 saniye boyunca uygulanmıştır. İşlenen numune kesitlerinde ana metalden yüzeye çıkıldıkça sertliğin arttığı, eriyip tekrar katılaşan katman üzerinde bu değerin yaklaşık 1700 HV'a ulaştığı belirlenmiştir. Ayrıca işleme sırasında dielektrik sıvıldan yüzeye HA toz katkısı nüfuziyeti gözlemlenmiştir.

**Anahtar Kelimeler:** Ti6Al4V, Hidroksiapatit, Toz Katkılı Elektriksel Kıvılcım İle İşleme(TKEKİ), Mikrosertlik

#### 1. Giriş

Elektriksel Kıvılcımla İşleme (EKİ); iş parçası ile elektrot arasında oluşturulan kıvılcımların termal etkileriyle malzeme şekillendirme yöntemidir. Yöntemle, çok sert, yüksek mukavemetli ya da yüksek sıcaklık malzemeleri gibi malzemelerin işlenmesi çok daha olanaklı hale gelmektedir. Yöntem, elektrot ile iş parçası arasında fiziksel bir temas olmaksızın, karmaşık geometrilere sahip küçük ve narin parçaların da işlenmesine olanak sağlamaktadır. Mikrosaniyeler içinde çok yüksek noktasal sıcaklara (~20000 °K) ulaşılmakta ve bu sıcaklıklar işlenen yüzeyde ve kesitte birçok metalürjik değişime sebep olmaktadır. İlaveten darbe akımı, darbe süresi ve dielektrik sıvı gibi birçok işleme parametresinin bulunması da işlemi oldukça karmaşık kılmaktadır. Öncelikle, 1770 yılında İngiliz bilim adamı Joseph Priestly elektriksel boşalmanın aşındırıcı etkisini fark etmiştir. Sonrasında 1930'larda metal ve elmasın elektriksel kıvılcım ile işlenmesi yapılmıştır. 1980'lerde EKİ'de Bilgisayarlı Sayısal Kontrolün (CNC) kullanılması ile işleme operasyonlarının verimliliği artarak dikkate değer bir gelişme sağlanmıştır. Böylece uyarılmalı bir kontrol sistemi tarafından sertliğinden bağımsız olarak elektriksel olarak iletken olan herhangi bir malzemenin işlenmesi sağlanmıştır. 1974 yılında, EKİ ile işlenmiş çelik ve bakır iş parçası yüzeyleri optik ve taramalı elektron mikroskopu ile incelenmiş ve yüzeylerin kompleks yapısına değinilmiştir. Sadece hızlı ısınma ve soğumanın değil, birbirinden ayrık rastgele kıvılcımların yüzeyleri bu denli kompleks kıldığı ve sadece konvansiyonel pürüzlülük ölçümlerinin yüzeylerin özelliklerini göstermek


Toz Katkılı Elektriksel Kıvılcımla İşleme (TKEKİ) yönteminde ise, dielektrik sıvı içerisine toz katkılarının katılmasıyla yüzey özelliklerinin ve mekanik özelliklerinin iyileştirilmesi amaçlanmıştır. Dielektrik sıvıya katılan tozlar da tabakaların metalürjik özelliklerini etkilemektedir. Toz tanelerinin alt ve üst kısımlarında sıralanan pozitif ve negatif elektrik yükleri dolayısıyla toz zincirleri oluşmakta böylece elektrik alan yoğunluğu azalarak yalıtkanlığın kırılması kolaylaşmaktadır. Elektriksel olarak iletken olan tozlar sayesinde iş parçası ile elektrot arasındaki kıvılcımlar daha kararlı ve yoğun hale gelmektedir. Böylece yüzey kalitesi ve talaş kaldırma oranı artmaktadır. [10]. SKD – 61 çeliğinin işlenmesinde dielektrik sıvıya Al ve Cr tozlarının eklenmesiyle korozyon direnci ve yüzey sertliğinin daha iyiye gittiği gözlemlenmiştir. Ayrıca kullanılan tozlar işleme sırasında yüzeye de nüfuz etmektedir [11]. Literatürde dielektrik sıvı içinde; grafit, alüminyum ve silisyum karbür tozu gibi toz katkılarının kullanımı[12] ve yüzeye toz katkısı nüfuziyetini içeren çalışmalar mevcuttur[13]. Hidroksiapatit toz katkısının kullanımına ilişkin çalışmalara ise son yıllarda rastlanmaktadır [14, 15].

Bu çalışmada Hidroksiapatit toz katkılı EKİ yöntemiyle işlenmiş Ti6Al4V alaşımının kesit mikroyapısı ve sertliğindeki değişimler incelenmiştir. İşlenen yüzeyden ana metale inildikçe 120 µm'ye varan mesafede meydana gelen katmanlar üzerinde oldukça hassas ve zorlu ölçümlerle her bir tabakada oluşan mikrosertlikler belirlenmiştir. Eriyip tekrar katılaşan katman üzerindeki sertliklerin 1700 HV'a ulaştığı gözlemlenmiştir.

#### 2. Malzeme ve Yöntem

Çalışmada kullanılan numuneler, Ti-6Al-4V (Grade 5) titanyum alaşımından boyları 8 mm, çapları ise 10 mm olacak şekilde hazırlanmıştır. Numunelere 600 <sup>0</sup>C'de gerilim giderme işlemi uygulanmış ve yüzeyleri zımpara ile temizlenip, parlatılmıştır. Sonrasında, Ajan CNC Model 983 marka 4 eksenli dalma tip erozyon tezgahında Hidroksiapatit toz katkılı EKİ (HA-TKEKİ) yöntemiyle işlenmişlerdir. Deiyonize suyun içine 15 gram/litre (g/l) olacak şekilde HA tozları karıştırılmıştır. Hazırlanan dielektrik sıvının mevcut sisteme zarar vermemesi için, tezgaha ayrıca bir havuz ve karıştırıcıdan oluşan sistem adapte edilmiştir (Şekil 1). Tozların havuz içindeki sirkülasyonu, 1000 dev/dak. olacak şekilde mekanik bir karıştırıcı ile sağlanmıştır. İşlemelerde, 25 mm çaplı saf titanyum malzemeden elektrot kullanılmıştır. Kullanılan HA tozları ortalama 20 nm çapında, küresel formda ve %99 saflıktadır. Sertlik ölçümlerini alabilmek için tabaka kalınlıklarının daha geniş olduğu 800 µs darbe süresi tercih edilmiştir. Darbe süresini sabit tutarak düşük ve yüksek akımdaki farkları da görebilmek amacıyla 7A ve 42 A darbe akımları seçilmiştir. Böylece 7 A, 800 µs ve 42 A, 800 µs işleme parametrelerinde 2 numune işlenmiştir.

The International Conference

aterials and Engineering Technology



Şekil 1. Elektro erozyon tezgahı ve deney setinin genel görünümü

İşlenen numunelerin ilk olarak Tylor Hobson yüzey pürüzlülük cihazı ile yüzey pürüzlülükleri ölçülmüştür. Sonrasında numunelerin QUANTA 450 Field Emission Gun (FEG) yüksek çözünürlüklü taramalı elektron mikroskobu (SEM) ile yüzey görüntüleri ve Enerji Dağılım Spektrometresi (EDS) ile elementel dağılım izleri alınmıştır. Yüzey incelemeleri biten numuneler işlenen yüzeyden tabana doğru kesilerek bakalite alınmış, zımparalarla parlatılmış ve Weck dağlama ajanı (5 g Amonyum Biflörür ve 100 ml saf su) ile dağlanarak kesitleri incelenmiştir. Kesitte oluşan katmanlar üzerindeki sertlik değişimleri Shimadzu HMV-G21 (Vickers, Knoop Indenter) mikro sertlik cihazı ile ölçülmüştür.

# 3. Sonuçlar ve Değerlendirme

#### 3.1 Yüzey pürüzlülüğü

HA-TKEKİ yöntemiyle işlenmiş numunelerin yüzeyinden 10 adet pürüzlülük ölçümü alınmış ve ortalama pürüzlülük değeri hesaplanmıştır (Tablo 1). Yöntemin doğası birçok parametreyi



içermektedir. Dielektrik sıvının özellikleri, darbe akımı, darbe süresi, elektrot ve iş parçası (numune) özellikleri yüzey ve yüzey altı karakterizasyonunu doğrudan etkilemektedir.

Tablo 1. HA-TKEI	KI yöntemiyle işlenmiş numunel	erin yüzey pürüzlülükleri
İşleme Parametreleri	Ortalama pürüzlülük (µm)	Standard Sapma (µm)
7 A, 800 μs	4.34	0.75
42 A, 800 μs	5.87	1.15

Kıvılcım atmasının sonlanmasıyla eriyen malzemenin oluşturduğu kraterler, darbe akımının artmasıyla derinleşmekte, darbe süresinin artmasıyla da yayvanlaşmaktadır. Bu durum pürüzlülük üzerinde etkilidir fakat salt olarak sadece bu iki parametreye bağlanamaz. Çünkü pürüzlülük üzerinde belirtilen dielektrik sıvı, toz katkıları gibi diğer parametreler de etkilidir. Burada aynı darbe süresinde işlenmiş iki numunede darbe akımının artmasıyla pürüzlülüğün de arttığı açıkça görülmektedir. Darbe akımının oluşturduğu derin kraterlerin etkisi gözlemlenmektedir.

# 3.2 SEM ve EDS Analizleri

HA-TKEKİ yönteminde, darbe süresi boyunca olgunlaşan elektriksel alanın kırılmasıyla ikincil hatta üçüncül kıvılcımlar oluşmaktadır. Bunlar yüzey üzerinde dalgalanmalara ve küçük krater yapılarının oluşumuna sebep olmaktadır. İşlenen numunelerde, yüzey üzerinde mikrosaniyeler içerisinde oluşan ard arda kıvılcımların ve eriyip ayrılan metalin oluşturduğu kraterler, yönelim izleri ve hızla katılaşan parçacıkların hakim olduğu görülmektedir (Şekil 2). 42 A darbe akımında işlenen numunede yüksek darbe süresinin etkisiyle bu dalgalanmaların daha derin ve belirgin olduğu göze çarpmaktadır.



a)



**Şekil 2.** HA-TKEKİ yöntemiyle işlenmiş numunelerin yüzey görüntüleri a) 7 A, 800 μs b) 42 A, 800 μs

TKEKİ yönteminde işleme sırasında yüzeye dielektrik sıvıdan hatta elektrottan malzeme transferi gerçekleşmektedir. Böylece fonksiyonel yüzeyler oluşmaktadır [15]. Özellikle HA'ca zengin yüzeylerin oluşumu tıbbi uygulamalarda talep edilmektedir. Her iki parametrede işlenen numune yüzeylerine bakıldığında dielektrik sıvıdan yüzeye nüfuz etmiş HA yapılarına rastlanmaktadır. HA yapısındaki Ca/P oranı, mol yüzdesi yaklaşık olarak 1,67 olarak bilinmektedir. EDS izlerinin analizinde yaklaşık olarak bu molar oranı gözlemlenmektedir. Yüzeydeki HA bakımından zengin bölgeler ana metale göre daha koyu renkte görülmektedir. Darbe akımına göre kıyaslanacak olursa 7 A düşük darbe akımında işlenen numunede (Şekil 3) 42 A yüksek darbe akımında işlenen numuneye (Şekil 4) göre bölgesel olarak daha yoğun nufüziyet gözlemlenmektedir.



Şekil 3. HA-TKEKİ yöntemiyle 7 A, 800 µs'de işlenmiş numunenin EDS izleri



Şekil 4. HA-TKEKİ yöntemiyle 42 A, 800 µs'de işlenmiş numunenin EDS izleri

# 3.3 Mikroyapı ve Mikrosertlik Analizleri

EKİ ile işlemede numune kesitlerinde işlenmiş yüzeyden ana malzemeye inildikçe farklı özelliklerde katmanlar bulunmaktadır. Kesitten bakıldığında en üstte yer alan katman, dağlamaya dirençli, ana metale göre çok daha sert ve genellikle beyaz renkte gözlemlenen eriyip tekrar katılaşan katmandır [6]. Bu katmandan sonra hızlı ısısal çevrimler sonucu ısıl etkilenmiş katman ve son olarak da ana malzeme yer almaktadır (Şekil 5).



Şekil 5. HA-TKEKİ yöntemiyle işlenmiş numune kesiti

EDM'deki karmaşık fiziksel olaylar ve oluşan katmanların inceliği, mikro sertliği incelemeyi zorlaştırır. Ayrıca, tabaka kalınlıkları, işleme parametrelerine göre değişmektedir ve aynı numune üzerinde bölgeden bölgeye değişmektedir. Bu bağlamda, incelenen tüm numunelerin kesit yüzeylerinde, her katmanın ortalama kalınlık değerine en yakın kalınlığa sahip bölgelerde sertlik ölçümleri yapılmıştır. Ölçümlerde Vickers uç ile, HV 0.025 kg (245.175 mN) kuvvetinde yük 25 saniye boyunca uygulanmıştır. Böylece yüzeyden başlayarak her kattan ana malzemeye doğru ölçümler alınmıştır (Şekil 6). Ölçümlere göre, eriyip tekrar katılaşan katmanın sertliği, ana metale inildikçe logaritmik olarak düşmektedir ve ana metalin ortalama sertliği 365 HV civarındadır. TKEKİ sırasında meydana gelen hızlı termal çevrimler ve faz dönüşümleri, oluşturulan katmanların mikro sertliğini önemli ölçüde etkilemektedir. 7 A darbe akımında işlenen numunede (Şekil 6a) de, 42 A darbe akımında işlenen numunede (Şekil 6 b) de en yüksek mikro sertlik değeri eriyip tekrar katılaşan katman üzerinde yaklaşık 1700 HV olarak saptanmıştır. Isıl etkilenmiş katmanda ise bazı sapmalar hariç 400-500 HV civarında tespit edilmiştir.



Şekil 6. HA-TKEKİ yöntemiyle işlenmiş numune kesiti üzerinde alınan sertlik izleri a) 7 A, 800 µs b) 42 A, 800 µs

# 4. Sonuçlar

Çalışma kapsamında HA-TKEKİ yöntemiyle 7 A, 800 µs ve 42 A, 800 µs işleme parametrelerinde işlenmiş iki numune incelenmiştir.



MET

The International Conference of Materials and Engineering Technology

- EDS analizlerinde işleme sırasında dielektrik sıvıdan yüzeye HA tozlarının nufuziyeti • görülmüştür. Düşük akım seviyesinde daha fazla toz katkısı gözlemlenmiştir.
- Her iki numunede kesit üzerinde oluşan tabakalardan alınan mikrosertlik ölçümlerinde • ana malzemeden işlenen yüzeye doğru ilerledikçe sertliğin arttığı gözlemlenmiş ve eriyip tekrar katılaşan katmanda bu değerin 1700 HV'ye ulaştığı gözlemlenmiştir. İsil etkilenmiş katmanda ise mikrosertlik değeri ana malzemeye göre yaklaşık % 25 artış göstermiştir.

# 5. Kaynaklar

- 1. Crookall, J.R. ve Khor, B.C., Electro-discharge machined surfaces. Proceedings of the Fifteenth International Machine Tool Design and Research Conference, September 18-20, England, 1974, 1:373-384.
- 2. Abu Zeid O.A., On the effect of electro discharge machining parameters on the fatigue life of AISI D6 tool steel. Journal of Materials Processing Technology, 1997, 68:27–32.
- 3. Zhang, Q.H., Zhang, J.H., Ren, S.F., Niu, Z.W., Ai, X., A theoretical model of surface roughness in ultrasonic vibration assisted electrical discharge machining in gas. Int. J. Manufacturing Technology and Management, 2005, 7:381-390.
- 4. Kunieda, M. ve Furuoya, S., Improvement of EDM efficiency by supplying oxygen gas into gap. Annals of the CIRP, **1991**, 40(1):215-218.
- 5. Rebelo, J.C., Morao Dias, A., Kremer, D., Lebrun, J.L., Influence of EDM pulse energy on the surface integrity of martensitic steels. Journal of Materials Processing Technology, **1998**, 84:90–96.
- 6. Ekmekci, B., White layer composition, heat treatment, and crack formation in electric discharge machining process. Metallurgical and Materials Transactions B, 2009, 40(B), 70-81.
- 7. Jabbaripour, B., Sadeghi, M. H., Faridvand, Sh., Shabgard, M. R., Investigating the effects of EDM parameters on surface integrity, MRR and TWR in machining of Ti-6Al-4V. Machining Science and Technology, 2012, 16:419–444.
- 8. Ekmekci, N., Farklı dielektrik ortamlarda elektriksel kıvılcımla işlenmiş DIN 1.4442 çift fazlı çeliğin mikroyapı ve mikrosertlik değişimleri. Karaelmas Fen ve Müh. Dergisi, 2016, 6(2):412-422.
- 9. Ekmekci, N., Keskin, İ., Surface characteristics and hardness variations in electrical discharge machining of enhanced nitrogen in vanadium steels. Metallurgical and Materials Transactions B, **2019**, 50(B):98-109.
- 10. Zhao, W.S., Meng, Q.G., Wang, Z.L., The application of research on powder mixed EDM in rough machining. Journal of Materials Processing Technology, 2002, 129:30-33.
- 11. Hwa Yan, B., Lin, Y.C., Huang, F.Y., Wang, C.H., Surface modification of SKD 61 during EDM with metal powder in the dielectric, Materials Transactions, 2001, 42(12):2597-2604.



- Ekmekçi, B., Ulusöz, F., Ekmekci, N., Yaşar, H., Suspended SiC particle deposition on plastic mold steel surfaces in powder-mixed electrical discharge machining. Proc IMechE Part B: J Engineering Manufacture, 2014, 1–12. doi:10.1177/0954405414530902.
- **13.** Öpöz, T.T, Yaşar, H., Ekmekci, N., Ekmekci, B., Particle migration and surface modification on Ti6Al4V in SiC powder mixed electrical discharge machining. Journal of Manufacturing Processes, **2018**, 31:744–758.
- **14.** Ekmekci, N. ve Ekmekci, B., Electrical discharge machining of Ti6Al4V in hydroxyapatite powder mixed dielectric liquid. Materials and Manufacturing Processes, **2016**, 31(13):1663-1670.
- 15. Ekmekci, N., Akbaş, S., Mikronize Hidroksiapatit Toz Katkılı Elektriksel Kıvılcımla İşlenmiş Tİ-6AL-4V Alaşımının Yüzey Özelliklerinin İncelenmesi ve Yüzeye Toz Katkısı Nüfuziyetinin Değerlendirilmesi. Uludağ Üniversitesi Mühendislik Fakültesi Dergisi, 2019, 24(1):193-206.



he International Conference of Materials and Engineering Technology

# ALPEREN TOZLU<sup>1</sup>, HAMİD YILMAZ<sup>1</sup>

<sup>1</sup>Bayburt University, Engineering Faculty, Mechanical Engineering Department, Bayburt, TURKEY.

#### Abstract

Organic Rankine cycle (ORC) is a well-known power production method in order to generate electricity from low-grade heat sources such as geothermal ground water, waste heat. The cycle consists of an evaporator, an ORC turbine, a condenser and a pump. The thermodynamic performance of cycle can be varied with respect to thermodynamic properties of working fluid and heat source as well as other properties of the ORC. This basic cycle should be considered as a fundamental of power production cycles to absolutely learn by undergraduate students. Due to this reason, a novel software, which is called Organic Rankine Cycle Design Teaching Software (ORCDTS), is performed for educational purposes in this study. The aim of the software is to support teaching activity for power production process of an ORC system and understanding thermodynamic relations behind the system. Thus, fundamental thermodynamic knowledge of undergraduate students can be improved by utilizing the conservation of mass, first and second law of thermodynamics into the ORC. The proposed VORCD is easy to use in the design stage of an ORC and suitable to support teaching thermodynamic relations for undergraduate students.

Keyword: organic Rankine cycle, power cycle, teaching activity, visual software.

#### **1. Introduction**

The technological developments in the 21st century, the modernization of industrial facilities and the innovative changes in the field of life are manifested in education. In this scope, there are various innovations for the engineers who lead the technological developments in order to carry out their undergraduate education more effectively. These innovations are also equally important, necessary and valid for mechanical engineering students. The developments in thermodynamics, which is undoubtedly one of the most fundamental areas of mechanical engineering, are very significant in the recent days when the energy demand is required all over the world. Better analysis of power production systems by undergraduate students can be contributed both to themselves and the society in which they live [1].

There are several studies in the open literature in order to solve thermodynamic problems by using the computer courseware. Liu [2] presented a computer courseware based on C# in order to solve three types of main thermodynamic problems which are determination of gas status, evaluation of a thermodynamic properties of pure substance at certain state and analysis of simple thermodynamic cycles. Tan and Chua [3] performed the application of thermodynamic properties of steam through the Java applets. It was concluded that the Java applets compute thermodynamic properties of steam and R134a refrigerant. The calculated values were compared to the standard reference properties tables for steam and refrigerant and the results were found accurate. Ngo and

Lai [4] carried out a Web-based Thermodynamics Tables Wizard and how it can be used for online courses or web-teaching to accommodate the student's requirement at different levels in looking up thermodynamics properties.

The International Conference

As mentioned above, researchers are focused on improving skills of engineering students in the thermodynamic fields. In this study, a novel design courseware is proposed in terms of teaching activity for power production process of an organic Rankine cycle (ORC) and understanding thermodynamic relations behind the system.

# 2. Organic Rankine Cycle

Organic Rankine cycle (ORC) is an important low-grade thermal energy recovery technology because of its small-scale feature. ORC can be used to all kinds of low-temperature heat sources including geothermal energy, solar energy, biomass energy, and especially waste heat energy [5-7].

An ORC has less components when compared to other cycles. Due to this reason, it is very important for undergraduate students to understand the ORC systems within the scope of the thermodynamics. Thus, they can apply thermodynamic laws to the more complex systems and they can perceive their working principles. For this reason, ORC was chosen for visual education software. An ORC is consisted of an evaporator (EVAP), an ORC turbine (OT), an ORC condenser (OC) and an ORC pump (OP). A typical ORC schematic layout is shown in Figure 1.



Figure 1. Schematic layout of the organic Rankine cycle

# 3. Visual Organic Rankine Software Design

ORC Education Software is designed to teach organic Rankine cycle for the students. The software is coded in C# object-oriented programming language. .NET allows coders to develop applications for desktop running on a language-independent framework. In the software, there are two basic windows. One of them is thermodynamic properties and the other one is calculation window. With the "Start Visual ORC" button as seen in Figure 2, the software is directed the user to the problem window.

Technology

aterials and Engineering



JWE

Figure 2 Welcome window of the software

In this part of the software, there are 5 test problems in cycle part. Within this window, the user can proceed with selecting any of these test problems as seen in Figure 3. After the cycle is selected, the cycle is visualized to the user as seen in Figure 4. At the same time, the user enters thermodynamic properties of the cycle and also the decision parameters by clicking the related button. At the end of the procedure, the net power output, the turbine and the pump power outputs, the energy and the exergy efficiencies of the system is performed by using the "Calculation" button which is shown in Figure 5.

💀 Form2		—	×
Cycles Cycle 1 Cycle 2 Cycle 3 Cycle 4 Cycle 5 Select Problem	Thermodynamic Properties         1         2         3         4         5         6         7         8         Decision Parameters         1         1         1         2         3         4         5         6         7         8         1         1         1         1         1         1         1         1         1         1         2         3         4         5         6         7         8         1         1         1         1         1         1         1         1         1         2         1         1         1         1         1         1         1         1		
Calculate			

Figure 3. Problem property window of the software







Figure 5. ORC calculation window

# 4. Results and Discussion

UWE Bristol

The novel design courseware which is performed in C# programing language can support the undergraduate mechanical engineering students to better understand basic thermodynamic principles, thermodynamic analyze approaches, as well as improve them the capability in solving analytical problems. Due to the perceiving of thermodynamic laws and the efficiency of novel design courseware, it can be applied in thermodynamic education at mechanical engineering



departments. Moreover, this study can also verifies that the development and application of instructional courseware are simple but effective tools in enhancing the teaching quality.

#### References

- [1] Z. L. Kahn-Jetter and P. A. Sasser, Using spreadsheets for studying machine design problems involving optimization, Computer Applications in Engineering Education, 5, 199-211, 1997.
- [2] Y. Liu, Development of instructional courseware in thermodynamics education, computer applications in engineering education, 19(1), 115-124, 2011.
- [3] F. L. Tan and P. S. K. Chua, Java applet on computation of thermodynamic properties of steam and R134a refrigerant, Computer Applications in Engineering Education, 11(4), 211-225, 2004.
- [4] C. Wu and D. C. Sherrill, Intelligent computer aided design, analysis, optimization, and improvement of thermodynamic systems, Computer Applications in Engineering Education, 9(4), 220-227, 2001.
- [5] Ozahi E., Tozlu A. and Abusoglu A., Thermoeconomic multi-objective optimization of an organic Rankine cycle (ORC) adapted to an existing solid waste power plant, Energy Conversion and Management, 168, 308-319, 2018.
- [6] D. Li, S. Zhang, and G. Wang, Selection of organic Rankine cycle working fluids in the low-temperature waste heat utilization, Journal of Hydrodynamics, 27, 458-464, 2015.
- [7] Ozahi E., Tozlu A. and Abusoglu A., Thermodynamic performance assessment of different fluids in a typical organic Rankine cycle for usage in municipal solid waste power plant, Acta Physica and Polonica A, 132, 807-812, 2017.

# U KESİTLİ PARÇALARDA İKİ FARKLI RADYÜSLE GERİ YAYLANMA TELAFİSİ

The International Conference of Materials and Engineering Technology

# TUĞÇE TURAN ABİ<sup>1</sup>, SELÇUK KERVANCI<sup>1</sup>, AHMET YILMAZ<sup>1</sup>

<sup>1</sup>Beyçelik Gestamp Teknoloji ve Kalıp Fabrikası, Arge, Bursa, TÜRKİYE.

#### Abstract

Geri yaylanma, U-kesitli sac metal parçaların formlanmasında ana problemdir. Geri yaylanmanın tasarım aşamasında tahmini ve telafi edilmesi, endüstriyel bakış açıları için çok önemlidir. Gerekli geometrileri istenen doğrulukta ve kısa sürede elde etmek, şirketlerin rekabet yönlerini arttırmaktadır. U formundaki parçalarda geri yaylanma telafisi, çok sayıda analiz ve atölye çalışması gerektirir. U formundaki parçalarda, dikey formların tek bir form işleminde istenen açıda şekillendirilmesi sac levhanın geri yaylanması nedeniyle mümkün değildir. Bu nedenle, iki form işlemi kullanılarak, birinci form işlemine belirli bir değere ulaşılır ve ikinci form işlemi, U biçiminde istenen değerden daha fazla olan açıklığı kapatmaya çalışır. Bu çalışmada, düz sac metal levhaya 2 bükme işlemi uygulanarak; U form ve 90 derecelik dikey duvar oluşumunu etkileyen parametrelerin belirlenmesi amaçlanmıştır. Bu amaçla 5 farklı sac kalınlığı (1,2-1,8-2-2,5-3,5 mm), 2 farklı sac kalitesi (HR340LA, DP600), 4 farklı radyüs değeri (4-8-16-32mm) ve 3 farklı küçültme oranı (% 5-10-15) kullanılmıştır. Tasarım aşamasında zaman ve maliyetin azaltılması için toplam 192 test gerçekleştirilmiş ve parametrelerin yaylanma üzerindeki etkisi araştırılmıştır.

Keyword: Springback, U form, Sac metal levha, formlanabilirlik

#### 1. Giriş

Sac metal formlama, uzun yıllardır başarıyla uygulanmış olsa da, tüm işlem hala tam olarak anlaşılmamıştır. İstenen ürüne en uygun tasarımı belirlemek için masraflı ve zaman alıcı bir deneme yanılma süreci gerekmektedir. [1]. Mühendislik uygulamasında, geri yaylanma formlanan parçaların (özellikle U-bükme parçaları) boyutsal doğruluğunu ve şeklini etkiler. Geri yaylanma, parçanın kalıptan çıkarılmasından sonra biçiminin ve boyutlarının değişmesine yol açar. Deforme olmuş malzemedeki gerilme ve gerilme durumunun değişmesine yol açabilir[2]

Sac metal şekillendirme işlemleri, özellikle otomotiv ve ilgili endüstrilerde seri üretimde yaygın olarak kullanılmaktadır. Bununla birlikte, sac parça preslemede yüksek mukavemetli çeliklerin kullanımının artması, kolayca çözülemeyen şekillenebilirlik ve şekil bozulma problemlerini ortaya çıkarmaktadır [3]. Geometrik toleransları karşılamak ve montaj gereksinimlerini kolaylaştırmak adına, geri yaylanmayı telafi etmek için bazı otomotiv kalıpları kalıp geliştirme ve denemeler sırasında üç ila beş kez yeniden işleme yapmaktadır. Tekrarlı kalıp işlemelerinin, deneme süresinin ve maliyetlerin azaltılması, otomotiv sac metal şekillendirme endüstrisine önemli faydalar sağlayacaktır [4]



Çalışmada, her kalite sac için (HR340LA ve DP600), farklı sac kalınlıklarında (1,2-1,8-2-2,5-3,5 mm) deneme yapılması planlanmıştır. Her kalite ve kalınlık için 4 farklı radyüs değeri (4-8-16-32mm) ve her radyüs değeri için de 3 farklı küçültme oranı (%5-10-15) kullanılmıştır (Şekil 1). Bu denemeler için iki operasyonlu bir adet tandem gözlü kalıp üretilmiştir (bkz. Şekil 3) Kalıbın iki operasyonu da u bükme işlemi yapmak için tasarlanmıştır. Küçültme oranının %75'i ilk operasyonda radyüse artı değer olarak ilave edilmiş ve kalan %25'i ikinci operasyonda radyüse eksi değer olarak ilave edilmiştir. Belirtilen şartlara göre toplam 192 adet durum ile ilgili deneme çalışması yapılmıştır. Deneme sonuçları minitab üzerinde değerlendirilerek sonuçlandırılmıştır.

The International Conference

aterials and Engineering Technology

Tablo 1. HR340LA ve DP60	0 malzeme mekanik	k özellikleri
Malzeme Mekanik Özellikleri	HR340LA	DP600
Akma Gerilmesi, MPa (min)	340	330
Akma Gerilmesi, MPa (max)	420	410
Çekme Gerilmesi, MPa (min)	410	600



Şekil 1. Parametreler

Projede, 2 adet form operasyonu kullanıldığı belirtildiği üzere; ilk form operasyonun iki duvar arası açıklığı belirli bir değere gelmekte ve ikinci form operasyonunda da U formdaki istenen değerden fazla olan açıklık kapatılmaya çalışılmıştır. Örneğin; orijinal radyüs 4 mm'de %5 küçültme oranında 1. Operasyonda radyüs %75 artırıldığında 4,15mm, 2.Operasyonda %25 azaltıldığında 3.95 mm olmaktadır. Her iki operasyonda radyüs gösterimi Şekil.2'de gösterilmiştir.



Şekil 2. Radyüs gösterimi

Belirlenen parametreleri atölye şartlarında denemek için yapılan prototip kalıp görseli Şekil 3'te gösterilmiştir.



Şekil 3. a) Kapalı kalıp b)Üst kalıp c) Alt kalıp tasarımı

Belirlenen kalıp tasarımı ile atölye şartlarında denemeler için kalıp imalatı gerçekleştirilerek (Şekil 4) u kesitli parçalarda iki duvar arası mesafeyi belirleyen parametreler incelenmiştir.



Şekil 4. Kalıp reel görüntüsü

Her deneme sonrası numunelerde açıklık manuel olarak ölçülmüştür (Şekil 5).



Şekil 5. Ölçüm çalışmaları

# 3. Bulgular ve Tartışma

Çalışma sonucunda iki dik duvar arası açıklık mesafesi farklı kalınlık ve kaliteye göre farklılıklar göstermiştir (Şekil 6).

HR340LA malzeme için aynı orijinal radyüs ve aynı kalınlıkta saclarda küçültme oranı arttıkça dik duvar (90 derece)'a daha çok yaklaşmıştır. Genel olarak aynı küçültme oranı ve orijinal radyüse sahip levhalarda sac kalınlığı arttıkça, geri yaylanma azalmış ve dik duvar elde etmek daha kolay olmuştur. Ayrıca genel olarak baktığımızda radyüs 32 mm'de dik duvar elde etmek zor olmuştur, geri yaylanma en fazla radyüs 32'de görülmüştür (Tablo 2).



Tablo 2. HR340LA atölye denemeleri operasyon açıklıkları						
Malzeme	Sac Kalınlığı	Orjinal Radyüs	Küçültme Oranı	1.Op. Sonrası Açıklık	2.Op. Sonrası Açıklık	
HR340LA	1,80	4	%5	93,0	92,5	
HR340LA	2,0	4	%5	93,2	92,5	
HR340LA	2,5	4	%5	91,5	91,5	
HR340LA	3,5	4	%5	91,7	91,5	
HR340LA	1,80	4	%10	92,5	91,8	
HR340LA	2,0	4	%10	93,5	92,5	
HR340LA	2,5	4	%10	91,2	91,2	
HR340LA	3,5	4	%10	91,0	91,0	
HR340LA	1,80	4	%15	93,5	90,5	
HR340LA	2,0	4	%15	93,2	91,2	
HR340LA	2,5	4	%15	92,5	90,5	
HR340LA	3,5	4	%15	92,0	90,8	
HR340LA	1,80	8	%5	94,4	92,5	
HR340LA	2,0	8	%5	94,9	93,5	
HR340LA	2,5	8	%5	92,5	92,0	
HR340LA	3,5	8	%5	92,7	91,0	
HR340LA	1,80	8	%10	94,5	91,0	
HR340LA	2,0	8	%10	95,0	91,5	
HR340LA	2,5	8	%10	92,8	90,5	
HR340LA	3,5	8	%10	92,5	90,0	
HR340LA	1,80	8	%15	95,2	92,0	
HR340LA	2,0	8	%15	95,0	92,5	
HR340LA	2,5	8	%15	93,0	91,2	
HR340LA	3,5	8	%15	93,0	90,0	
HR340LA	1,80	16	%5	97,0	95,0	
HR340LA	2,0	16	%5	97,3	95,4	
HR340LA	2,5	16	%5	94,0	92,5	

TICMET'19

The International Conference of Materials and Engineering Technology

UWE Bristol Bristol	جامعة حمد بن خليفة HAMAD BIN KHALIFA UNIVERSITY	TI	CME	r'19	echnology
		The Int	ernational Conference of Ma	rerials and Engine	
HR340LA	3,5	16	%5	93,8	91,8
HR340LA	1,80	16	%10	97,0	94,0
HR340LA	2,0	16	%10	95,2	92,2
HR340LA	2,5	16	%10	94,5	93,0
HR340LA	3,5	16	%10	93,5	90,0
HR340LA	1,80	16	%15	97,8	94,5
HR340LA	2,0	16	%15	97,7	94,2
HR340LA	2,5	16	%15	94,0	91,5
HR340LA	3,5	16	%15	93,5	90,0
HR340LA	1,80	32	%5	101,0	98,8
HR340LA	2,0	32	%5	101,5	99,5
HR340LA	2,5	32	%5	96,4	96,0
HR340LA	3,5	32	%5	93,5	93,0
HR340LA	1,80	32	%10	102,0	98,0
HR340LA	2,0	32	%10	101,5	98,3
HR340LA	2,5	32	%10	97,0	97,0
HR340LA	3,5	32	%10	94,5	92,0
HR340LA	1,80	32	%15	101,7	97,7
HR340LA	2,0	32	%15	101,5	98,5
HR340LA	2,5	32	%15	96,8	95,0
HR340LA	3,5	32	%15	94,0	90,5

DP 600 Malzeme için deneme sonuçlarına bakıldığında aynı orijinal radyüse ve aynı küçültme oranına sahip sac levha grubunda sac kalınlığı arttıkça geri yaylanma azalmıştır ve 90 derece dik açıya daha çok yaklaşmıştır (Tablo 3).

896



	<b>Tablo 3.</b> DP600 ato	lye denemelei	rı operasyon açı	ikliklari	
Malzeme	Sac Kalınlığı	Orjinal Radyüs	Küçültme Oranı	1.Op. Sonrası Açıklık	2.Op. Sonrası Açıklık
DP600	1,2	4	%5	95,0	93,5
DP600	1,8	4	%5	94,5	93,7
DP600	2,0	4	%5	93,6	92,8
DP600	2,5	4	%5	93,2	92,5
DP600	1,2	4	%10	94,0	93,0
DP600	1,8	4	%10	93,5	92,0
DP600	2,0	4	%10	94,0	91,8
DP600	2,5	4	%10	93,5	91,7
DP600	1,2	4	%15	95,2	92,7
DP600	1,8	4	%15	94,0	91,6
DP600	2,0	4	%15	93,2	91,0
DP600	2,5	4	%15	93,0	92,0
DP600	1,2	8	%5	97,5	95,0
DP600	1,8	8	%5	96,5	94,5
DP600	2,0	8	%5	95,0	93,9
DP600	2,5	8	%5	93,7	92,9
DP600	1,2	8	%10	99,0	93,0
DP600	1,8	8	%10	96,5	92,0
DP600	2,0	8	%10	95,5	91,9
DP600	2,5	8	%10	94,0	91,0
DP600	1,2	8	%15	99,7	94,5
DP600	1,8	8	%15	97,5	93,7
DP600	2,0	8	%15	96,5	92,8
DP600	2,5	8	%15	94,0	91,5
DP600	1,2	16	%5	104,0	100,5
DP600	1,8	16	%5	99,5	97,5
DP600	2.0	16	%5	99.2	97.0

CMET'1

The International Conference of Materials and Engineering Technology

9

	المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة الم المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة الم المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة المعاملة الم	TI	CME		echnology
		The Inte	ernational Conference of Ma	terials and Ling.	
DP600	2,5	16	%5	96,5	94,5
DP600	1,2	16	%10	105,5	99,5
DP600	1,8	16	%10	100,0	96,2
DP600	2,0	16	%10	99,0	94,5
DP600	2,5	16	%10	96,0	93,0
DP600	1,2	16	%15	104,5	99,0
DP600	1,8	16	%15	101,8	96,9
DP600	2,0	16	%15	99,7	95,0
DP600	2,5	16	%15	96,1	94,0
DP600	1,2	32	%5	111,5	108,0
DP600	1,8	32	%5	105,8	102,8
DP600	2,0	32	%5	103,5	101,0
DP600	2,5	32	%5	99,2	96,8
DP600	1,2	32	%10	112,5	106,2
DP600	1,8	32	%10	107,5	102,5
DP600	2,0	32	%10	104,5	99,5
DP600	2,5	32	%10	99,5	97,4
DP600	1,2	32	%15	113,0	106,2
DP600	1,8	32	%15	107,5	102,0
DP600	2,0	32	%15	104,5	99,0
DP600	2,5	32	%15	99,7	96,8

HR340LA ile DP600 kalitelerinde sac malzemelerin sonuçları kıyaslandığında Tablo 4.'de görüldüğü gibi malzeme kalitesi arttıkça dik duvar elde etmek zorlaşmaktadır, geri yaylanma miktarı daha fazla olmaktadır.



ials and Engineering Technology



Şekil 6. Farklı kalite ve kalınlık için numune sonuçları

Parametre ve ölçüm sonuçlarının verileri minitab programına girilerek değerlendirilmiştir (Şekil 7,8).



aterials and Engineering Technology

UWE Bristol

Şekil 7. Minitab sonuçları- ana etki grafiği



Minitab' ta yapılan regresyon analizi sonucu olması gereken duvar açısı formülü elde edilmiştir ( denklem 1).

İlk Op. Duvar Açısı = 93,73 + 0,01118 Çekme Dayanımı - 3,258 Sac Kalınlığı(mm)+ 0,392 İlk Radyüs - 0,112 İkinci Radyüs(1)

900



Projede düz sac levhaya 2 kes bükme operasyonu uygulayarak U form ve 90 derece dik duvara sahip parça elde etmeye etki eden parametrelerin belirlenmesi amaçlanmıştır. Bu amaçla sac kalınlığı, malzeme cinsi(çekme mukavemeti), operasyonda uygulanacak radyüs değerleri parametre olarak alınmıştır. 2 kez tekrar yapılarak toplam 192 adet deneme yapılmıştır. Deneyde 2 Adet çıktı vardır. Bunun sebebi ilk operasyon sonrası ve ikinci operasyon sonrası ölçüm alınmasıdır. Amaç ikinci operasyon sonunda 90 dereceyi elde etmektir. Minitab analizlerine göre;

The International Conference of Materials and Engineering Technology

- Sac Kalitesi arttıkça (HR340LA →DP600) 2 bükme operasyonu sonrası dik duvar açısından uzaklaşmaktadır.
- Sac Kalınlığı arttıkça 2 bükme sonrası dik duvar elde etmek kolaylaşmaktadır.
- R4 ve R32 için ilk ve ikinci radyüs değerleri birbirinden ne kadar uzaksa dik duvara o kadar çok yaklaşmaktadır. R8 ve R16 için bir optimum değer mevcut. Bir süre sonra artırmak dezavantaj olmaktadır.
- Malzeme kalitesi arttıkça dik duvar elde etmek zorlaşmaktadır.
- 3,5 mm'lik sac neredeyse tüm şartlarda en iyi dik duvar açısı sağlamaktadır.
- 1,2 m' lik sac neredeyse tüm şartlarda en kötü dik duvar açısı sağlamaktadır.

**Teşekkür:** Bu çalışma Beyçelik Gestamp Teknoloji ve Kalıp Fabrikası A.Ş. Arge merkezi tarafından yürütülen bir projedir. Proje giderleri firma öz kaynaklarından karşılanmıştır.

#### Kaynaklar:

- 1. Meinders, T., Burchitz, I.A., Bonte, M.H.A., Lingbeek, R.A., Numerical product design: Springback prediction, compensation and optimization, International Journal of Machine Tools & Manufacture.2008, 48, (499–514)
- Nanu, N., Brabie, G., Analytical model for prediction of springback parameters in the case of U stretch-bending process as a function of stresses distribution in the sheet thickness, Int. J. Mech. Sci.2012, 64,(11-21)
- **3.** Esener, E., Sönmez, E., Özsoy, M. and M. Fırat, Determining Springback Behavior of High-Strength Steels via Channel Forming Process, ICCESEN 2016
- 4. Konzack, S., Radonjic, R., Liewald, M., Altan, T., Prediction and reduction of springback in 3D hat shape forming of AHSS, 17th Interantional Conference on Metal Forming, Metal Forming 2018, 16-19 September 2018

# ESTIMATION OF ACUTE WATER TOXICITY AGAINST DAPHNIA MAGNA USING THE LEVENBERG-MARQUARDT NEURAL NETWORK METHOD

The International Conference of Materials and Engineering Technology

# ALI YASAR<sup>1</sup>, ISMAIL SARITAS<sup>2</sup>

<sup>1</sup>Selcuk, University, Technology Faculty, Department of Mechatronics Engineering, Konya, TURKEY <sup>2</sup>Selcuk, University, Technology Faculty, Department of Electrical and Electronics Engineering, Konya, TURKEY

#### Abstract

In this study, an ANN model was developed from a data set of 546 organic molecules to predict acute water toxicity to Daphnia magna. The input values of our data set consisted of eight molecular identifiers that encoded information about lipophilicity, formation of H-bonds, polarization, polar surface area, electrophilicity and nucleophilicity. As a result of this data input, LC50 output value was calculated with NN. When our dataset and ANN values are compared, it is shown that an accuracy value of 79.2% is calculated.

Keyword: Water Toxicity, Daphnia Magna, UCI Database, ANN

#### 1. Introduction

Toxicant is a poison made by humans or entering the environment through human activities. Many pesticides are toxic substances. Most of the chemicals involved in water are soluble in water, which can have adverse effects on aquatic systems. These water-soluble chemical structures can damage aquatic species and food webs. They may also harm mammals, birds and other organisms in the ecosystem [1]. Acceptable concentrations of heavy metals in the aquatic environment are normally assessed by evaluating the lethal concentration in acute poisoning [2]. The adverse effects of the toxicants can be induced through non-specific or specific mechanisms of action. Non-specific interactions, e.g. narcosis and overall reactivity result from high concentrations of toxic substances in the cell or cellular membrane. It is therefore strongly associated with the ability of chemicals to enter the organism. Some chemicals may interact directly with biological targets in the aquatic organism, resulting in higher toxicity. These interactions or reactions generally occur between the toxic agent and the critical cellular macromolecules. The assessment of aquatic toxicity of chemicals is the most important element to be addressed [1]. Recently, Artificial Neural Networks (ANNs) have been used successfully in many applications. In this study, we have tried to calculate the LC50 value by training the artificial neural network.

#### 2. Materials and Methods

Data we used for our work has been obtained from UCI machine learning database. This dataset was used to develop quantitative regression QSAR models to predict acute aquatic toxicity towards the fish Pimephales promelas (fathead minnow) on a set of 908 chemicals. to predict acute aquatic toxicity towards Daphnia Magna. LC50 data, which is the concentration that causes death in 50%



he International Conference

#### 2.1. Dataset

Data set containing values for 8 attributes (molecular descriptors) of 546 chemicals used to predict quantitative acute aquatic toxicity towards Daphnia Magna. Out of these 546 values 382 were used in training, 137 were used for testing and 27 were used for validation.

#### 2.2. Levenberg-Marquardt Algorithm

In function optimization problems with artificial neural networks, the training algorithm is designed to determine the best network parameters to minimize network error. Various function optimization methods can be applied to ANN model education. One of these methods is the Levenberg-Marquardt algorithm [4]. Levenberg-Marquardt (LM) algorithm is preferred because of the speed and stability it provides in training artificial neural networks [5]. Levenberg-Marquardt (LM) algorithm is a quadratic derivative algorithm and it increases the learning speed effectively [6,7,8]. As a result of the study performed according to LM algorithm, error histogram graph is given in Figure 1 and, train, validation, test and regression graph of all data are given in Figure 2.



Technology

aterials and Engineering

903



Figure 45. Regression Plotter

#### **3. Results and Discussion**

In artificial neural networks, the LM algorithm produces uniform results on the QSAR aquatic toxicity Data Set. This work has been done using MATLAB and ANN toolbox. LM gave more accurate results. When the results are considered LM algorithm in ANN has shown to give successful results.

#### 4. Conclusions

In artificial neural networks, the LM algorithm produces uniform results on the QSAR aquatic toxicity Data Set. This work has been done using MATLAB and ANN toolbox. LM gave more accurate results. When the results are considered LM algorithm in ANN has shown to give successful results.

#### **References:**

1. Cassotti, M., Ballabio, D., Consonni, V., Mauri, A., Tetko, I. V., & Todeschini, R. (2014). Prediction of acute aquatic toxicity toward daphnia magna by using the GA-k NN method. Alternatives to Laboratory Animals, 42(1), 31-41.

The International Conference of Materials and Engineering Technology

- 2. Peres, I., & Pihan, J. C. (1991). Copper LC50 to Cyprinus carpio. influence of hardness, seasonal variation, proposition of maximum acceptable toxicant concentration. Environmental technology, 12(2), 161-167.
- 3. URL https://archive.ics.uci.edu/ml/datasets/QSAR+aquatic+toxicity#
- 4. Sharifahmadian, A. (2015). Numerical Models for Submerged Breakwaters: Coastal Hydrodynamics and Morphodynamics. Butterworth-Heinemann.
- Çavuşlu, M. A., Becerikli, Y., & Karakuzu, C. (2012). Hardware implementation of neural network training with Levenberg-Marquardt algorithm. Turk J Elec Eng & Comp Sci, 5, 31-38.
- Wilamowski, B.M., Chen, Y., 1999, Efficient algorithm for training neural Networks with one hidden layer, in Proc. of the International Joint Conference on Neural Networks, Cilt: 3, 1725-1728.
- Dohnal ,J., 2004, Using of LevenbergMarquardt method in identification by neural networks, In Student EEICT 2004. Student EEICT 2004. Brno: Ing. Zdeněk Novotný CSc., 2004, pp. 361 - 365, ISBN 80-214-2636-5.
- Khosravi, Z,M.H., Barghinia, S., Ansarimehr, P., 2006, New momentum adjustment technique for Levenberg-Marquardt neural network used in short term load forecasting, in Proc. of 21<sup>st</sup> International Power System Conference (PSC 2006), Tehran, Iran.

# EXPERIMENTAL STUDY ON INVESTIGATION OF MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF AL B4C COMPOSITE MATERIAL

The International Conference of Materials and Engineering Technology

# Necip Fazil YILMAZ<sup>1</sup>, Musa YILMAZ<sup>2</sup>, Mahmut Furkan KALKAN<sup>3</sup>

<sup>1</sup>Gaziantep University, Engineering Faculty, Mechanical Engineering Department, Gaziantep, Turkey

#### Abstract

Today, due to the high strength to weight ratios of aluminum alloys and composites, their usage areas are increasing day by day. The objective of this experimental study is to characterize boron carbide reinforced with Al matrix composites with under different casting conditions. The addition of  $3.0 \ \mu m \pm 0.5 \ \mu m$  B4C particles with different weight fractions mixed with pure melted Al. The mixture of Al B4C was held under several holding times at 1000° C. Pouring of mixture was achieved after stirring process at 300 rpm for 5 minutes. Optimum charpy impact tests and hardness test results were researched. In addition to mechanical tests and SEM device were used to observe the microstructure of B<sub>4</sub>C reinforced Al.

Keyword: Stir Casting, Metal Matrix Composites, Boron Carbide

#### **1.INTRODUCTION**

Composite consists of two different part, they are reinforced and matrix phases. These phases are macroscopically combined and are not soluble between each other. Composite materials are classified according to the geometry of the reinforcement which are flake, particulate and fibers or according to the type of, metal matrix composites (MMCs), ceramic matrix composites (CMCs) and matrix such as polymer matrix composites (PMCs) [1,2].

Metal matrix composites (MMCs) are a new group of advanced materials reinforced with other metals, organic compounds and ceramics. Reinforcements in the metal matrix are generally dispersed in the matrix to improve the features of the base metal. Such composite materials are frequently used in many areas of daily life and industry due to their light weight, low thermal expansion and high strength properties.[2-4].

Aluminum or its alloys, which have good engineering features, but most important one is low density- high strength. Because of this reason these aluminum or its alloys materials are generally preferred as matrix material in metal matrix composites [3-6]. B<sub>4</sub>C particles are the mainly used in metal matrix composite materials as a reinforced material where aluminum and its alloys are used as matrix. Among the known elements and compounds, boron carbide is one of the hardest materials know in literature. The selection of reinforcement materials is important because of the compatibility with the matrix materials to be used together and ease of supply in the production of metal matrix composite. B<sub>4</sub>C is a suitable reinforcement material for many applications in engineering due to high hardness, good strength and low density [5-8].

In this study,  $B_4C$  reinforced Al matrix composites were casted with using stirring operation at 1000°C. Main parameters of these experiments are holding time and additions rate of  $B_4C$ . The samples produced under different conditions were investigated by mechanic tests and



The International Conference of Materials and Engineering

#### 2. Material Method

In this study, pure aluminum and mostly 3 microns  $B_4C$  was used. The chemical composition of used aluminum is given in Table 1.

<b>Table 1.</b> Chemical composition of pure aluminum							
Al	Cu	Fe	Mg	Mn	Si	Zn	Other
99,95	0,005	0,015	0,001	0,005	0,02	0,001	0,002

During the experimentation, stir casting method was used to produce aluminum-boron carbide composites in 12 different casting conditions which are listed in Table 2 depending on the holding time and addition rate of boron carbide. Boron carbide addition was performed within 5, 10, 15 and 20 weight percentage of pure aluminum and casting process were carried out at temperature  $900^{\circ}$ C. All samples were also hold 30, 45 and 60 minutes in the oven at elevated temperatures before casting into dies.

During the last 5 minutes holding period, the molten material was thoroughly mixed with the graphite rod. At the end of the period the dross were cleaned and molded to produce boron carbide reinforced aluminum matrix composite.

Samula No	Casting	Holding	Addition Rate
	Temperature	Time	(%)
1	1000	30	5
2	1000	30	10
3	1000	30	15
4	1000	30	20
5	1000	45	5
6	1000	45	10
7	1000	45	15
8	1000	45	20
9	1000	60	5
10	1000	60	10
11	1000	60	15
12	1000	60	20

All samples were subjected to impact and hardness tests to observe the variations of mechanical properties.

The Impact test samples were prepared to have 3 identical samples for each condition. The samples were 55 mm long and 10x10 mm cross section. All test specimens have "v" type notch with the degree of 45° and 2 mm depth. Experimentation of impact tests were applied by Tinius Olsen charpy type impact test device. Hardness of the composites was measured using Micro Vickers

Technology

907

hardness method. Hardness test values were evaluated by averaging the results of 3 successive measurements in all samples.

he International Conference

aterials and Engineering Technology

In microstructural examination, specimens were carefully grinded and polished for reducing scratches and damages. All samples were grinded with 360,600, 1000 mesh SiC emery papers. After grinding, polishing was performed by using a diamond solution  $(0.3\mu m)$ . Metkon Forcipol 2V grinding and polishing device was used for grinding and polishing operation. Polished samples were etched for 50 seconds. Etching reagent contains 25 ml methanol, 25 ml hydrochloric acid, 25 ml nitric acid and 1 drop hydroflouric acid. At the end of etching process, samples were cleaned with alcohol. Microstructure of the composites samples were investigated by Zeiss Gemini Sem.

#### **3. Result and Discussion**

After AL B<sub>4</sub>C composite sample was produced by using stir casting method, impact test and hardness tests were applied to the samples to compare the effect of parameters on mechanical properties. Microscopic and SEM images are also used in this section to clarify the microstructure examinations of the material produced at  $1000^{\circ}$  C. Experimental results are discussed according to parameters such as boron carbide addition rate and retention time.

#### **3.1 Impact Test Result**

Impact test results are represented in Table 3. The optimum impact energy value of 10,2 was obtained 20 % boron carbide addition and 45 min holding duration. The impact energy variations of the samples produced under different conditions as a function of holding time and addition rate are showed in Figure 1.

Table 5. Impact test results (kg m)							
	Pure	%5 B4C	%10 B <sub>4</sub> C	%15 B <sub>4</sub> C	%20 B <sub>4</sub> C		
30 min.	2,8	9,8	7,8	8,2	9,4		
45 min.	5,0	9,6	9,6	9,5	10,2		
60 min.	4,5	9,8	9,6	8,8	9,5		

 Table 3. Impact test results (kg\*m)







b)

Figure 1. Variations of impact energy according to a) Holding time b) Addition rate

It is obtained that there is a powerful relationship between the holding time and impact energy. Increasing the holding time to 45 minutes was observed to increase the impact resistance while no significant difference was observed in increasing the holding time to 60 minutes. On the other hand, as shown in figure 1a, there is a slightly small decrease in impact resistance during a holding time of 60 minutes. When figure 1b. is examined, the addition rate of boron carbide appears to be highest impact resistance at 20%. Figure 2 shows the best specimen that gives highest impact energy.



Figure 2. Sample with the highest impact energy (%15 B<sub>4</sub>C and 45 min.)

# **3.2 Hardness Test Result**

Hardness test results of specimens under different conditions are given in Table 4. Variations of the hardness results of the samples as a function of holding time and addition rate are displayed in Figure 3. Similar to impact properties, higher hardness values were observed when the holding time was 30 minutes and 5%  $B_4C$  addition rate. However, further increasing holding time and addition rate caused a reduction in hardness values.



# TICMET 19 The International Conference of Materials and Engineering Technology

<b>Tuble 4.</b> That alless test results							
	%5 B <sub>4</sub> C	%10 B <sub>4</sub> C	%15 B <sub>4</sub> C	%20 B <sub>4</sub> C			
30 min.	31,75	28,2	25,2	26,8			
45 min.	21,24	26	27,7	27,6			
60 min.	23	22,2	19,4	25			

 Table 4. Hardness test results



b)

Figure 3. Hardness variations of the samples according to a) Holding time b) Addition rate



Figure 5. SEM image of produced Al-B<sub>4</sub>C composite

Figure 5 shows the scanning electron micrograph image (SEM) of the same sample. Boron carbide particles are clearly seen in Figure 5 and particles are marked in red. In general, it has been observed that the boron carbide have a homogeneous distribution. According to the SEM image, boron carbide particles have a nearly square shape.

# 4. Conclusion

In this paper, stir casting operations has been achieved successfully and composite material has been produced. It is possible to say that mechanical properties of aluminum can be enhanced by reinforced with  $B_4C$ .

By imposing various boron carbide additions and holding times on the samples, the following conclusions can be drawn from this study:

- The impact strength of composite samples produced under different 12 condition is much better than pure aluminum.
- According to the hardness test results, the maximum hardness value was obtained from 30 minutes holding duration and 5% B<sub>4</sub>C addition.
- Optimum impact strength was observed in the samle using 45 minutes holding duration and 20% B<sub>4</sub>C addition.
- > It has been observed that holding duration and addition rate of  $B_4C$  are important parameters in the production of  $B_4C$  reinforced Al composites.
- Microscopic observations showed that B<sub>4</sub>C particles were homogeneously dispersed in general.



### References

1. Kaw A. K., Mechanics of composite materials, CRC press. 2005

**2.** Kerti, I., Toptan, F., "Microstructural variations in cast B4C-reinforced aluminum matrix composites (AMCs)", Materials Letters, Vol. 62, pp. 1215-1218, 2008

**3.** RAMULU M., RAO P.N., KAO H. Drilling of (Al2O3)p/6061 metal matrix composites [J]. Journal of Materials Processing Technology. 124: 244–254. 2002

4. Terry B., Jones G., Metal Matrix Composite. Oxford. 1990.

**5.** ASM Specialty Handbook. (1993) Aluminum and Aluminum Alloys. ASM International Press **6.** Gilbert Kaufman J., Properties of Aluminum Alloys; Tensile, Creep, and Fatigue Data at High and Low Temperatures. ASM International 2002

**7.** P. T. B. SHAFFER, in "Engineered Materials Handbook,"edited by M. M. Gauthier (ASM, Metals Park, OH, 1991) vol. 4, p. 804

**8.** Lillo, T.M., Enhancing Ductility of AL6061 + 10 wt.%B4C Through Equal-Channel Angular Extrusion processing. Materials Science & Engineering. A410-411: 443-446. 2005
# AN EXTREME LEARNING MACHINE APPROACH FOR DETECTION OF EPILECTIC SEIZURE

The International Conference of Materials and Engineering Technology

## Ilker Ali OZKAN<sup>1</sup>, Abdulkadir SADAY<sup>2</sup>

<sup>1</sup>Selcuk University, Faculty of Technology, Computer Engineering Department, Konya, TURKEY. <sup>2</sup>Selcuk University, Faculty of Technology, Konya, TURKEY.

#### Abstract

Irregularities in the electrical activities of the brain can cause epileptic attacks. In the diagnosis and classification of epileptic attacks and epilepsy, data showing the electrical activity of the brain obtained by EEG has an important place. This study presents an EEG classification approach based on the extreme learning machine (ELM). The ELM algorithm is used for the characteristics of single hidden layer feedforward neural network (SLFN). EEG recordings of healthy individuals and individuals who had epileptic attacks were classified by using ELM. As a result of the study, the classification success of the method we applied was found to be 99.67%. It is predicted that the obtained system may be useful in evaluating the pre-diagnosis for physicians.

Keywords: Extreme Learning Machine, Classification, Electroencephalogram, Epileptic Seizure

### **1. Introduction**

The extreme learning machine (ELM) is a learning algorithm applied to single hidden layer feedforward neural networks (SLFNs) [1]. The ELM does not need to adjust the hidden layer weights. The weights between the input layer and the hidden layer are assigned randomly. The weights between the hidden layer and the outputs are calculated analytically [1, 2]. The ELM structure which can output only the local minimums by solving a single linear equation, contains fewer design parameters than conventional neural network-based classifiers [1, 3]. In this way, the learning process of the model is realized very fast. In addition to its ability to learn quickly, ELM is superior to feed-forward networks that learn with conventional back-propagation algorithms in terms of generalization success [1, 4]. Furthermore, offers high performance by minimizing the training errors and norm of output weights for binary classification, multiple classification and regression problems [5].

Classifiers built using the Excessive Learning Machine (ELM), which has superior capabilities such as rapid learning and generalization, are now used in many areas [3, 4, 6, 8].

Transient electrical disorders of the brain cause epilepsy. Sometimes seizures cannot be detected, and sometimes they are confused with migraine and impact events. Approximately one in every hundred people has epileptic attacks in their lives [9, 10]. Although different methods are available for the treatment of epilepsy, these seizures cannot be controlled in 25% of patients [11]. Epilepsy patients may encounter many problems in their social lives. Different tests are performed for the diagnosis of epilepsy. The most important of these is electroencephalography (EEG). EEG is a sign shows the electrical activity in the brain and is a commonly used method [12]. The placement of its electrodes given in Figure 1.



**Figure 1.** Electrode locations for EEG recording according to the international 10-20 system [13].

Detection of discharges between epilepsy seizures with EEG has an importance in the diagnosis of epilepsy [9, 14]. For this reason, the EEG device is used extensively in neurology clinics for the observation of epileptic attacks. Different classification models have been developed so far due to the complex nature of the epileptic EEG signal [15]. There are two types of abnormal EEG recordings as interictal and ictal in EEG recordings obtained from people suffering from epilepsy. Interictal signals consist of signals between epileptic seizures, while ictal consists of signals obtained during epileptic seizures [14, 16].

Tzallas et al. [17] stated that it is important to detect seizures automatically during long-term EEG recordings in their study. They proposed a method based on time frequency analysis on EEG signals. They used ANN to classify the properties that they obtained. In the model they developed, they have achieved a classification success of 97.72%.

Yıldırım et al. [18] in their study, have presented an automatic pattern recognition system using signs from subjects who is healthy and had epileptic seizures. They obtained spectral information of EEG signals using the Peridogram and Welch methods. The feature vectors they obtained were classified with k nearest neighbor algorithm (k-NN), support vector machine (SVM), extreme learning machine (ELM). They reported that they achieved the best performance with SVM.

Sood et al. [19] used nonlinear features to classify EEG signals and epileptic seizures. They achieved the highest success in the MLPNN classifier with 98.4% in their study.

In this study, to classify epileptic EEG signals obtained in different conditions and from different regions of the brain, extreme learning machine (ELM) that has the advantages of fast learning speed and good generalization performance was used. In this study, it was aimed to detect epileptic attacks by using EEG signals obtained from healthy individuals and individuals having epileptic attacks.

## 2. Materials and Methods

In this study, ELM method which provides generalizable performance and does not need parameters according to classical ANN algorithms is used. The proposed method for epileptic seizure prediction consists of five steps. Firstly, healthy and ictal data sets were obtained from the UCI database over the EEG dataset. Segments were formed with 50% overlap on the data set.



Then, to obtain the attributes to be used in the classification, 4th level wavelet coefficients were determined with Discrete Wavelet Transform (DWT). The classification process was performed by using the obtained properties were used as input features for the ELM classification algorithm. Finally, according to the classification results, the decision-making process has been carried out. The flow diagram, which generally describes these steps, is shown in Figure 2.



Figure 2. General flow diagram of the proposed method

## 2.1. EEG Dataset

In this study, the EEG dataset published by Andrzejak et al. [14] was used. The data set contains 100 channels of EEG data sampled at a frequency of 173.61 Hz and divided into 5 different classes: Set A, Set B, Set C, Set D and Set E. Each cluster consists of 100 single-channel EEG data with 4096 samples. In our study, only two of the five different data sets (Set A and Set E) were used. Normal data (Set A); were obtained from five different people who healthy and different ages. Patient (Set E) data were obtained from five different people with epilepsy and different ages [14, 20]. Example signs for clusters used in this study are shown in Figure 3.



Figure 3. 10-second samples from sets A and E

The signals have transferred to a computer environment after being converted by a 12-bit analog to digital converter. Since the epileptic properties manifest themselves in frequency bands below 30-40 Hz, 0.53-40 Hz bandpass filter is applied to the signals whose spectral range is 0.5-85 Hz [14, 21].





Wavelet transform; is a transformation technique that separates data into different frequency components and examines each component with its resolution at that scale. The wavelet transform of a signal, which is a function of time, depends on the frequency and time variables [22]. EEG signals are type of the non-stationary signals. In this study, EEG signals are separated by using DWT according to frequency component and the characteristics of frequency bands are investigated. Since DWT uses a small sized window for high frequencies and a large sized window for low frequencies, it tries to provide optimal resolution in terms of time and frequency [23].

The International Conference

With DWT, the signal is passed through the high-pass filter to analyze the high frequencies in the input signal. The signal is passed through the low pass filter to analyze the low frequencies. As shown in Figure 4, the input signal is indicated by x[n]. Here n is an integer. The low pass filter is indicated by g, while the high pass filter is indicated by h [23, 24].



### 2.3. Extreme Learning Machine (ELM)

ELM is an effective learning algorithm proposed by Huang et al. for single hidden layer feedforward neural networks [1]. As shown in Figure 5, the ELM model consists of an input layer, a hidden layer and an output layer.



Figure 5. Model structure of extreme learning machine [26]

The output value  $y_j \in \mathbb{R}^n$  of the ELM network is calculated according to N training samples consisting of  $(x_i, t_i) \in \mathbb{R}^n \times \mathbb{R}^m$   $i = 1, 2, \dots, N$ . Here  $x_1, x_2, \dots, x_t$  refers to the property vectors given to the input of the system and  $y_i$  refers to the decision vector. The mathematical model of

Technology

aterials and Engineering

single hidden layer feedforward neural networks which has *L* number of neurons in its hidden layer is as in Equation (1) [1].

International Conference

$$\sum_{i=1}^{L} \beta_i g(w_i \cdot x_j + b_i) = O_j j = 1 \dots N$$
(1)

Here  $w_i = [\omega_{i1}, \omega_{i2}, ..., \omega_{in}]$  indicates the input layer weight sequence,  $\beta_i = [\beta_{i1}, \beta_{i2}, ..., \beta_{im}]$ indicates the output weight sequence connected to the hided nerve cell and the exit nerve cells,  $b_i$ indicates the threshold values of hidden layer neurons and  $O_j$  indicates output vector. g(.)represents the activation function.

In order to maximize the performance of this SLFNs network structure, which is formed in its most basic form, it is accepted that the error could approached the "zero" error value. That is, the desired output and the given output relationship can be expressed as  $\sum_{j=1}^{L} ||o_j - y_j|| = 0$ . The ELM can be expressed in Equation (2) in a more general expression.

$$\mathbf{H}\boldsymbol{\beta} = \mathbf{Y} \tag{2}$$

H represents the hidden layer output values in this equation and can be expressed in matrix form as shown in Equation (3).

$$\mathbf{H} = \begin{bmatrix} g(\mathbf{w}_1, \mathbf{x}_1 + b_1) & \cdots & g(\mathbf{w}_L, \mathbf{x}_1 + b_L) \\ \cdots & \cdots & \cdots \\ g(\mathbf{w}_1, \mathbf{x}_N + b_1) & \cdots & g(\mathbf{w}_L, \mathbf{x}_N + b_L) \end{bmatrix}_{N \times L}$$
(3)

The  $\beta$  in Equation (4) represents the weight values between the hidden layer and the output layer, and *Y* represents the output vectors.

$$\beta = \begin{bmatrix} \beta_1^T \\ \vdots \\ \beta_L^T \end{bmatrix}_{Lxm} \quad \text{ve } \mathbf{Y} = \begin{bmatrix} \mathbf{y}_1^T \\ \vdots \\ \mathbf{y}_N^T \end{bmatrix}_{Nxm} \tag{4}$$

According to Moore-Penrose generalized matrix inverse theorem, the representation of a non-square matrix of  $n \times m$  dimensions of the system given in Equation (2) which provides the smallest norm and least squares solution is as in Equation (5) [1, 3, 27].

$$\boldsymbol{\beta} = \mathbf{H}^{\dagger} \mathbf{Y} \tag{5}$$

Here  $\mathbf{H}^{\dagger}$  represents the generalized inverse matrix of the output matrix.

### 3. Results

In this study, it is aimed to classify two different EEG types, which belong to healthy individuals and individuals who had epileptic attacks, with ELM. After obtaining individuals' EEG signals, who are healthy and had epileptic attacks, from raw data, it divided into 5-second segments. As

917



given in Table 1, the EEG signals are subdivided into the detailed sub-bands D1-D4 and finally the A4 sub-band. For the calculation of wavelet coefficients for each segment, 4th level WDT was applied using 2nd order Daubechies (db2) wavelet.

Sub-bands	Frequency range (Hz)
D1	43.4-86.8
D2	21.7-43.4
D3	10.8-21.7
D4	5.4-10.8
A4	0-5.4

Table 1. Ranges	of frequency	bands in	wavelet	decomposition.
I abre It Itanges	or mequency	oundo m		

Statistical properties were applied on the set of wavelet coefficients to reduce the size of the feature vectors obtained after the WDT transformation. Statistical properties including the maximum, minimum, mean and standard deviation of wavelet coefficients in each subband were used to represent the time-frequency distribution of the EEG signals. In total, 20 properties were obtained for each segment.

The classification process performed over the obtained properties by using ELM method. The parameters of the ELM network used are given in Table 2.

<b>Table 2.</b> The learning parameters of ELM network.				
Number of Input Layer Neurons	20			
Hidden Layer	1			
Number of Hidden Layer Neurons	5200			
Output Layer	1			
Applied Activation Functions	Hard limit sigmoid, sin, Radial basis, Triangular basis			
Learning Algorithm	ELM for single layer feed-forward network			

The performance of the ELM network is influenced by the number of neurons in the latent layer and the activation function to be used. Therefore, trials have been performed on different neuron numbers and activation functions. The number of neurons in the hidden layer was obtained by performing trials by increasing one by one, between 5-200 (Figure 6). In order to see the effect of activation function changes, Hard limit sigmoid, sin, Radial basis, Triangular basis activation functions were applied to ELM network. The optimal activation function and the number of neurons were determined according to the training and test performance of the network.



Figure 6. Effect of neuron number change on ELM performance

In this study, the best performance value of the ELM network was obtained in the network structure which has 82 neurons in the hidden layer and has sinus activation function. In the study, 99.67% classification accuracy was obtained. The comparison of this study, which made to classify A and E clusters on the EEG dataset used, with similar studies in the literature is given in Table 3.

Method	Classifier	Training/test	Acc.(%)
Aarabi et al. [28]	BNN	Hold-out (50.00–50.00%)	93.00
Subasi [10]	ME	Hold-out (62.50–37.50%)	94.50
Yuan et al. [29]	ELM	Hold-out (50.00–50.00%)	96.50
Khan et al. [30]	LDA	Hold-out (80.00–20.00%)	91.80
Kumar and Kolekar [31]	SVM	Hold-out (66.67–33.33%)	97.50
Hussein et al. [32]	ESD-LSTM	Hold-out (66.67–33.33%)	100.00
Proposed method	ELM	Hold-out (75.00-25.00%)	99.67

Table 3. Seizure detection results of recommended and other methods: For Classes A-E.

With this study, it is seen that reached the result which can be considered as fast and effective in the diagnosis of epileptic attacks by using the WDT and ELM algorithm in the classification. It is thought the proposed method may be a useful tool for decision-making stage in medical diagnostic systems. Furthermore, the obtained method can be further developed with different EEG clusters and different classification techniques.

### References

1. G.-B. Bin Huang et al., "Extreme learning machine: Theory and applications," Neurocomputing, vol. 70, no. 1–3. Elsevier, pp. 489–501, 01-Dec-2006.

The International Conference of Materials and Engineering Technology

- **2.** G. Huang, G.-B. Huang, S. Song, and K. You, "Trends in extreme learning machines: A review," Neural Networks, vol. 61, pp. 32–48, Jan. 2015.
- **3.** F. Ucar, B. Dandil, and F. Ata, "Classification of power quality events using extreme learning machine," in 2015 23nd Signal Processing and Communications Applications Conference (SIU), 2015, pp. 970–973.
- **4.** O. F. Ertugrul, M. E. Tagluk, Y. Kaya, and R. Tekin, "EMG signal classification by extreme learning machine," in 2013 21st Signal Processing and Communications Applications Conference (SIU), 2013, pp. 1–4.
- 5. Guang-Bin Huang, Hongming Zhou, Xiaojian Ding, and Rui Zhang, "Extreme Learning Machine for Regression and Multiclass Classification," IEEE Trans. Syst. Man, Cybern. Part B, vol. 42, no. 2, pp. 513–529, Apr. 2012.
- **6.** Z. Comert and A. F. Kocamaz, "Cardiotocography analysis based on segmentation-based fractal texture decomposition and extreme learning machine," in 2017 25th Signal Processing and Communications Applications Conference (SIU), 2017, pp. 1–4.
- 7. S. Ding, X. Xu, and R. Nie, "Extreme learning machine and its applications," Neural Comput. Appl., vol. 25, no. 3–4, pp. 549–556, Sep. 2014.
- 8. S. Ding, N. Zhang, X. Xu, L. Guo, and J. Zhang, "Deep Extreme Learning Machine and Its Application in EEG Classification," Math. Probl. Eng., vol. 2015, pp. 1–11, May 2015.
- 9. H. Adeli, Z. Zhou, and N. Dadmehr, "Analysis of EEG records in an epileptic patient using wavelet transform," J. Neurosci. Methods, vol. 123, no. 1, pp. 69–87, 2003.
- **10.** A. Subasi, "EEG signal classification using wavelet feature extraction and a mixture of expert model," Expert Syst. Appl., vol. 32, no. 4, pp. 1084–1093, 2007.
- A. B. Gardner, A. M. Krieger, G. Vachtsevanos, and B. Litt, "One-class novelty detection for seizure analysis from intracranial EEG," J. Mach. Learn. Res., vol. 7, no. Jun, pp. 1025– 1044, 2006.
- **12.** S. Noachtar and J. Rémi, "The role of EEG in epilepsy: a critical review," Epilepsy Behav., vol. 15, no. 1, pp. 22–33, 2009.
- **13.** G. M. Rojas, C. Alvarez, C. E. Montoya, M. de la Iglesia-Vayá, J. E. Cisternas, and M. Gálvez, "Study of resting-state functional connectivity networks using EEG electrodes position as seed," Front. Neurosci., vol. 12, p. 235, 2018.
- 14. R. G. Andrzejak, K. Lehnertz, F. Mormann, C. Rieke, P. David, and C. E. Elger, "Indications of nonlinear deterministic and finite-dimensional structures in time series of brain electrical activity: Dependence on recording region and brain state," Phys. Rev. E, vol. 64, no. 6, p. 61907, 2001.
- **15.** A. T. Tzallas et al., "Automated epileptic seizure detection methods: a review study," in Epilepsy-histological, electroencephalographic and psychological aspects, IntechOpen, 2012.
- 16. K. Lehnertz et al., "Seizure prediction by nonlinear EEG analysis," IEEE Eng. Med. Biol. Mag., vol. 22, no. 1, pp. 57–63, 2003.
- **17.** A. T. Tzallas, M. G. Tsipouras, and D. I. Fotiadis, "Automatic seizure detection based on time-frequency analysis and artificial neural networks," Comput. Intell. Neurosci., vol. 2007, 2007.
- **18.** M. Yildirim and A. Yildiz, "Farklı zaman ölçekli EEG işaretlerinden epilepsi nöbetinin otomatik tespiti," DÜMF Mühendislik Derg., vol. 8, no. 4, pp. 745–757.

19. M. Sood and S. V Bhooshan, "Prognosis of epileptic seizures using EEG signals," in 2015 Third International Conference on Image Information Processing (ICIIP), 2015, pp. 12– 16.

The International Conference of Materials and Engineering Technology

- 20. İ. Güler and E. D. Übeyli, "Adaptive neuro-fuzzy inference system for classification of EEG signals using wavelet coefficients," J. Neurosci. Methods, vol. 148, no. 2, pp. 113– 121, Oct. 2005.
- **21.** E. C. Andrzejak RG, Lehnertz K, Rieke C, Mormann F, David P, "EEG time series download page." [Online]. Available: http://epileptologie-bonn.de/cms/front\_content.php?idcat=193&lang=3&changelang=3.
- **22.** S. Kara, F. Dirgenali, and Ş. Okkesim, "Estimating Gastric Rhythm Differences Using a Wavelet Method from the Electrogastrograms of Normal and Diabetic Subjects," Instrum. Sci. Technol., vol. 33, no. 5, pp. 519–532, Sep. 2005.
- 23. L. Debnath and F. A. Shah, Lecture notes on wavelet transforms. Springer, 2017.
- **24.** M. Coşkun and A. İstanbullu, "EEG İşaretlerinin FFT ve dalgacık dönüşümü ile analizi," XIV. Akad. Bilişim Konf., pp. 1–3, 2012.
- **25.** Y. Seo, Y. Choi, and J. Choi, "River stage modeling by combining maximal overlap discrete wavelet transform, support vector machines and genetic algorithm," Water (Switzerland), 2017.
- **26.** Y. Li, S. Zhang, Y. Yin, W. Xiao, and J. Zhang, "A novel online sequential extreme learning machine for gas utilization ratio prediction in blast furnaces," Sensors (Switzerland), 2017.
- 27. Z. M. Yaseen, S. O. Sulaiman, R. C. Deo, and K.-W. Chau, "An enhanced extreme learning machine model for river flow forecasting: State-of-the-art, practical applications in water resource engineering area and future research direction," J. Hydrol., vol. 569, pp. 387–408, Feb. 2019.
- **28.** A. Aarabi, F. Wallois, and R. Grebe, "Automated neonatal seizure detection: A multistage classification system through feature selection based on relevance and redundancy analysis," Clin. Neurophysiol., vol. 117, no. 2, pp. 328–340, 2006.
- **29.** Q. Yuan, W. Zhou, S. Li, and D. Cai, "Epileptic EEG classification based on extreme learning machine and nonlinear features," Epilepsy Res., vol. 96, no. 1–2, pp. 29–38, 2011.
- **30.** Y. Khan, N. Faraz, S. Kumar, and A. Yildirim, "No Title," U. P. B. Sci. Bull. Ser. A, vol. 74, p. 1, 2012.
- **31.** A. Kumar and M. H. Kolekar, "Machine learning approach for epileptic seizure detection using wavelet analysis of EEG signals," in 2014 International Conference on Medical Imaging, m-Health and Emerging Communication Systems, MedCom 2014, 2014, pp. 412–416.
- **32.** R. Hussein, H. Palangi, R. K. Ward, and Z. J. Wang, "Optimized deep neural network architecture for robust detection of epileptic seizures using EEG signals," Clin. Neurophysiol., vol. 130, no. 1, pp. 25–37, 2019.

# DESIGN OF A SIMPLE SQUARE ROOT CALCULATOR ON FPGA BY USING VHDL

The International Conference of Materials and Engineering Technology

### Abdulkadir SADAY<sup>\*1</sup>, Ilker Ali OZKAN<sup>2</sup>

<sup>1</sup> Selcuk University, Faculty of Technology, Konya, TURKEY. <sup>2</sup> Selcuk University, Faculty of Technology, Computer Engineering Department, Konya, TURKEY.

#### Abstract

The square root calculator is an important mathematical calculation process in digital systems and is frequently used in digital signal processing. There are different methods and algorithms for performing the basic square root calculation process in digital systems. The design and performance of the calculator may vary depending on the method and programming format of the designer follows. The digital square root calculator designed in this study is mainly based on the non-restoring calculation algorithm. The proposed design was prepared using the VHDL language on FPGA.

Keywords: FPGA, square root calculator, VHDL

### 1. Introduction

The calculation of the square root of a number can be done with different methods and rules. In digital systems, as in normal mathematics, square root calculation is performed with different methods. In digital signal processing and applications, the computation of the square root of a number requires a more complex processing structure than the normal computing, due to the nature of the digital processors. The need for complex processing leads to high power and memory usage in the calculation process. Therefore, it emerges that the necessity to use algorithms that can compute with low power and memory usage and to design methods. [1, 2].

Square root calculation is a mathematical expression that is needed in many systems. Many methods have been developed to achieve the exact result and it is very difficult to achieve the exact result for the square root calculation. Mainly Newton method, step-by-step method, Babylonian method, rough estimation method, restoring and non-restoring method are used in the calculation of square root. However, the proposed methods cause long delays in calculations due to the intensive processing requirements [3, 4].

Field Programmable Gate Arrays (FPGA) with its advanced structure and ability to perform parallel processing, has a very fast response time in calculation operations. However, the use of a direct hardware language in programming the FPGA results in some process limitations. As with most types of processors, there is no applicable mathematical library on FPGA that processes directly with real numbers and special mathematical expressions. Therefore, each operation must be defined in simple mathematical expressions. The definition of square root acquisition on FPGA is made by methods that give approximate results to the square root value of the number [5].

In the non-restoring calculation algorithm, which is one of the calculation methods, the remainder obtained from the dividing of the number is not added to the calculation again. In this way, more speed and less memory are required in the calculation. The non-restoring algorithm is based on step-by-step calculation, where each step is evaluated separately. With this method, a value closer to the actual square root value of the number can be reached [3, 6].

Construction of complex mathematical operations on the FPGA is very difficult. Complex operations need to be expressed in simple addition, subtraction, multiplication and division operations. The methods developed for this purpose enable complex mathematical operations to be performed easily with FPGA. However, the result obtained by the calculation operation is found close to the actual square root value of the number [5, 7].

In FPGA, the calculation operations are kept in variables with bit values. Therefore, the greater the bit value of the variable in which the number is defined and the variable allocated for the result, the closer the actual value is obtained. However, using the high bit value will increase the amount of space occupied by the variables in direct proportion. For this reason, it is necessary to make the selection according to the desired sensitivity in the programming stage [7, 8].

In this study, it is aimed to design a simple square root calculator that returns fast results by using VHDL language and to obtain the result easily with the non-restoring calculation algorithm.

### 2. Materials and Methods

In this study, a simple square root calculator that returns the square root of integers as a result using the VHDL language on FPGA was designed as a function. In the design, the non-restoring calculation algorithm is used as a basis and the calculator is simulated using this method. The designed calculator is created as a simple module and can be easily integrated into the system when it is needed.

The input and output diagram for the square root calculator with VHDL is given in Figure 1.



Figure 1. Square root calculator diagram.

The calculator is defined by VHDL as a function that returns the result to the main program. The desired number value is assigned as input and the square root value of the calculated number is returned. For this process, each calculation step of the method is designed to take place in one clock pulse of the FPGA. The clock frequency is set depending on the size of the number and the duration of the calculation.

The calculator prepared in the study is designed to have a 16-bit input size. It provides 8-bit output against 16-bit input. The algorithm is a function that non-restoring, based on the portion and remaining value, and returns an integer for the integer input. With the calculator based on step-by-step calculation and digit shifting method used during the calculation process, the result of the

Technology

The International Conference of Materials and Engineering



The International Conference

$$y = sqrt(x)$$

(1)

nd Engineering Technology

The result returned in the function and the input of the function are of the unsigned data type. The sample code snippet of the coding required to run the function in the main program is given in Figure 2.

```
...
signal data_in : unsigned(2n-1 downto 0);
signal data_out : unsigned(n-1 downto 0);
...
data_out <= sqrt(data_in);
...</pre>
```

Figure 2. Example function call.

In the code snippet shown in Figure 2, a 2*n*-bit size number against the adjustable *n* integer value of the function and the *n*-bit size calculated square root of the number are obtained.

# 3. Results

The programming of FPGAs can be done with hardware description languages and hardware level programming. Verilog and VHDL are different programming languages used for programming FPGAs [8]. The square root calculator designed in this study was designed using VHDL programming language. The process steps of the algorithm of the designed calculator are given in Figure 3.

```
Step 1: Assigning input, division and remainder variables
Step 2: Divide the input into groups as two binary numbers
Step 3: Select the group starting from the left
Step 4: Selection of a square and its subtraction
Step 5: Two-bit shift, inference by applying the predictive
frame 01
Step 6: Repeat step 5 until the last pair
Figure 3. Algorithm processing steps.
```

The processing steps of the algorithm are coded in accordance with the VHDL language. The resulting function takes the square root of the given number for the 2n-bit input and produces the result closest to the actual value. The designed calculator is synthesizable and can work on hardware as well as simulation. The synthesized calculator block structure is shown in Figure 4.



The International Conference

Materials and Engineering Technology

Figure 4. Block view of the calculator.

Synthesis is the process that provides the necessary verification for the program or function prepared in FPGA to work on real hardware [5]. It is confirmed that the design prepared by synthesis is able to work not only in simulation environment but also on hardware. The hardware structure of the system's inputs and outputs is given in Figure 5.



Figure 5. System circuit view.

The square root calculator can only process integers depending on the computational capability of the programmed algorithm. Returns the square root of the number as an integer with the nearest value for the integer applied to its input. According to the data obtained from the simulation, it is concluded that the designed calculator is easy to apply on FPGA and it is appropriate to solve the simple square root problem. Some values obtained in the simulation against to inputs given are given in Table 1.

Table 1. Square root calculator simulation values.					
Input (data_in)	16	64	256	32200	12600
Output (data_out)	4	8	16	179	112
Actual value	4	8	16	179,44	112,24

925



According to the results obtained in the study, it is observed that the calculator works correctly and returns the result closest to the actual value in the intermediate values. Working with integer values in the calculator causes the resulting number to get the integer value that is closest to its actual value. The fact that there is a digit shifting in the algorithm makes it difficult to implement this algorithm in fixed point and floating-point data types. However, the function obtained in the study operates at a speed that can return the result in a 10ns clock pulse. This makes it possible to use the square root calculator as a function that can produce fast results in many applications. The simulation screen of the calculator is shown in Figure 6.

Name	Value		1,010 ns	1,020 ns	1,030 ns	1,040 ns	1,050 ns	1,060 ns	1,070 ns
le clk	1								
V V input[15:0]	0010	0010	0040	0100	7dc8	fe01	2710	3138	31e2
14 [15]	0								
[14]	0								
14 [13]	0								
16 [12]	0							1	
la [11]	0				Į.				
16 [10]	0								
16 [9]	0								
16 [8]	0								
16 [7]	0								
6]	0								
16 [5]	0							1	
<b>a</b> [4]	1	1							
<b>a</b> [3]	0							1	
16 [2]	0								
<b>16</b> [1]	0								
16 [0]	0								
> 😻 output[7:0]	04	04	08	10	b3	ft ft	64	70	71
lo output_real	4	4	8	16	179	255	100	112	113

Figure 5. Calculator simulation screenshot.

### References

- Kaur, S., M. Singh, and R. Agarwal, Vhdl implementation of non restoring division algorithm using high speed adder/subtractor. International Journal of Advanced Research in Electrical, Electronics and Instrumentation Engineering, 2013. 2(7): p. 3317-3324.
- Jena, A. and S.K. Panda, FPGA-VHDL implementation of pipelined square root circuit for VLSI signal processing applications. International Journal of Computer Applications, 2016. 975: p. 8887.
- 3. Sutikno, T., et al., *Simplified VHDL coding of modified non-restoring square root calculator*. International Journal of Reconfigurable and Embedded Systems, 2012. **1**(1): p. 37.
- 4. Franke, M., et al., Implementation of different square root algorithms. 2007.
- 5. Sutikno, T., *An efficient implementation of the non restoring square root algorithm in gate level.* International journal of computer theory and engineering, 2011. **3**(1): p. 46.
- 6. Piromsopa, K., C. Aporntewan, and P. Chongsatitvatana. An FPGA implementation of a fixedpoint square root operation. in Proceedings of the International Symposium on Communications and Information Technology. 2001. Citeseer.
- Nanhe, A., et al., Implementation of fixed and floating point square root using nonrestoring algorithm on FPGA. International Journal of Computer and Electrical Engineering, 2013. 5(5): p. 533.
- 8. Kachhwal, P. and B.C. Rout. *Novel square root algorithm and its FPGA implementation*. in 2014 *International Conference on Signal Propagation and Computer Technology (ICSPCT 2014)*. 2014. IEEE.



# STATIC STRUCTURAL ANALYSIS OF A RC HELICOPTER TAIL ROTOR

# BURAK ÇİFTCİOĞLU<sup>\*1</sup>, İBRAHİM GÖV<sup>2</sup>, M.HANİFİ DOĞRU<sup>3</sup>

 <sup>1</sup> Affiliation: Gaziantep University, Faculty of Aeronautics and Aerospace, Aircraft and Aerospace Engineering Department, Gaziantep, TURKEY.
 <sup>2</sup> Affiliation: Gaziantep University, Faculty of Aeronautics and Aerospace, Aircraft and Aerospace Engineering Department, Gaziantep, TURKEY.
 <sup>3</sup> Affiliation Gaziantep University, Faculty of Aeronautics and Aerospace, Pilotage Department, Gaziantep, TURKEY.

#### Abstract

Helicopters are the aircrafts which have rotating wings and ability to take off and land vertically. In addition to lift producing, flight controls about related axis of aircraft and variety of flight maneuvers are also maintained by the aerodynamic effects of that rotating wings which are also known as propellers. These effects can be used for some other applications which also have rotating wings for other purposes different from lift producing. Different ways on different applications are used to alter these aerodynamic effects on propellers or blades, so that desired movement of aircraft's propellers or blades of the systems like wind turbines which needs these alterations, can be obtained. Defined as the angle between the propeller and the rotation plane, pitch is the main variable to alter aerodynamic effects on propellers. In this paper, variable pitch mechanism of a RC Helicopter tail was investigated for structural analysis. Tail rotors provide stability to the rotorcraft and also they are used for rotorcraft's movement around directional axis which also known as yaw movement. Unlike the conventional main rotor pitch mechanism of RC Helicopters which have swashplate, tail rotors have a simple mechanism for pitch control. Behavior of the tail rotor and tail rod under static structural load during hovering condition was analyzed. For these purposes, Static Structural section of the ANSYS was used.

Keyword: Rotorcraft, Pitch Mechanism, Finite Element Analysis, Structural Analysis.

### **1. Introduction**

Helicopters are the devices which have ability to flight in 6 degrees of freedom. Because of their inherently unstable flight characteristic different kind of applications are used to obtain a stable flight. Main rotor rotation brings about high amount of torque values on the fuselage of the rotorcraft. Tail rotors are used to compensate this torque reaction by creating an equivalent amount of anti-torque force. Beside of this major duty, they also provide directional control of rotorcraft about the yaw axis and their aerodynamics maintain weathercock stability which can be referred as the yawing moment caused by the side slip [1]. To maintain these various duties, alterations needs to be applied to the blade pitch angle which is defined as the angle between chord line of the blade and plane of the rotation. For the conventional normal size helicopters there are two pedals for pilot to control the collective pitch of tail rotor so that the desired heading value of the rotorcraft, or the value required to compensate the torque caused by main rotor can be obtained [2].

CMET'

The International Conference of Materials and Engineering Technology

Unlike fixed wing aircrafts, rotorcrafts have various complex loads acting on different kind of components during their normal flight regimes. A review of the different kind of loads exerted on the helicopters and their related components and some early estimation techniques have been previously gathered up [3, 4]. Rotor system types are one of the key features for load distribution because different kind of loads can be distributed to variety of components. For example, while rigid rotor systems cannot lead lag and flap and absorb these loads with blade bending, fully articulated rotor systems' blades can lead lag with their own hinges and flap independently from each other[5]. However, for this paper's study case of RC Helicopters there is a simplified rigid rotor system which has variable pitch mechanism actuated with servo motor is used.

Finite element analysis (FEA) method is a powerful technique for solving the real life problems which have complex domains and different kind of boundary conditions. Its ability to approximate the solutions of these problems has put forward this method as an important design tool for varying engineering applications so that designers can predict the critical points and after that compare with experimental results. Different kind of analysis for different rotating wing vehicle components had been carried out. Behavior of the chassis of a helicopter which is designed by the University Teknologi Malaysia investigated with FEA and had been compared with the experimental tests [6]. Kinematic and strength analysis of another application of pitch changing mechanism involved in wind turbines studied with separate strength analysis of separate critical parts[7]. On another study the helicopter main rotor blade which has airfoil shape represented by class function/shape function transformation (CST) coefficients had been investigated under static structure analysis[8]. Also there are some different types of frame analyses available for different rotating wing aircrafts like quadcopters and newly designed concepts.

### 2. Materials and Methods

Before getting into structural analysis in ANSYS, concepts which lying behind the software's working principle needs to be understood for correct evaluation of the results gained from software's own solution. Finite element method procedure for structural analysis consists of these progresses:

- Dividing of structure to elements
- Understanding of behavior of these elements' physical specifications



- Assembling these elements with nodes to gain structure as a whole body and get a system of equations
- Solving of these equations and unknown values located at nodes
- Calculating quantities to evaluate at boundaries and other specified elements[9]

### 2.1 3D Solid Mechanics

For a better understanding of how ANSYS works, basic governing equations for 3D bodies should be defined. For these purposes, let us consider an infinitesimal body which is located inside a three dimensional body to be analyzed. These infinitesimal body has normal and shear stresses over related axes as shown on Figure 1.



Figure 1. Normal and Shear Stresses

On this infinitesimal body  $\Delta x$ ,  $\Delta y$  and  $\Delta z$  values needs to be considered as limits tend to zero. And normal force on a surface can be written as:

$$\Delta x \to 0, \, \Delta y \to 0, \, \Delta z \to 0$$

$$F_x = \sigma_x \, \Delta y \, \Delta z$$

$$F_y = \sigma_y \, \Delta x \, \Delta z$$

$$F_z = \sigma_z \, \Delta x \, \Delta y$$
(1)



And due to rotational equilibrium,  $\tau_{xy} = \tau_{yx}$  and this equilibrium can be applied to all other shear stresses. Considering as two dimensional case of one surface, one can write the force equilibrium equations with Taylor series expansion for related direction, then can derive the remaining equations of three dimensional case with contribution of additional shear stress change. It should become as:

$$\frac{\partial \sigma_x}{\partial x} + \frac{\partial \tau_{xy}}{\partial y} + \frac{\partial \tau_{xz}}{\partial z} + f_x = 0$$

$$\frac{\partial \sigma_y}{\partial y} + \frac{\partial \tau_{yx}}{\partial x} + \frac{\partial \tau_{yz}}{\partial z} + f_y = 0$$
(2)

 $\frac{\partial \sigma_z}{\partial z} + \frac{\partial \tau_{zx}}{\partial x} + \frac{\partial \tau_{zy}}{\partial y} + f_z = 0$ 

Where,  $f_x$ ,  $f_y$ ,  $f_z$  is body forces for related directions which can be gravitational force. There is 3 equations with 6 unknown functions. Additional equations can be added from the Hooke's Law to these derived equations known as 3D Elasticity. For an isotropic material these additional equations which are involving normal strain and shear strain due to related stresses can be written in the elastic range as:

$$\begin{cases} \sigma_{x} \\ \sigma_{y} \\ \sigma_{z} \\ \tau_{xy} \\ \tau_{yz} \\ \tau_{zx} \end{cases} = \frac{E}{(1+\nu)(1-2\nu)} \begin{bmatrix} 1-\nu & \nu & \nu & 0 & 0 & 0 \\ \nu & 1-\nu & \nu & 0 & 0 & 0 \\ \nu & \nu & 1-\nu & 0 & 0 & 0 \\ 0 & 0 & 0 & \frac{1-2\nu}{2} & 0 & 0 \\ 0 & 0 & 0 & 0 & \frac{1-2\nu}{2} & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1-2\nu}{2} \end{bmatrix} \begin{bmatrix} \varepsilon_{x} \\ \varepsilon_{y} \\ \varepsilon_{z} \\ \gamma_{xy} \\ \gamma_{yz} \\ \gamma_{zx} \end{bmatrix}$$
(3)

This matrix represents the strain-strain relationship in stiffness form where E and v given as elastic modulus and Poisson ratio, respectively and they are defined in ANSYS as material properties in Engineering Data tab. Stiffness matrix gives additional 6 equations with 6 unknowns. Finally, strain-displacement relation equations for normal and shear strains should be added to governing equations to obtain the final form of set of equations.



Considering there are small strains for both normal and shear strains and  $\Delta u$ ,  $\Delta v$ ,  $\Delta w$  as the displacements over *x*, *y*, *z* axes of lines with initial lengths of  $\Delta x \Delta y \Delta z$ , as they tend to zero, one can write the normal strain equations with the Taylor series expansion as:

$$\varepsilon_{x} = \frac{\Delta u}{\Delta x} \quad \varepsilon_{y} = \frac{\Delta v}{\Delta y} \quad \varepsilon_{z} = \frac{\Delta w}{\Delta z}$$
(4)  

$$\varepsilon_{x} = \frac{\partial u}{\partial x} \qquad \Delta x \to 0$$
(5)  

$$\varepsilon_{y} = \frac{\partial v}{\partial y} \qquad \Delta y \to 0$$
  

$$\varepsilon_{z} = \frac{\partial w}{\partial z} \qquad \Delta z \to 0$$

Similarly for shear strains which involve changes in angle can be defined as:

$\gamma_{xy} =$	$\frac{\partial u}{\partial y} + \frac{\partial v}{\partial x}$	(6)
$\gamma_{yz} =$	$\frac{\partial v}{\partial z} + \frac{\partial w}{\partial y}$	
$\gamma_{zx} =$	$\frac{\partial w}{\partial x} + \frac{\partial u}{\partial z}$	

As a summary, there are six equations from force balance, six equations from three dimensional Hooke's Law, and six equations from strain-displacement relations with fifteen unknown fields which involve stress, strain and displacement components. It is vital to understand these concepts for accurate evaluation of the related data given and taken from software.

### 2.2 Method of Analysis

Among of the different kind of commercial RC helicopter models, Align Trex450 series with belt driven tail rotor selected for analyze. Tail rotor section was modelled with Solidworks 2016. Static Structural section of ANSYS Workbench 19 used for analysis. To avoid the increasing solution time and complex contacting body types, small dimensioned fasteners on the geometry were simplified on Solidworks as solid bodies and all remaining contacts had been set as bonding contacts to prevent nonlinearity issues due to contact conditions on ANSYS[10].

Model was considered in the scope of the hovering flight with ideal conditions and carried out linear section of the stress-strain curve. One of the most critical fatigue regimes a tail rotor faces is when helicopter is in hovering condition because on that condition tail rotor providing all or most required force to balance the torque of the main rotor. On a hovering tail rotor, loading can be considered as essentially static. This should be applicable for a rotor with perfectly isolated and balanced blades and eliminated wind conditions.[3] Meaning of the ideal condition contributes these assumptions and static structural analysis could be carried out with them.



Geometry is consisting of two main parts which are tail boom and rotor assembly. Rotor assembly has attached propellers, pitch changing mechanism and shaft with pulley attached to it as shown in Figure 2.



Figure 2. Geometry of tail

Materials had been selected from the engineering data. Tail pitch control yoke arm selected as polyethylene and both propellers selected as graphite epoxy. All other parts defined as aluminum alloy from library. Table 1 shows the related information about materials. Since composite material behavior varies with directions, XY, YZ, ZX values shows these directional strength properties respectively.

Table 1. Material Properties					
Matarial	Young's	<b>Poisson Ratio</b>	<b>Tensile Yield</b>		
Material	Modulus (GPa)	(v)	Strength (MPa)		
Polyethylene	1,1	0.42	25		
Aluminum Alloy	71	0,33	280		
Epoxy Carbon Woven	61.34, 61.34, 6.9	0.04, 0.3, 0.3	805, 805, 50		

Boundary condition set as fixed support and located as the surface of the boom where it actually connects to the main frame of RC helicopter via a boom mount. Lift force acting on the pressure surface of the blades determined as 2.5 N per blade for all cases.





Figure 3. Force acting on pressure surface

Optimum element type selected as Hex Dominant which is comprised of hexahedral elements for solid body because of hexahedral elements' ability to fill a given volume better and skill of providing more degrees of freedom to elements with a relatively uniform meshing quality. Element quality has been reviewed from the mesh metric. Different kind of element sizes had been used for different cases to obtain convergence of analysis and mesh independency.

## 2.3 Analysis

Element size of 0.5 mm selected for the analysis because of higher quality values for selected mesh method. Figure 4 shows that hexahedron shaped elements' qualities and numbers are dominant over tetrahedrons and other shape types. Desired element distribution and fine meshing required for correct analysis and 220339 elements and 927791 nodes had been obtained.





After the complete solution and evaluation of results, Total Deformation and Equivalent Stress values investigated for both rotor mechanism and the boom. Maximum deformation occurred on the tip of the blades as expected which were subjected to distributed forces. Figure 5 also shows the undeformed model with auto scale.







Equivalent von Misses stress values can be seen from the Figure 6. Highest values occurred on the root section of the blades where it attached to the blade holders. Blades made from the carbon fiber reinforced polymer composite material highly used for RC helicopters along with the different CFRP components such as vertical fins, can easily handle this stress values with higher yield strength values.



Figure 6. Equivalent von Misses Stress distribution of entire model

Also stress and deformation values for boom obtained. Detailed explanation of this intention discussed comprehensively in Results and Discussion chapter.



Figure 7. Deformation occurred on the boom



It can be seen from the Figure 7, maximum deformation of the boom occurred where it connected to tail rotor mechanism and, Figure 8 shows that maximum stress occurred near the fixed support. There were some stress concentrations occurred due to geometry of the related part.



Figure 8. Equivalent von Misses Stress distribution of boom

### 3. Results and Discussion

To gain a good point of view and evaluate the results for accuracy, some equations can be applied for boom. Because of the symmetry of geometry and acting loads, a two dimensional approach applied. Maximum stress value and deformation of the boom which can be accepted as a cantilever beam, had been found by hand calculations. For this purpose, bending moment acting on the tubular, circular cross sectioned member calculated. Total inertia of the given area, *I*, obtained and then maximum stress and deformation values was found for aluminum circular cantilever beam. Inner and outer diameter of the boom is 12 mm and 10.5 mm, respectively.

$$\sigma_m = \frac{Mc}{I} \qquad \delta = \frac{FL^3}{3EI} \tag{7}$$

$$M = F L \tag{8}$$

M = 5,4 N (0,245 m) = 1.325 N . m

$$c = \frac{12 \ mm}{2} = 6 \ mm$$

$$I = \frac{\pi}{4} \ (r_0^4 - r_I^4) = 421.26 \ \times \ 10^{-12} \ m^4$$

$$\sigma_m = 18.87 \ MPa \quad \delta = 0.89 \ mm$$
(9)



Where, F is the force acting on a single point which is located at the tip of the boom, L is the horizontal distance between F and the fixed support, c is the distance between top point of the boom and its neutral axis, and E is the elastic modulus for aluminum alloy which was chosen as 71 GPa. These values can be used for validation of our analysis.

Once Figure 8 be investigated, it can be said that the stress value obtained from the tip point, where the maximum bending moment should be occurred is about 19 MPa. It is an acceptable result compared with hand calculations. Stress concentrations can be associated with the sharp geometry on the fixed support point and maximum value of stress higher on that region. Deformation values comparison with analysis also support the results.

Also maximum deformation of boom occurred where it connected to rotor mechanism as expected with a value of 0.92 mm. 0.03 mm of error occurred which can be associated with the numerical analysis error. These results are comprised the validation of analysis with the hand calculations.

## 4. Conclusions

Structural analysis for a commercially popular RC helicopter tail rotor had been investigated to gain knowledge about critical points and review stress and deformation distributions for a scaled model. After the meshing process, related boundary conditions applied to the model. Validation had been applied with the hand calculations. Results taken from the analysis shows that for such a small scaled system with simple pitch changing mechanism and components, material selection can be done easily because of the relatively small loads and deformations. Different type of materials which can provide weight reduction can be used in different components but related fatigue life investigations should be performed. Dynamic and vibrational analysis can be done along with the structural analysis for better understanding of behavior of the related model. These studies can be seized upon a future work.

## **References:**

- 1. Leishman, G.J., *Principles of helicopter aerodynamics with CD extra*. 2006: Cambridge university press.
- 2. Seddon, J. and S. Newman, *Basic helicopter aerodynamics*. 2001: American Institute of Aeronautics and Astronautics.
- 3. Lombardo, D., *Helicopter structures-a review of loads, fatigue design techniques and usage monitoring.* 1993, DEPARTMENT OF DEFENCE CANBERRA (AUSTRALIA).
- 4. Polanco, F.G., *Estimation of structural component loads in helicopters: a review of current methodologies*. 1999, DEFENCE SCIENCE AND TECHNOLOGY ORGANISATION MELBOURNE (AUSTRALIA).
- 5. Flight Standards Service, *Helicopter Flying Handbook*. U.S. Department of Transportation , FEDERAL AVIATION ADMINISTRATION, 2012.
- 6. Ammoo, M.S., Z.A. Awal, and K.S. Khairudin, *Structural Analysis of UTM Single-Seat Helicopter Chassis*. Jurnal Teknologi, 2014. **71**(2).



- 7. Zhang, S.H., K. Jia, and P.M. Zhang. *Kinematics and Strength Analysis on Variable Propeller Pitch Mechanism of a Wind Turbine Generators*. in *Applied Mechanics and Materials*. 2011. Trans Tech Publ.
- 8. Reddy, K.P., *Static Structural Analysis of a Helicopter Rotor Blade*. International Journal of Novel Research in Electrical and Mechanical Engineering, 2015. **2**(3): p. 83-88.
- 9. Liu, G.-R. and S.S. Quek, *The finite element method: a practical course*. The Finite Element Method: A Practical Course. 2013: Butterworth-Heinemann.
- 10. Madenci, E. and I. Guven, *The finite element method and applications in engineering using ANSYS*<sup>®</sup>. 2015: Springer.





**Günmak Makine**; 1975 yılında ilkokul 3. Sınıftan terk Sıddık KAYABAŞ tarafından 75 m2 bir alanda temelleri atılan, zaman içerisinde kendini bilgi ve tecrübe olarak geliştirmenin yanında, alan ve ekipman olarak ta büyüme sağlayan şu anda 4. Organize sanayi bölgesinde 25.000 m2 bir kurulu alanda, makine ve helezon üretimi yapmaktadır. Ürün yelpazesi oldukça geniş olan GÜNMAK MAKİNE, başlıca üretimi; Anahtar teslimi Bulgur tesisleri, kırmızı mercimek tesisleri, bakliyat çeşitlerinin(arpa, yulaf, bakla, mısır, kinoa, sorgum, bezelye, nohut, pirinç vb.) her türlü makinaları(temizleme, kabuk soyma, taş ayırma, yıkama, eleme ve sınıflandırma vb.) mısır çerezi üretim makinaları, Antep fıstığı kurutma ve taşıma sistemleridir. Ayrıca plastik geri dönüşüm tesisleri, beton santralleri , asfalt plantleri, yağ fabrikaları, bakliyat üretim tesisleri, maden ocakları gibi bir çok iş kolu için ise değişik çap ve modellerde helezon üretimi yapmaktadır. Bugün itibarı ile 2 ülkeye ihracat geçekleştirmekte, ihracat ağını genişletmek için yoğun dış ticaret faaliyetlerinde bulunmaktadır.

Sürekli ve köklü değişim fırtınaları her alanda tüm sektörleri vuruyor; işletmeler, bireyler, meslekler ve kariyerler savruluyor. Başlarını kuma gömüp fırtınaların dinmesini beklemek, eskiden öğrenilenler, geleneksel yaklaşımlar yeni durumla başa çıkmaya yetmiyor. Öğrenilmiş savunma mekanizmaları, durumu kötüleştirmekten başka bir işe yaramıyor. Firmalar yenilik için ARGE merkezleri kurarak değişim rüzgarına karşı en önemli adımları atıyorlar.

Günmak değişimin gerekliliğini, yenilikçiliğin olmadığı firmaların yarınlara yelken açamayacağının bilincindedir. Bu kapsamda sürekli kendi ile yarışan yeni ürünler geliştiren ve üretenlerin başında gelmektedir. 1990 lı yıllardan beri Üniversite Sanayi işbirliği ile AR-GE bilincini başlatan, personelini bu bilinç ile yetiştiren firma, Tübitak'tan 4 projede 7 Adet AR-GE yaparak gıda dalında en fazla proje yapan firma olmuştur. 2017 yılında yapmış olduğu Bakliyat, hububat pişirme ve kurutma ve Antep fıstığı kurutma ünitesi ile, üretim kapasitesinde ve enerji tasarrufunda çok büyük ilerleme kaydederek patent başvurusu yapmış ve Türk Patent Kurumundan belgeleri almaya hak kazanmıştır. Yöresel bir ürün olan hububat ve Antep fıstığının hijyenik şartlarda el değmeden üretilebilmesi maksadı ile geliştirilen bu üniteler Avrupa gıda normlarına ve CE standartlarına uygun yüksek kapasiteli, istenilen nem oranında kurutma imkanı sağlamaktadır. Düşün enerji tüketimi, uygun kurutma oranları ile müşterilerine , geniş rekabet ortamında büyük avantaj sağlamıştır. GÜNMAK bundan sonra da aynı düşünceler ile uluslararası firma olma yolunda, Güzel ülkemizin istihdamına ihracatına ve gelişimine katkı sağlamak üzere tüm enerjisi ile yoluna devam etmektedir.





Gazi Haksan 1994 yılında Kadir FIRAT tarafından CNC dik işlem makine,CNC kayar otomat,CNC c eksen sürücülü makineleri ile yedek parça imalatına başlamıştır.

2000 yılında metal enjeksiyon, plastik enjeksiyon kendi kalıplarını üreterek, asansör ve çelik para kasaları yedek parça imalatı ile Gaziantep sanayisine katkıda bulunarak Türkiye'nin bir çok yerinde çeşitli sektörler ile çalışarak hizmet vermektedir. 1000 metrekare arazi üzerindeki fabrikada Türkiye'nin birçok bölgesindeki şehirlere uzanan hizmet ağı ile ülke ekonomisine katkıda bulunmaya devam etmiştir.



**YÜCE TEKNİK**; Firmamız 2007 Yılında kurulmuş, kuruluşundan Bu Yana Edindiği Ticari Tecrübesini Yenilikçi Yapısı Ve Zengin Ürün Yelpazesiyle Birleştirerek, Metal Sektöründe Faaliyet Gösteren Firmalara Tedarikçi Konumuyla Perakende ve Toptan satış olarak Hizmet Vermektedir.

Teknik hırdavat ve kalıp Elemanları ürünleri üzerine kurulan firmamız 2017 yılından itibaren kendi markası (HERO, WINDBREAKER,YTH) adı altında ithalat ve ihracat yapmaya başlamıştır.

Yüce Teknik, metal sektöründe müşteri odaklılık prensibi üzerinde çalışan, sektörünü iyi tanıyan, paydaşlarının beklentilerini bilen ve yeni beklentiler tanımlayıp, bunların en üst düzeyde tatminini hedefleyen, yaratıcı insan gücüne sahip ve bu doğrultuda tedarik sürecinde en iyi hizmeti veren, güvenilir, Teknik Hırdavat ve Kalıp elemanlar değer zinciri alanlarında verimli ve kârlı hizmet sunmayı hedefleyen bir Teknik Hırdavat şirketidir.





Anadolu Mühendislik 1988 yılında demir & çelik fabrikalarına refrakter malzeme üretimi için Gaziantep'te kurulmuştur. Kısa sürede geniş bir müşteri portföyü edinen firmamız, ilerleyen yıllar içerisinde ithalat ve ihracata ağırlık vermiş, Avrupa, Asya, Afrika ve Ortadoğu ülkeleri gibi çok geniş bir coğrafya ile ticari ilişkiler kurmuş çok çeşitli refrakter ürünleri ithal ve ihraç etmektedir.

# Firmamızın üretmiş olduğu ürünler:

- ✓ Pota sürgü plakaları ve Reftakterleri
- 🗸 Tandiş Zirkonya Nozulları
- ✓ Şekilli Refrakterler
- ✓ Çelik Könvertörler için Curuf kesme dartı
- 🖌 Pota nozul kumu
- ✓ Refrakter harçları (Alumina, Manyezit)
- ✓ DC-AEF için iletken sıcak tamir harcı
- ✓ Pota sürgü sistemleri, Tandiş mekanizmaları ve yedekleri
- ✓ Tandiş akış kontrol sistemi
- ✓ Sürgü Plakası ve Nozul sacları





Aslankaya Döküm yakın zamanda, kendi alanında uzman bir ekip ile Gaziantep 5. Organize Sanayi Bölgesi'nde kurulmuştur. Bölgede ürün kalitesi ve üretim teknolojisi olarak öncü bir marka olma konusunda emin adımlarla ilerleyen firmamız, kaliteyi kendine ilke edinmiştir. 5000 m2 kapalı alanda üretim faaliyetlerimizi gerçekleştiriyoruz. Şirket olarak teknolojik gelişmeleri yakından takip ederek Güneydoğu Anadolu bölgesinin en gelişmiş dökümhane laboratuvarı ve son teknoloji test ve analiz cihazlarıyla döküm sektöründe hizmet vermekteyiz.

Bulunduğu bölgede ilkleri gerçekleştirme becerisine sahip yetenekli insan gücü ve bilgi birikimine sahip olan Aslankaya Döküm, özel dökümhanelere kıyasla ürettiği parça ve yedeklerle bu bilgi birikimi ve becerisini ortaya koymuş ve kendini ispatlamıştır.

Firmamız çevreye duyarlı, kaliteli ve rekabetçi hizmet anlayışıyla üretimin her aşamasında müşteri memnuniyetini ilke edinmiştir. İnşaat ve Maden Endüstrisi, Çimento Endüstrisi, Makine Endüstrisi, Enerji Sektörü başta olmak üzere birçok sektöre ve alana en üst kalite ile üretim yapmaktayız.





# INTERNATIONAL ORGANISATION **FOR MIGRATION (IOM)**



The International Conference of Materials and Engineering Technology

ME

Established in 1951, IOM is the leading inter-governmental organization in the field of migration and works closely with governmental, intergovernmental and non-governmental partners.

With 173 member states, a further 8 states holding observer status and offices in over 100 countries, IOM is dedicated to promoting humane and orderly migration for the benefit of all. It does so by providing services and advice to governments and migrants.

IOM works to help ensure the orderly and humane management of migration, to promote international cooperation on migration issues, to assist in the search for practical solutions to migration problems and to provide humanitarian assistance to migrants in need, including refugees and internally displaced people.

The IOM Constitution recognizes the link between migration and economic, social and cultural development, as well as to the right of freedom of movement. IOM works in the four broad areas of migration management:

- Migration and development
- Facilitating migration
- Regulating migration
- Forced migration.

IOM activities that cut across these areas include the promotion of international migration law, policy debate and guidance, protection of migrants' rights, migration health and the gender dimension of migration.

# **IOM IN TURKEY**

IOM Turkey, the UN Migration Agency first opened its offices in Turkey in 1991 following the aftermath of the Gulf War. IOM's partnership with the Republic of Turkey was formalized in November 2004 when Turkey was

granted member status to IOM. The partnership between IOM and Turkey continued since then, including support drafting the Law of Foreigners and International Protection, as well as establishing the Ministry of Interior's Directorate General for Migration Management in 2013. Now in its 27th year of operations in Turkey, the mission addresses the full scope of migration issues, supporting and developing government capacity to manage migration.

Initially focusing its attention on resettlement for Iraqi refugees in the 1990s, IOM Turkey later expanded in response to the devastating earthquake of 2011 in Van to include emergency response programmes. The mission's emergency response programmes have continued to grow rapidly since 2012 and 2015 with the start of the Syrian crisis and Mediterranean crisis.

Alongside IOM's role in addressing the needs of migrants during crises, the mission works in close collaboration with the Government of Turkey to address the longer-term impact of migration, including migrant assistance programmes, labour integration and migration management, immigration and border management and research and data collection on migrant movement.

Our resettlement programme has also grown to play an important role in the process of resettling refugees abroad through cultural orientation, medical checks and flight arrangements to third countries.

With over 25 years of operational experience in Turkey, the Mission is now one of the largest globally and has more than 900 staff working across the country, with sub-offices in Istanbul, Gaziantep, Hatay, Sanliurfa and Izmir and head office in Ankara.













