



MME SEE

CONGRESS 2023

5th Metallurgical & Materials Engineering
Congress of South-East Europe
Trebinje, Bosnia and Herzegovina
7-10th June 2023

BOOK OF
ABSTRACTS

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C O N G R E S S 2 0 2 3

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The Faculty of Technology and Metallurgy at the University of Belgrade, Serbia;
The Faculty of Technology at the University of Banja Luka, Bosnia and Herzegovina;
The Faculty of Metallurgy at the University of Zagreb in Sisak, Croatia;
The Faculty of Natural Sciences and Engineering at the University of Ljubljana, Slovenia;
The Faculty of metallurgy and technology at the University of Podgorica, Montenegro.

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Editors:

Dr. Miroslav Sokić,

Institute for Technology of Nuclear and Other Mineral Raw Materials

Dr. Branislav Marković

Institute for Technology of Nuclear and Other Mineral Raw Materials

prof. Dr. Vaso Manojlović

Faculty of Technology and Metallurgy, University of Belgrade

Technical editor:

M. Sc. Gvozden Jovanović

Institute for Technology of Nuclear and Other Mineral Raw Materials

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PREFACE

On behalf of the Scientific and Organizing Committee, it is a great honor and pleasure to wish all the participants a warm welcome to the Fifth Metallurgical & Materials Engineering Congress of South-East Europe (MME SEE 2023) which is being held in Trebinje, Bosnia and Hercegovina, 07 - 10 June 2023.

The MME SEE 2023 is a biannual meeting of scientists, professionals, and specialists working in the fields of metallurgical and materials engineering. The aim of the Congress is to present current research results related to processing/structure/property relationships, advances in processing, characterization, and applications of modern materials. Congress encompasses a wide range of related topics and presents the current views from both academia and industry: Future of metals/materials industry in South-East European countries; Raw materials; New industrial achievements, developments and trends in metals/materials; Ferrous and nonferrous metals production; Metal forming, casting, refractories and powder metallurgy; New and advanced ceramics, polymers, and composites; Characterization and structure of materials; Recycling and waste minimization; Corrosion, coating, and protection of materials; Process control and modeling; Nanotechnology; Sustainable development; Welding; Environmental protection; Education; Accreditation & certification.

The editors hope that Congress will stimulate new ideas and improve knowledge in the field of metallurgical and materials engineering. The Congress has been organized by the Association of Metallurgical Engineers of Serbia, with the co-organization of the Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia, Faculty of Technology and Metallurgy, University of Belgrade, Serbia, Faculty of Technology, University of Banja Luka, Bosnia and Herzegovina; the Faculty of Metallurgy, University of Zagreb, Sisak, Croatia; the Faculty of Natural Sciences and Engineering, University of Ljubljana, Slovenia; and the Faculty of Metallurgy and technology, University of Podgorica, Montenegro.

Financial support from the Ministry of Science, Technological Development and Innovation of the Republic of Serbia to researchers from Serbia for attending the congress is gratefully acknowledged. The support of the sponsors and their willingness to cooperate have been of great importance for the success of MME SEE 2023. The Organizing Committee would like to extend their appreciation and gratitude to all sponsors and friends of the conference for their donations and support.

We would like to thank all the authors who have contributed to this book of abstracts and also the members of the scientific and organizing committees, reviewers, speakers, chairpersons, and all the conference participants for their support of MME SEE 2023. Sincere thanks to all the people who have contributed to the successful organization of MME SEE 2023.

On behalf of the 5th MME SEE Scientific and Organizing Committee

Miroslav Sokić, PhD

Table of Contents

Plenary Lectures	1
<i>Corby Anderson</i> STRATEGIC & CRITICAL METAL & MATERIAL PRODUCTION WITH NSC & ASL HYDROMETALLURGICAL TECHNOLOGIES	3
<i>Julia Glaum</i> PIEZOELECTRIC MATERIALS FOR BIOMEDICAL APPLICATIONS: FUNCTIONALITY UNDER SPECIAL BOUNDARY CONDITIONS	4
<i>Malia Athirah Badruddin, Mohd Sabri Mohd Ghazali, Muhamad Syaizwadi Shaifudin, Abdul Mu'iz Aniq Aiman Mohd Suhaimi, Wan Mohamad Ikhmal Wan Mohamad Kamaruzzaman, Nursabrina Amirah Mohd Nasir, Nusaibah Yusof, Chen Soo Kien</i> EFFECT OF DIFFERENT CaSiO ₃ CONTENTS ON THE MICROSTRUCTURE AND ELECTRICAL PERFORMANCE OF ZnO-BASED VARISTOR CERAMICS	5
<i>Subramshu Bhattacharya, Horst Hahn</i> THE FASCINATING WORLD OF MULTICOMPONENT HIGH ENTROPY OXIDES: SYNTHESIS, CHARACTERIZATION AND APPLICATIONS	6
Invited lectures	7
<i>Miljana Radivojević</i> THE RISE OF METALLURGY IN EURASIA: GOING BEYOND THE 'FIRST' AND THE 'EARLIEST' AFTER A DECADE OF ARCHAEOMETALLURGICAL RESEARCH IN THE BALKANS	9
<i>Branimir Grgur</i> CHALLENGES IN ENERGY STORAGE	10
<i>Alexander Birich, Bernd Friedrich</i> GREEN GOLD PRODUCTION FROM PRIMARY AND SECONDARY RESOURCES	11
<i>Srđan Stanković, Stefanie Hetz, Axel Schippers</i> PERSPECTIVES OF LATERITE BIOHIDROMETALLURGY	12
<i>Branko Matović</i> SINTERING OF CERAMICS MATERIALS BY USING ULTRA-HIGH PRESSURES	13
Oral presentations	15
<i>Mile Đurđević, Srećko Manasijević</i> DETERMINATION OF OPTIMAL MODIFIER LEVEL FOR EUTECTIC SILICON PHASE IN HYPOEUTECTIC AlSi ₇ Mg _{0.3} Cu _{0.5} CAST ALLOY.....	17
<i>Vladimir Malbašić, Aleksandar Vasić</i> PRODUCTION AND USE PERSPECTIVES FOR LJUBIJA IRON ORES	18
<i>Grozdana Bogdanović, Sanja Petrović</i> A NOVEL APPROACH TO COPPER LEACHING FROM CHALCOPYRITE WITH SULPHURIC ACID SOLUTIONS.....	19
<i>Dragana Ranđelović, Gvozden Jovanović, Branislav Marković, Miroslav Sokić</i> REE EXTRACTION FROM HYPERACCUMULATING PLANTS: CHALLENGES AND PROSPECTS	20

Miloš Ognjanović, Biljana Dojčinović, Dalibor Stanković, Marija Mirković, Sanja Vranješ-Đurić, Bratislav Antić MULTICORE FLOWER-LIKE MAGNETITE FOR POTENTIAL APPLICATION IN CANCER NANOMEDICINE.....	21
Boštjan Markoli, Iztok Naglič, Blaž Leskovar, Adam Zaky, Žan Kresnik, Andrej Resnik SPECIAL COPPER ALLOYS FOR MOULDS IN PRODUCTION OF HIGH-END GLASS PRODUCTS	22
Iztok Naglič, Blaž Leskovar, Adam Zaky, Boštjan Markoli, Jovica Stojanović, Željko Kamberović QUASICRYSTALS IN Al-Mn-BASED ALLOYS CONTAINING COPPER, CALCIUM AND STRONTIUM.....	23
Adam Zaky, Blaž Leskovar, Iztok Naglič, Boštjan Markoli OPTIMISATION OF AN Al-Mn ALLOY REINFORCED WITH QUASICRYSTALS.....	24
Zdenka Zovko Brodarac, Franjo Kozina, Davor Stanić, Martin Folta QUALITY ASSESSMENT OF THE AlSi ₉ Cu ₃ (Fe) ALLOY WITH REGARD TO RECYCLING RATIO	25
Gordana Marković, Vaso Manojlović, Miroslav Sokić, Jovana Ružić, Dušan Milojkov, Aleksandra Patarić PREDICTING THE MODULUS OF ELASTICITY OF BIOCOMPATIBLE TITANIUM ALLOYS USING MACHINE LEARNING	26
Tatjana Volkov-Husović, Sanja Martinović, Ana Ali, Milica Vlahović CAVITATION EROSION RESISTANCE OF SOME ENGINEERING MATERIALS	27
Sanja Martinović, Ana Alil, Aleksandar Savić, Dragomir Glišić, Dragana Živojinović, Tatjana Volkov-Husović PRINCIPAL COMPONENT ANALYSIS OF MORPHOLOGICAL DESCRIPTORS FOR ASSESSMENT OF SURFACE DEFECTS INDUCED BY EXTREME CONDITIONS	28
Radomir Radiša, Srećko Manasijević, Nedeljko Dučić, Aleksandar Jovičić CIRCULAR ECONOMY AND DIGITALIZATION IN CAST PRODUCTION.....	29
Jovana Ružić, Marko Simić, Dušan Božić, Nikolay Stoimenov, Stanislav Goshev, Jelena Stašić STUDY OF THE CHANGES IN MECHANICAL PROPERTIES OF THE COPPER-ZIRCONIUM ALLOYS INFLUENCED BY MINOR BORON ADDITION	30
Nikola Kanas, Reshma Madathil, Annu Sharma, Vladimir Srdić, Bojan Miljević, Srđan Rakić, Subramshu Bhattacharya, Stevan Armaković UNDERSTANDING THE ELECTRONIC STRUCTURE AND TRANSPORT PROPERTIES OF A-SITE CO-SUBSTITUTED SrTiO ₃ -δ CERAMICS WITH ENHANCED CONFIGURATION ENTROPY	31
Jelena Vujančević, Špela Trafela, Neža Sodnik, Zoran Samardžija, Kristina Žagar Soderžnik ELECTROCHEMICAL SENSORS FOR DETECTION OF BISPHENOLS	32
Wan Mohamad Ikhmal Wan Mohamad Kamaruzzaman, Muhamad Syaizwadi Shaifudin, Nursabrina Amirah Mohd Nasir, Nur Aiman Syafiq Mohd Hamidi, Nusaibah Yusof, Azila Adnan, Lee Oon Jew, Wan Mohd Norsani Wan Nik, Mohd Sabri Mohd Ghazali ANTICORROSIVE IMPROVEMENTS OF WATERBORNE POLYURETHANE COATINGS USING EGGSHELLS BIOWASTE FOR MILD STEEL IN ARTIFICIAL SEAWATER.....	33
Nadira Bušatlić, Ilhan Bušatlić, Dženana Smajić-Terzić GEOPOLYMERS BASED ON FLY ASH FROM THE STANARI THERMAL POWER PLANT	34
Milica Stojković, Sofija Dedić, Maja Đolić, Mirjana Čujić, Vladimir Pavičević, Mirjana Ristić, Aleksandra Perić-Grujić, THE OPTIMIZATION OF METALS AND METALLOIDS EXTRACTION FROM FLY ASH	35

<i>Sonja Smiljanić, Uroš Hribar, Matjaž Spraitzer, Jakob König</i> CRYSTALLIZATION CONTROL IN FOAM GLASS PREPARATION FROM CONTAINER GLASS WASTE	36
<i>Marija Marković, Aleksandra Daković, Danijela Smiljanić, Milena Obradović, Milica Ožegović</i> BENTONITE CLAYS AS AFLATOXIN B ₁ ADSORBENTS.....	37
Poster presentations	39
<i>Milica Vidak Vasić, Lidija Radovanović, Zagorka Radojević</i> RAW KAOLINITIC-ILLITIC CLAYS FOR THE PRODUCTION OF REFRACTORY CERAMICS ...	41
<i>Vladimir Jovanović, Dejan Todorović, Branislav Ivošević,</i> <i>Dragan Radulović, Mladen Bugarčić, Sonja Milićević</i> THE ADVANTAGES OF USING PELLETIZED GYPSUM COMPARED TO POWDERED GYPSUM.....	42
<i>Sanja Jevtić, Vaso Manojlović, Milisav Ranitović,</i> <i>Nataša Gajić, Marija Štulović, Željko Kamberović</i> JAROSITE FROM ZINC HYDROMETALLURGY: THERMODYNAMIC AND KINETIC APPROACH	43
<i>Nenad Milosavljević, Tamara Ristić, Goran Jevtić, Mijajlo Jauković</i> ANALYSIS OF EFFECTIVENESS OF BOTTOM STIRRING AFTER THE MODERNIZATION ON BASIC OXYGEN FURNACE NO. 2 IN HBIS GROUP SERBIA	44
<i>Ivan Stojković, Vladimir Pavićević, Ivana Banković-Ilić, Vlada Veljković</i> COMPARING PURIFICATION METHODS OF CRUDE BIODIESEL OBTAINED BY TRANSESTERIFICATION OF VARIOUS OILY FEEDSTOCKS OVER CALCIUM-BASED CATALYSTS	45
<i>Miloš Marković, Darija Đorđević, Vaso Manojlović, Aleksandar Vasić, Željko Kamberović</i> THE EFFECT OF THE PLASTIC TO COKE RATIO ON THE MOST CRITICAL PROCESS PARAMETERS IN A BLAST FURNACE.....	46
<i>Tomislav Bradarić, Aleksandar Vasić</i> CONTRIBUTION TO LOWER COSTS BY INCREASE OF MANGANESE IN HOT METAL IN HBIS`S STEELWORKS IN SMEDEREVO	47
<i>Vladimir Pavkov, Gordana Bakić, Vesna Maksimović, Ivana Cvijović-Alagić,</i> <i>Marija Prekajski Đorđević, Dušan Bučevac, Branko Matović</i> HIGH-DENSITY GLASS-CERAMIC MATERIALS OBTAINED BY POWDER METALLURGY.....	48
<i>Jelena Stašić, Milan Trtica, Jovana Ružić, Marko Simić, Andrijana Žekić, Dušan Božić</i> SURFACE EFFECTS INDUCED BY SHORT-PULSED LASERS ON PM-SYNTHESIZED CuCrZr ALLOY	49
<i>Marko Simić, Jovana Ružić, Jelena Erčić, Dušan Božić, Željko Radovanović, Jelena Stašić</i> INFLUENCE OF THE MILLING TIME ON THE STRUCTURAL PARAMETERS OF Cu-Zr-B ALLOYS	50
<i>Dimitrije Anđić, Vaso Manojlović, Željko Kamberović,</i> <i>Branislav Marković, Nataša Gajić, Miljana Popović, Gvozden Jovanović</i> EVALUATION OF PARAMETERS FOR THE PRODUCTION OF Al-WO ₃ COMPOSITES.....	51
<i>Muhamad Syaizwadi Shaifudin, Mohd Sabri Mohd Ghazali,</i> <i>Wan Mohamad Ikhmal Wan Mohamad Kamaruzzaman, Wan Rafizah Wan Abdullah, Lee Oon Jew</i> MICROSTRUCTURE AND ELECTRICAL PERFORMANCE OF BARIUM TITANATE DOPED ZINC OXIDE VARISTOR CERAMICS VIA SIMPLIFIED CITRAGE-GEL METHOD	52

<i>Danina Krajišnik, Snežana Uskoković-Marković, Aleksandra Daković, Danijela Smiljanić, Maja Kozarski, Vesna Lazić</i> PREPARATION AND CHARACTERIZATION OF CHITOSAN-CLAY COMPOSITES AS POTENTIAL DRUG CARRIERS	53
<i>Ljubiša Balanović, Dajana Milkić, Dragan Manasijević, Ivana Marković, Milan Gorgievski, Uroš Stamenković, Kristina Božinović</i> MICROSTRUCTURAL AND THERMAL CHARACTERIZATION OF THE Bi-In-Sn TERNARY ALLOYS	54
<i>Ljubica Radović, Fernando Carrasco López, Igor Radisavljević, Nenad Radović</i> DISTRIBUTION OF ALLOYING ELEMENTS IN FRICTION STIR WELDED AA2024-T351 ALLOY	55
<i>Dragana Mihajlović, Marko Rakin, Đorđe Veljović, Bojan Međo, Veljko Đokić</i> ADVANCEMENT OF BIOCOMPATIBILITY AND MECHANICAL SURFACE CHARACTERISTICS OF THE Ti-13Nb-13Zr ALLOY USING ELECTROCHEMICAL ANODIZATION	56
<i>Natalija Dolić, Zdenka Zovko Brodarac, Franjo Kozina</i> INFLUENCE OF HOMOGENIZATION ON CHEMICAL PROPERTIES OF ALUMINUM ALLOY EN AW-5083	57
<i>Dragan Manasijević, Ljubiša Balanović, Ivana Marković, Milan Gorgievski, Uroš Stamenković, Kristina Božinović, Duško Minić, Milena Premović Zečević</i> MICROSTRUCTURE AND THERMAL CONDUCTIVITY OF THE Ag-Bi-Sn TERNARY ALLOYS	58
<i>Marija Mihailović, Aleksandra Patarić, Snežana Aksentijević, Branka Jordović</i> MICROSTRUCTURE DEVELOPMENT DURING HEAT TREATMENT OF HIGH CHROMIUM WHITE CAST IRON	59
<i>Aleksandra Patarić, Gordana Marković, Nataša Đorđević, Slavica Mihajlović, Marija Mihailović</i> MICROSTRUCTURE ASSESSMENT OF Co ALLOY INTENDED FOR DENTISTRY	60
<i>Jakov Dimitrijević, Julijana Tadić, Vukašin Ugrinović, Tamara Matić, Đorđe Janačković, Đorđe Veljović</i> IMPROVED MECHANICAL AND BIOLOGICAL PROPERTIES OF CHITOSAN COATED SCAFFOLDS BASED ON HYDROXYAPATITE DOPED WITH Mg, Sr AND Cu.....	61
<i>Aleksandra Popović, Branimir Grgur</i> OPTIMIZING ELECTROCHEMICAL ANALYSIS FOR THE ACCURATE CHARACTERIZATION OF ELECTRODE MATERIALS IN ENERGY STORAGE APPLICATIONS: A COMPREHENSIVE GUIDE TO ELECTROCHEMICALLY ANALYZING MATERIALS.....	62
<i>Maja Kokunešoski, Aleksandra Šaponjić, Zvezdana Baščarević, Ivan Jovanović, Jelena Filipović Tričković, Ana Valenta Šobot</i> PRELIMINARY CYTOTOXICITY TESTING OF NEWLY SYNTHESISED SBA-15 MATERIAL....	63
<i>Jelena Nikolić, Vladimir Topalović, Marija Došić, Veljko Savić, Milena Obradović, Srđan Matijašević, Snežana Grujić</i> DISSOLUTION OF POLYPHOSPHATE GLASS: IMPACT OF pH ON DIFFUSION COEFFICIENTS OF MODIFYING CATIONS AT LOW TEMPERATURES	64
<i>Tatjana Šoštarić, Stefan Kolašinac, Zorica Lopičić, Anja Antanasković, Vladimir Adamović, Jelena Avdalović, Mladen Bugarčić</i> CHARACTERIZATION OF RAW PEACH STONES AND ITS BIOCHAR BY SEM, FTIR AND RAMAN SPECTROSCOPY	65
<i>Aleksandar Đorđević, Milena Zečević, Duško Minić</i> EFFECT OF CHEMICAL COMPOSITION ON THE MICROSTRUCTURE, HARDNESS, AND ELECTRICAL CONDUCTIVITY OF THE TERNARY Bi-Cu-Ge SYSTEM.....	66

<i>Uroš Stojaković, Aleksandar Jovanović, Ivana Mikavica, Branislav Marković, Vladimir Pavićević</i> DIRECTIONS AND CHALLENGES OF THE CIRCULAR ECONOMY: MOVEMENT OF MUNICIPAL SOLID WASTE IN CITY OF PARAČIN	67
<i>Silvana Dimitrijević, Stevan Dimitrijević, Aleksandra Ivanović, Marija Korać, Milisav Ranitović</i> COMPARATION OF CORROSION AND MECHANICAL PROPERTIES OF COMMERCIAL AND RECYCLED 6060 AND 6082 ALUMINIUM ALLOYS	68
<i>Milisav Ranitović, Željko Kamberović, Marija Korać, Dragana Ivsić, Nikola Jovanović</i> INFLUENCE OF WPCBS MECHANICAL PRE-PROCESSING ON BASE METALS LEACHING EFFICIENCY	69
<i>Aleksandra Ivanović, Silvana Dimitrijević, Stevan Dimitrijević, Renata Kovačević</i> DEALLOYING OF PdNi ₅ ALLOY IN 1.0 M NITRIC ACID	70
<i>Marija Korać, Stevan Dimitrijević, Kemal Delijić, Silvana Dimitrijević, Željko Kamberović</i> INFLUENCE OF ZINC ADDITION ON ANTI-TARNISH SILVER ALLOYS IN FOUR DIFFERENT SYSTEMS.....	71
<i>Miljana Popović, Ana Alil, Bojan Gligorijević, Endre Romhanji</i> INFLUENCE OF COLD ROLLING AND ANNEALING ON THE MECHANICAL AND CORROSION PROPERTIES OF AN AA5182 Al-Mg ALLOY.....	72
<i>Milovan Stoiljković, Vladimir Pavkov, Gordana Bakić, Aleksa Luković, Vesna Maksimović</i> CORROSION OF CERAMIC-METAL COMPOSITES IN ARTIFICIAL ACID RAIN	73
<i>Milana Zarić, Mirjana Kijevčanin, Ivona Radović</i> INFLUENCE OF FUNCTIONAL GROUPS ON THERMODYNAMIC PROPERTIES OF BINARY MIXTURES	74
<i>Gvozden Jovanović, Vaso Manojlović, Miroslav Sokić, Alen Delić, Milorad Gavrilovski</i> INFLUENCE OF MOLD PREHEATING ON RAILWAY ALUMINOTHERMIC WELDING CASTING SIMULATION.....	75
<i>Miloš Ognjanović, Biljana Dojčinović, Bratislav Antić, Dalibor Stanković</i> NANOSCALE METAL OXIDES AS MATERIALS USED FOR MODIFICATION OF CRBORN-BASED ELECTRODES IN ELECTROCHEMICAL SENSORS	76
<i>Dušan Milojkov, Miroslav Sokić, Angelina Mitrović, Danijela Smiljanić, Jelena Petrović, Marija Simić, Vukosava Živković-Radovanović</i> DEVELOPMENT OF SUSTAINABLE METHOD FOR METAL RECOVERY FROM OLD FLOTATION TAILINGS (MAJDANPEK, SERBIA) USING <i>Aspergillus niger</i> FUNGUS	77
<i>Nemanja Barać, Katarina Dimić-Mišić, Monireh Imani, Petar Uskoković, Ernesto Barceló, Patrick Gane, Đorđe Janačković</i> APPLICATION OF SUSTAINABLE MATERIALS IN THE NO _x REDUCTION OF AIR POLLUTION.....	78
<i>Vesna Lazarević, Ivan Stojković, Ivana Banković-Ilić, Vlada Veljković</i> DESIGN OF A PILOT PLANT FOR CHEMICAL TREATMENT OF THE SPENT MINERAL OIL-IN-WATER EMULSION FROM NON-FERROUS METAL PROCESSING	79
<i>Stefan Dikić, Mihajlo Arandjelović, Simon Sedmak, Radomir Jovičić, Dragomir Glišić</i> EFFECTS OF MULTIPLE WELDING DEFECTS ON MECHANICAL PROPERTIES OF WELDED JOINT	80
<i>Nebojša Tadić, Mitar Mišović, Žarko Radović, Ratka Petrović</i> CHARACTERIZATION OF FRICTION STIR WELDING JOINTS IN 7075-T6 ALLOY PLATES.....	81
<i>Aleksandar Jovanović, Mladen Bugarčić, Marija Stevanović, Miroslav Sokić, Aleksandar Marinković</i> SEQUENCING BATCH REACTOR SYSTEMS FOR THE TREATMENT OF WASTEWATER.....	82

<i>Marija Simić, Jelena Petrović, Marija Ercegović, Marija Koprivica, Jelena Dimitrijević, Aleksandar Jovanović, Dušan Milojkov</i> Mg/Fe-PYRO-HYDRO CHAR DERIVED FROM CORN COB AS EFFECTIVE ADSORBENT OF LEAD REMOVAL FROM AQUEOUS SOLUTIONS	83
<i>Janko Živanić, Mladen Bugarčić, Anja Antanasković, Zorica Lopičić, Miroslav Sokić, Aleksandar Jovanović, Milan Milivojević</i> THE ADSORPTION OF ARSENATE IONS FROM AQUEOUS SOLUTIONS BY COMPOSITE PARTICLES OF NATURAL POLYMERS AND METAL OXIDES	84
<i>Danijela Smiljanić, Aleksandra Daković, Milena Obradović, Marija Marković, Milica Ožegović, Milena Pantić, Danina Krajišnik</i> CLAY-CHITOSAN-SURFACTANT COMPOSITES AS EFFICIENT ADSORBENTS OF ZEARALENONE.....	85
<i>Jelena Petrović, Marija Ercegović, Marija Simić, Marija Koprivica, Marija Marković, Jelena Dimitrijević</i> HYDROCHAR-DERIVED HIGHLY EFFICIENT ADSORBENT FOR COPPER IONS REMOVAL ..	86
<i>Nela Vujović, Vesna Alivojvodić, Miroslav Sokić, Gvozden Jovanović, Branislav Marković, Željko Kamberović</i> INNOVATIVE INDUSTRIAL SYMBIOSIS APPROACH FOR ACIDIC MINE DRAINAGE NEUTRALIZATION.....	87
<i>Jovana Bošnjaković, Nataša Knežević, Aleksandar Marinković, Srećko Manasijević, Andrija Savić, Milena Milošević</i> REMOVAL OF MESOTRIONE BY HETEROGENEOUS PHOTOCATALYTIC TREATMENT USING UV-VIS LAMP AS LIGHT SOURCE	88
<i>Nataša Knežević, Jovana Bošnjaković, Marija Vuksanović, Jovana Milanović, Ljiljana Janković-Mandić, Adela Egelja, Aleksandar Marinković</i> TESTING OF THE ADSORPTION-DEGRADATION CAPACITY OF CRYSTAL VIOLET DRY WITH OXIDIZED COTTON LINTERS	89
<i>Katarina Sokić, Jelena Dikić, Đorđe Veljović, Jovana Đokić, Zoran Anđić, Sanja Jevtić</i> MECHANOCHEMICAL SYNTHESIS AND CHARACTERIZATION OF THE ADSORBENTS BASED ON NATURAL ZEOLITE AND HYDROXYAPATITE.....	90
<i>Borislav Malinović, Anđela Bojić, Draženko Bjelić, Tijana Đuričić</i> NICKEL RECOVERY FROM STATIC RINSING IN THE ELECTROPLATING PROCESS	91
<i>Dimitrije Petrović, Marija Egerić, Yi-nan Wu, Aleksandar Vukadinović, Aleksandar Devečerski, Ljiljana Matović, Radojka Vujasin</i> PHOTOCATALYTIC DEGRADATION OF CONGO RED DYE USING UiO-66 MOF-METAL OXIDES COMPOSITES.....	92
<i>Abhilash Krishnamurthy, Špela Trafela, Kristina Žagar Soderžnik</i> DETECTION AND SENSING OF BENZENEDIOLS USING MODIFIED SCREEN PRINTED ELECTRODES	93
<i>Prince Anand, Arnab Das, Vivek Bajpai</i> MACHINABILITY OF Ti ₆ Al ₄ V IN HIGH SPEED MICRO TURNING UNDER DRY CONDITION.....	94
<i>Abhipsa Kar, Vivek Bajpai, Ravi Shankar Rai</i> ANALYSIS OF HOLE QUALITY IN MICRO DRILLING OF METAL-OXIDE NANOSTRUCTURED CFRP COMPOSITES	95
<i>Rajesh Sahoo, Vivek Bajpai, Nirmal Kumar Singh</i> APPROACH TOWARDS GREEN MANUFACTURING IN MAGLEV EDM USING DIFFERENT BIO-DIELECTRICS AT VARIABLE DISCHARGE CONDITIONS	96

Rajesh Sahoo, Sakshit Wankhede, Vivek Bajpai

GEOMETRICAL INSPECTION OF TOOL WEAR EFFECT

IN MAGLEV MICRO-EDM PROCESS 97

AUTOR INDEX 98

Plenary Lectures

**STRATEGIC & CRITICAL METAL & MATERIAL PRODUCTION
WITH NSC & ASL HYDROMETALLURGICAL TECHNOLOGIES**

Corby Anderson¹

e-mail: cganders@mines.edu

¹-Colorado School of Mines, Department of Metallurgical and Materials Engineering, 1500 Illinois St., Golden, CO 80401, USA

The need for domestic high quality Strategic & Critical Metal and Material production is growing rapidly as the global Renewable Energy transformation occurs. Hydrometallurgy innately offers enhanced separations for efficiently creating these. The author has intellectual property and industrial experience in two key applied hydrometallurgical technologies that can readily address this domestic challenge. These are Nitrogen Species Catalyzed (NSC) and Alkaline Sulfide Leaching (ASL). This presentation will elucidate the history and fundamentals of these proven industrial technologies. Further, specific applications to domestic production of Strategic & Critical Metals and Materials from both primary and recycled sources will be detailed and based upon 35 years of focused development and application by the Author.

Keywords: environmental pollution, biological treatment, nutrient removal, sequencing batch reactors.

PIEZOELECTRIC MATERIALS FOR BIOMEDICAL APPLICATIONS: FUNCTIONALITY UNDER SPECIAL BOUNDARY CONDITIONS

Julia Glaum¹

e-mail: Julia.glaum@ntnu.no

¹-Department of Materials Science and Engineering, NTNU Norwegian University of Science and Technology, Trondheim, Norway

The ability to convert an electrical field into a mechanical perturbation and vice versa makes piezoelectric materials fundamentally interesting objects of study as well as versatile components for industrial applications. In recent years, research on piezoelectric materials for biomedical applications, as for nerve and bone tissue repair, *in vivo* sensors or energy harvesting components, has gained significant momentum. However, the boundary conditions that have to be met to make these materials work in an *in vivo* environment are quite different to the ones in their established industrial applications. And even though piezoelectric materials could enable many *in vivo* applications, e.g. localized pressure sensing, energy harvesting from muscle motion or stimulation of tissue re-generation after surgery, they have not made the jump into clinical usage yet. The main challenge here is that they have to be biocompatible. This is a concept that goes way beyond simple chemical toxicity but covers all aspects that influence the safe performance of a material at the implant site under the complex conditions that the body imposes. Material and implant design have to be re-thought to match these complex requirements.

In this presentation, we will take an in-depth look at the applicability and biocompatibility of piezoelectric materials for *in vivo* biomedical applications. We will investigate the boundary conditions that the body imposes on implant materials in different applications and how these might impact the functionality and stability of piezoelectric implants. And vice versa we will look into the influence of material properties, such as surface topography, chemical composition and mechanical properties, on the living system. Furthermore, I will give an overview of some steps we have taken to enable piezoelectric ceramics for bone implant applications in terms of material design, long-term stability and compliance with existing clinical procedures.

Piezoelectric materials show great promise for a wide range of biomedical applications. Each comes with their specific set of biochemical and mechanical boundary conditions. To transfer piezoelectric materials safely into the biomedical realm, a fundamental understanding of the complex interplay between them and their host environment before and during the implantation period is crucial – it's a wide field of research!

Keywords: piezoelectric, biomedical, ceramics

EFFECT OF DIFFERENT CaSiO₃ CONTENTS ON THE MICROSTRUCTURE AND ELECTRICAL PERFORMANCE OF ZnO-BASED VARISTOR CERAMICS

Malia Athirah Badruddin¹, Mohd Sabri Mohd Ghazali¹, Muhamad Syaizwadi Shaifudin¹,
Abdul Mu'iz Aniq Aiman Mohd Suhaimi¹, Wan Mohamad Ikhmal Wan Mohamad Kamaruzzaman¹,
Nursabrina Amirah Mohd Nasir¹, Nusaibah Yusof¹, Chen Soo Kien²

e-mail: mohdsabri@umt.edu.my

1-Faculty of Science and Marine Environment, University Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia,

2-Department of Physics, Faculty of Science, University Putra Malaysia, 43400 UPM Serdang, Selangor

The present study highlights the microstructural and electrical evaluation of ZnO-based varistor ceramics with CaSiO₃ addition. The pelletized samples were prepared via the solid-state reaction method by varying the CaSiO₃ concentrations (0, 3, 6, 9, 12, and 15 wt.%) and sintered at 1300 °C. The characterization was carried out using density measurement, scanning electron microscopy (SEM), energy dispersive X-ray (EDX), X-ray diffraction (XRD), and current density-electric field (J-E) characteristics measurement. The introduction of CaSiO₃ inhibited grain growth; the average grain size reduced from 18.09 μm to 10.85 μm, whereas their relative density increased up to 99.79%. The existence of the CaSiO₃-rich phase was confirmed via structural analysis without any other secondary phase formation detected. It is also discovered that different CaSiO₃ contents significantly affect the electrical properties, particularly in the 12 wt.% CaSiO₃ addition sample. The highest values of the nonlinear coefficient, breakdown voltage, and barrier height obtained in this work were 4.41, 0.80 V/mm, and 0.691 eV, respectively, along with a sudden drop in leakage current density to 373.93 μA/cm². Further increase of CaSiO₃ content (15 wt.%) eventually deteriorated the varistor behaviour.

Keywords: Varistor ceramics, Zinc oxide, Calcium silicate, Microstructure, Electrical behaviour

THE FASCINATING WORLD OF MULTICOMPONENT HIGH ENTROPY OXIDES: SYNTHESIS, CHARACTERIZATION AND APPLICATIONS

Subramshu Bhattacharya¹, Horst Hahn^{2,3}

e-mail: ssb@iitm.ac.in

1-Nanofunctional Materials Technology Centre (NFMTC), Dept. of Metallurgical & Materials Engg, IIT Madras, Chennai, India,

2-Institute of Nanotechnology, Karlsruhe Institute of Technology, Karlsruhe, Germany,

3-University of Oklahoma, Chemical, Biological and Materials Engineering, Norman, OK, United States

Multicomponent equimolar oxides or high entropy oxides (HEOs) with phase-pure crystal structures have been recently garnering a lot of research interest due to their fascinating properties and potential in a large number of applications. The fundamental premise appears to point to the fact that the presence of a large number of cations in a random fashion in the crystal sublattice(s) results in its stabilization in a phase-pure form due to the increased configurational entropy and that the overall properties of the compounds are superior to those exhibited by the individual unary oxides. Several different cases of nanocrystalline HEOs have been considered here wherein 5 or more transition metal or rare earth cations or a combination of the two have been incorporated in the crystal sublattice(s) to form a phase-pure (a) cubic rocksalt structure (R-HEO), (b) fluorite structure (F-HEO), (c) spinel structure (S-HEO) and (d) perovskite structure (P-HEO). The selection of the cations in all the cases was made on the basis of Hume-Rothery and Pauling's rules.

The phase-pure nanocrystalline powders were synthesized by wet chemical, reverse co-precipitation (RCP) processes as well as by aerosol processes (flame spray pyrolysis, FSP or nebular spray pyrolysis, NSP) using nitrate precursors. Standard characterization techniques were used to ascertain the phases and phase composition, and the crystallite size (X-ray diffraction, XRD), particle size and morphology (high resolution transmission and scanning electron microscopy, HRTEM and HRSEM), phase stability at different temperatures (thermogravimetry and differential scanning calorimetry, TG-DSC), bond structure (Fourier transform infra-red and Raman spectroscopy, FT-IR and FT Raman), oxidation states (X-ray photoemission spectroscopy, XPS) and bandgap energy (photoluminescence and diffuse reflectance spectroscopy, PL and DRS) were measured. While the rocksalt type R-HEO was entropy stabilized, factors other than entropy were found to stabilize the single phase in the case of the fluorite type F-HEO. R-HEO showed high room temperature lithium ion conductivity with superior capacity retention ability when used as an electrode material for secondary Li-ion batteries. All the HEOs exhibited a narrow bandgap, lower than the constituent unary oxides. These studies show that high entropy oxides form a novel class of materials that can be used in a variety of practical applications along with a wide range of tuning possibilities.

Keywords: High entropy oxide (HEO); Entropy stabilization; Phase-pure Rocksalt, Fluorite, Spinel and Perovskite structures

Invited lectures

THE RISE OF METALLURGY IN EURASIA: GOING BEYOND THE ‘FIRST’ AND THE ‘EARLIEST’ AFTER A DECADE OF ARCHAOMETALLURGICAL RESEARCH IN THE BALKANS

Miljana Radivojević¹

e-mail: m.radivojevic@ucl.ac.uk

1-UCL Institute of Archaeology

Metallurgy of the Vinča culture (5300 – 4400 BC) is currently known as the earliest in the world and dates to the beginning of the 5th millennium BC. Besides the earliest documented copper and lead smelting, tin bronze-making emerges in 4650 BC alongside the appearance of gold in the East Balkans.

These finds demonstrate that the technology of metal making in the Balkans evolved in a dynamic that is different from traditional models of the emergence of metallurgy based on the Near Eastern evidence, in the case of tin bronze even 1,500 years before the earliest finds in the ‘Old World’. Such discoveries from the Balkan prehistory, point out the necessity to revise current hypotheses on the evolution of Eurasian and global metallurgy.

Here I present a synthesis of all results to date that address the technology, provenance, and circulation of the 5th millennium BC Balkan metals and metal making knowledge with the focus on Vinča culture metal artefacts. Using materials science approaches, I reveal the recipes for making the earliest copper to date, the experimental reconstruction of these in the workshop settings, as well as the role of aesthetics in inventing and innovating in metallurgy 7,000 years ago.

With complex networks methodology, I present the scope of cooperation that can be observed in the trade and exchange patterns of metals across the Balkans for the duration of the Vinča culture, and beyond.

Multiple methodologies and approaches applied thus far in this research call for a novel model of the evolution of metallurgy worldwide, one that offers a greater focus set on understanding both cultural and technological preferences of the early metal making communities.

Keywords: archaeometallurgy, Balkans, copper smelting, Vinča culture, networks

CHALLENGES IN ENERGY STORAGE

Branimir Grgur¹

e-mail: BNrgur@tmf.bg.ac.rs

1-University of Belgrade, Faculty of Technology and Metallurgy, Department of Physical chemistry and electrochemistry, Karnegijeva 4, 11120 Belgrade, Serbia

Portable electronic devices, electric vehicles, and renewable electric energy storage are today of enormous importance for human civilization's prosperity. But, different uses require different electrochemical power sources concerning size, capacity energy and power.

Lecture will consider different metal-ion systems (Li, Na, Ca, Mg) [1-4], lead-acid UltraBattery [5] redox flow batteries [6, 7], and rechargeable metal-air batteries [8, 9]. For all the systems, merits and drawbacks of numerated systems will be considered. The main objective of this lecture will be devoted to the state of the art of different electrochemical energy storage systems, and challenges concerning their price, electrical characteristics, and safety, as well as possibilities of further improvements.

Keywords: Metal-ion battery, Lead-acid, Redox flow batteries, Metal-air batteries

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GREEN GOLD PRODUCTION FROM PRIMARY AND SECONDARY RESOURCES

Alexander Birich¹, Bernd Friedrich¹

e-mail: abirich@ime-aachen.de

¹-RWTH Aachen University, Institute for Process Metallurgy and Metal Recycling

The excellent chemical resistance of gold is an important property of the precious metal, but it becomes a disadvantage when it comes to gold extraction. Many metallurgical processes are based on highly aggressive or toxic reagents. Especially artisanal and small-scale gold mining activities are heavily criticized, as their environmental impact can hardly be controlled. Ecological concerns are forcing society and industry to find a solution for efficient gold recovery by the use of alternative methods to replace the mercury or cyanide process. In the last decades, many alternative leaching reagents have been investigated extensively. Researchers showed promising results in terms of technical and environmental characteristics. Furthermore, different demonstration and pilot-scale application tests were conducted around the world to generate information for a potential industrial application. Nevertheless, to this day there is just one reported industrial-scale application using the cyanide alternative thiosulfate, which is the Barrick Gold process in Nevada, US. Besides primary production, there is no notable industrial application of cyanide alternatives in the recycling sector. Especially gold recycling from secondary resources would have a high ecological impact, as mining activities could be avoided or reduced. For example, electronic waste is one of the most promising secondary resources due to its broad consumption and relatively high gold concentration.

In this work, the potential of cyanide alternative reagents for industrial application in gold production and recycling is discussed along with various reasons that explain the restrained acceptance of gold producers.

Keywords: gold, cyanide alternatives, green gold, recycling

PERSPECTIVES OF LATERITE BIOHIDROMETALLURGY

Srđan Stanković¹, Stefanie Hetz¹, Axel Schippers¹

e-mail: srdjan.stankovic@bgr.de

¹-Federal Institute for Geosciences and Natural Resources, Stillweg 2, 30655 Hannover; Germany

Biohydrometallurgy has been applied for decades mostly for copper and gold extractions from mineral raw materials. Conventional biohydrometallurgy for processing of metal sulfides is based on microbial oxidation of iron. Microorganisms, such as bacteria and archaea, catalyze the dissolution of sulfide minerals by very efficient oxidation of reduced (ferrous) iron, generating ferric iron which acts as oxidizing agent on sulfide minerals (such as pyrite or copper sulfides). Biohydrometallurgy of laterites is based on a different approach - a so-called reductive bioleaching. Laterites are supergene ore bodies which are hosting approximately 70 % of global nickel reserves. These ores are constituted of oxidized minerals. The idea behind reductive bioleaching is based on a bacterial reduction of ferric iron to ferrous iron coupled with the microbial oxidation of elemental sulfur to sulfuric acid. Microbially generated ferrous iron acts as a reducing agent dissolving some of the nickel and cobalt-bearing oxide minerals. Research on laterite biohydrometallurgy is relatively new, the first papers have been published at the beginning of the second decade of the 21st century. The purpose of this paper is to give an overview of progress in research on laterite biohydrometallurgy and to predict its future development. The main driving force behind research in this field is the development of a process that is more friendly to the environment in comparison to other available hydrometallurgical technologies for the treatment of lateritic ores. The current scientific efforts are focused on the development of a relatively simple process with reduced CO₂ emission and waste generation.

Keywords laterites, nickel, cobalt, biohydrometallurgy

SINTERING OF CERAMICS MATERIALS BY USING ULTRA-HIGH PRESSURES

Branko Matović¹

e-mail: mato@vinca.rs

¹University of Belgrade, Vinča Institute of Nuclear Sciences - National Institute of the Republic of Serbia,
Centre of Excellence "CEXTREME LAB", Mike Petrovica Alasa 12-14, 11000 Belgrade, Serbia

Pressure is a fundamental thermodynamic variable that can provide a unique possibility to control the structure and properties of materials due to breaking existing or forming of new chemical bonds. The pressure induced changes in chemical affinities can significantly alter the reactivity of elements and compounds, opening door to synthesis of new classes of materials. Pressure is a versatile tool in materials engineering, geological sciences, solid state physics, and chemistry. Under the influence of high pressure and temperature, there are a lot of changes in physical, chemical, and structural properties of materials. It gives a possibility to generate of new, non-existent nature phases, or phases which occur only in inaccessible places, such as the earth's core. On the other side, the application of high compression pressure leads to deformation of the particles, such that the green density increases. Also, the occurrence of pressure-induced phase transformation promotes densification (metastable crystal structures to a more stable structure), which is often accompanied by a significant reduction in free volume. In this presentation, a high quasi-hydrostatic pressure of 4 GPa was applied with subsequent annealing of green pellets at four different temperatures for 4 minutes in an ambient atmosphere using a Bridgman-type toroidal apparatus (heating rate 450 °C/min). This technique can be successfully applied to obtain full-density ceramics without any additives. Five different compounds (oxides and non-oxides) as well as their composites have been studied to determine the effects of high pressures on their densification. The use of high pressures at comparatively low temperatures and short sintering time makes it possible to obtain mentioned materials with improved mechanical properties as compared to all other known methods.

Keywords: Ultra-high pressure; Densification; Oxides; Nonoxides; Composites; Microstructure.

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Oral presentations

DETERMINATION OF OPTIMAL MODIFIER LEVEL FOR EUTECTIC SILICON PHASE IN HYPOEUTECTIC $\text{AlSi}_7\text{Mg}_{0.3}\text{Cu}_{0.5}$ CAST ALLOY

Mile Đurđević¹, Srećko Manasijević²

e-mail: mile.djurdjevic@nemak.com

1-Nemak Linz GmbH, Zeppelinstrasse 24, 4030 Linz Austria;

2-Lola Institut Ltd., Knez Visaslava 70a, 11030 Belgrade, Serbia

The modification of silicon particles in as-cast aluminum alloys can be done with different modifiers. Strontium and sodium are mostly used modifiers in the aluminum casting industry. Both lose concentration (fade) while held in the melting furnace. Three types of chemical reactions in the melt may cause modifier fading. The modifier may vaporize due to high vapor pressure at melt temperatures, oxidize due to an excessive chemical affinity for oxygen, or react with some other elements from the melt building intermetallics. Due to sodium and strontium's very low vapor pressure, their vaporization from the aluminum melt was excluded as a reason for the modifier's fading. Oxidation looks like the major chemical reaction that causes fading of sodium and strontium from the aluminum melt. This study examined the effect of strontium and sodium fading on the modification level of silicon particles in the $\text{AlSi}_7\text{Mg}_{0.3}\text{Cu}_{0.5}$ (356) aluminum alloy. The cooling curve analysis has been applied to quantify modifier fading. Approximately 105 ppm of strontium in the form of master alloy (AlSr10) and 67 ppm of sodium in the tablet form were separated and added to the melt. Both modifiers were held for approximately 200 minutes in the melt to observe their fading. The present paper aimed to quantify the fading of sodium and strontium in an $\text{AlSi}_7\text{Mg}_{0.3}\text{Cu}_{0.5}$ alloy. The loss of modifiers (Na and Sr) during melt holding in a furnace can be analytically quantified using equations taken from literature. The calculated surface reaction rate constant (k_s) can be used to estimate modifiers loss during melt holding in industrial and laboratory furnaces.

Keywords: Thermal Analysis, Modifier, Fading, $\text{AlSi}_7\text{Mg}_{0.3}\text{Cu}_{0.5}\text{Mg}$ alloy, Surface reaction rate constant

PRODUCTION AND USE PERSPECTIVES FOR LJUBIJA IRON ORES USE

Vladimir Malbašić¹, Aleksandar Vasić²

e-mail: vladimir.malbasic@rf.unibl.org

1-University of Banja Luka-Mining Faculty Prijedor,

2-External associate IC-TMF Belgrade

The paper presents the current state, quantities and quality of proven reserves, as well as the perspectives for finding additional iron ore reserves in the area of the Ljubija mine. The production of iron ore on an industrial level has been carried out in the area of the Ljubija mine since 1916 and lasted until 1992. Mainly due to known events, the market for this iron ore types was lost, production was not continued in the past period until now. The appearance of the possibility of its placement raises the questions of techno-economic profitability of starting production and defining the conditions of exploitation, primary processing and use for steel production

The paper briefly provides an overview of the geological conditions as well as the types, quantities and quality of iron ores of the Ljubija Mine, with analysis of the production and technical conditions of mining exploitation and preparation, metallurgical characteristics and usability with a proposal for the valuation of the Ljubija ores.

At the end, the authors of the paper tried to give proposals for future activities on the sustainable continuation of iron ore production at the Ljubija Mine.

Keywords: Ljubija mine, iron ores, mining exploitation, metallurgical evaluation

A NOVEL APPROACH TO COPPER LEACHING FROM CHALCOPYRITE WITH SULPHURIC ACID SOLUTIONS

Grozdanka Bogdanović¹, Sanja Petrović²

e-mail: gbogdanovic@tfbor.bg.ac.rs

1-University of Belgrade, Technical Faculty in Bor, VJ 12, P.O. Box 50, 19210 Bor, Serbia,

2-Mining and Metallurgy Institute Bor, Zelene bulevar 35, 19210 Bor, Serbia

Chalcopyrite is the most abundant copper sulphide mineral. Extensive research in the field of hydrometallurgy was conducted with the aim of developing efficient processes for leaching copper from chalcopyrite concentrates and low-grade ores and/or complex chalcopyrite-containing ores. Chalcopyrite leaching in sulphuric acid solution was most studied with Fe (III) ions as an oxidant, with or without the assistance of microorganisms due to the simplicity and low cost of the process.

In recent years, there has been increased interest of potential use of different oxidizing reagents (hydrogen peroxide, ozone, chromium (VI) ions, etc.) for chalcopyrite leaching, with research being conducted only at the laboratory level, probably due to their higher cost. The addition of organic compounds, such as alcohols (methanol, ethanol, ethylene glycol, isopropanol), in the acidic aqueous solution of hydrogen peroxide and sulfuric acid, had increased chalcopyrite leaching. The aim of this paper is to present a brief overview of the oxidative leaching of chalcopyrite by means of hydrogen peroxide in sulphuric acid solutions and with the presence of alcohols.

Keywords: leaching, chalcopyrite, sulphuric acid, hydrogen peroxide, alcohols

REE EXTRACTION FROM HYPERACCUMULATING PLANTS: CHALLENGES AND PROSPECTS

Dragana Randelović¹, Gvozden Jovanović¹, Branislav Marković¹, Miroslav Sokić¹

e-mail: g.jovanovic@itnms.ac.rs

¹Institute for Technology of Nuclear and other Mineral Raw Materials, Belgrade, Serbia

Phytomining although predominantly in its early stages on the broader scientific scope of investigation has garnered interest in metals such as Ni, Au, Zn or REE. Rare earth elements (REE) are commonly defined as the 15 lanthanides with the addition of Sc and Y. Since studies of other hyperaccumulating plant species have been conducted before, similar techniques were tried with REE. With the concentration threshold at 0.1 wt% of the dry biomass there have been identified about 22 plant species that hyperaccumulate REE from five families (Phytolaccaceae, Gleicheniaceae, Blechnaceae, Juglandaceae, Thelypteridaceae). One of the most studied REE hyperaccumulators is *Dicranopteris linearis* (formerly known as *D.dichotoma*), a fern native to China that grows on mine tailings. Generally, the techniques for REE phytomining can be separated into Pyrometallurgy, Hydrometallurgy, Biometallurgy and Novel approaches. Currently most of the research is still focusing on the optimal utilization of various hiperaccumulators and the enchantment of REE extraction from the soil as well as from the enriched biomass. Since recovery of REEs via phytomining is just in its infancy further multidisciplinary investigations are needed, especially when it comes to valorization and REE recovery. This paper presents a quick review for the concept of REE phytomining, current state of research, challenges and prospects.

Keywords: Phytoextraction, Rare earth elements, Secondary resources

MULTICORE FLOWER-LIKE MAGNETITE FOR POTENTIAL APPLICATION IN CANCER NANOMEDICINE

Miloš Ognjanović¹, Biljana Dojčinović², Dalibor Stanković^{1,3}, Marija Mirković¹, Sanja Vranješ-Đurić¹, Bratislav Antić¹

e-mail: miloso@vin.bg.ac.rs

1-University of Belgrade, VINČA Institute of Nuclear Sciences-National Institute of the Republic of Serbia, Belgrade, Serbia, 2-Institute of Chemistry, Technology and Metallurgy, National Institute of the Republic of Serbia, University of Belgrade, Belgrade, Serbia, 3-Faculty of Chemistry, University of Belgrade, Belgrade, Serbia

Nanomaterials are intensively researched both from the fundamental aspect due to new properties at the nanoscale, as well as the aspect of their application in many areas of technology. Magnetic nanoparticles (MNPs) are being tested for use in the diagnosis and therapy of diseases. A new field of medicine, Magnetic nanomedicine is primarily based on the application of MNPs as drug carriers, diagnostic agents in Magnetic Resonance Imaging (MRI) and heat generators in magnetic hyperthermia. Among nanoparticles, magnetic nanoplateforms based on iron oxides for cancer diagnosis and therapy (Cancer nanomedicine) are the most researched and clinically tested. This study presents the results of research into the physicochemical properties of iron oxide nanoparticles prepared by the polyol route, as well as their testing for potential applications as agents in magnetic hyperthermia (MH) and radionuclide carriers (vectors) for the diagnosis and therapy of malignant diseases. Multicore iron oxide structures synthesized by the "polyol" method represent clusters of single-core nanoparticles or crystallites. The dimensions of the single core particles are ~13.5 nm, while the nanoflowers formed by clustering are ~25 nm, depending on the applied synthesis parameters. For targeted medical applications, nanoflowers are coated with different ligands in order to increase colloidal stability and biocompatibility. The best results were by coating MNPs with polyacrylic acid (PAA). The multifunctionality of nanoflowers was investigated by measuring their hyperthermic efficiency for applications in magnetic hyperthermia and radiolabeling with diagnostic (^{99m}Tc) and therapeutic radionuclides (¹⁷⁷Lu, ⁹⁰Y). In addition to traditional methods of cancer therapy (surgery, radiotherapy, and chemotherapy), new ways of therapy such as MH are constantly being developed. MH is a therapy based on the property of MNPs that when placed in an alternating (AC) magnetic field, transform the electromagnetic energy of the field into heat. When located inside a tumor, MNPs can locally generate a temperature of 42-46 °C and destroy cancer cells by heat. The hyperthermic efficiency of MNPs is expressed through the Intrinsic Loss Power (*ILP*) parameter. The measured *ILP* was 7.3 nHm²/kg which is considered one of the higher reported values found in the literature for iron oxides. Nanoflowers were radiolabeled with ^{99m}Tc, ¹⁷⁷Lu, and ⁹⁰Y radionuclides. The *in vitro* stability of radiolabeling was investigated. Good *in vitro* stability indicates that the formed radioactive particles can be used simultaneously for bi-modal cancer therapy (MH and radionuclide therapy) or for MH therapy and diagnostics (theranostics), in the case of labeling with ^{99m}Tc.

Keywords Cancer nanotechnology, magnetic nanoparticles, iron oxide, microstructure

SPECIAL COPPER ALLOYS FOR MOULDS IN PRODUCTION OF HIGH-END GLASS PRODUCTS

Boštjan Markoli¹, Iztok Naglič¹, Blaž Leskovar¹, Adam Zaky¹, Žan Kresnik², Andrej Resnik²

e-mail: bostjan.markoli@ntf.uni-lj.si

1-Faculty Natural Sciences and Engineering, University of Ljubljana, 2-OMCO Metals Slovenia d.o.o.

Molds for the glass industry can be made from special copper alloys that have excellent thermal conductivity and resistance to thermal fatigue. The most structurally critical points of the molds have special nickel-based plates either welded or nickel inserts engulfed by a molten alloy. At the end of their service life, the used molds are recycled together with the nickel plates and/or inserts. This means that the composition of recycled molds departs from the original one having a profound influence on the constitution of alloys and thus their properties.

The aim of our work was to characterize the microstructure of five samples of the special copper alloy designated as OMX, containing 15.0-16.5 wt. % Ni, 9.5-10.0 wt. % Al, 7.5-9.0 wt. % Zn and up to 1.25 wt. % Fe, focusing on impurities. The microstructures of the nickel plate and nickel insert and their joints with a special copper alloy of the molds were also investigated to further explain the source of impurities in recycled OMX alloys. Characterization methods used in this work include optical microscopy (LE), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD).

The microstructure of the OMX special copper alloy samples usually consists of equiaxed dendrites of primary phase β -AlNi and binary eutectic (α_{Cu} + β -AlNi). The samples also contained several impurities, especially chromium and iron-rich borides, while complex oxides and lead were also found very frequently. The amount of iron in the borides was found to significantly affect their shape. The chemical composition and microstructure of the nickel plate and nickel insert are quite different. It was also found that borides form at the weld between the nickel plate and the special copper alloy, while aluminum-based oxides form most frequently near the joint between the insert and the special copper alloy.

Keywords: special copper alloys, borides, microstructure, SEM, EDS

QUASICRYSTALS IN Al-Mn-BASED ALLOYS CONTAINING COPPER, CALCIUM AND STRONTIUM

Iztok Naglič¹, Blaž Leskovar¹, Adam Zaky¹, Boštjan Markoli¹, Jovica Stojanović², Željko Kamberović³

e-mail: iztok.naglic@ntf.uni-lj.si

1-University of Ljubljana, Faculty of Natural Sciences and Engineering, Aškerčeva cesta 12, 1000, Ljubljana, Slovenia

2-Institute for Technology of Nuclear and Other Mineral Raw Materials, Franše d' Eperea 86, 11000 Belgrade, Serbia

3-University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, 11000 Belgrade, Serbia

This work deals with the effect of copper, calcium, and strontium on the formation of quasicrystals in Al-Mn-based alloys. The prepared alloys are cast at a temperature of 880 °C in a copper mould with a circular casting cavity of 3 mm diameter. The cooling rates for the casting were estimated to be 970 K/s at the centre of the casting cross-section and 1080 K/s at the surface of the casting using a one-dimensional transient heat conduction analysis.

The microstructure of the alloys produced is visualised by backscattered electron images (BEI) using scanning electron microscopy (SEM). The presence of quasicrystals (QC) is determined by their characteristic morphology and, in some cases, by electron backscatter diffraction (EBSD), which makes it possible to distinguish between different QC phases formed in these alloys. Energy dispersive X-ray spectroscopy (EDS) at an accelerating voltage of 17 kV is used to determine the composition of the prepared alloys and the composition of the QC phases that form in these alloys.

The results confirm that alloying copper with an Al-Mn-based system promotes the formation of decagonal QC, which is already known. The results also suggest that both calcium and strontium promote the formation of QC in these alloys. In addition, it was found that a ternary phase is likely to form as the primary phase at larger calcium contents in the alloys. Since the ternary phase also contains manganese, it limits the possibility of forming QC. Copper was found in quasicrystalline phases in all alloys that contain copper. Strontium, on the other hand, was not found in a quasicrystalline phase despite its presence in the alloy.

Keywords: Casting, Aluminium alloys, Quasicrystals, Microstructure

OPTIMISATION OF AN Al-Mn ALLOY REINFORCED WITH QUASICRYSTALS

Adam Zaky¹, Blaž Leskovar¹, Iztok Naglič¹, Boštjan Markoli¹

e-mail: adam.zaky@ntf.uni-lj.si

¹-University of Ljubljana, Faculty of Natural Sciences and Engineering, Department of Materials and Metallurgy

Ever since the first publications about quasicrystals (QC) in the 1980s, a lot of effort has been put towards the synthesis of new Al alloys with high QC content as it is believed that their presence in the matrix would enhance some mechanical properties (alloy hardness, Ultimate Tensile Strength etc...). Such an alloy could be categorised as a metal matrix composite.

This study builds on previous work conducted by authors on the topic of Al-Mn based alloys. Several alloys from the Al-Mn system with different additions of Si, Cu, Mg were synthesized by gravity casting into a copper mould of length 76 mm and diameter of 5.5 mm.

The goal was to study the influence of the chemical composition on QC formation and to optimise it so as to produce an alloy with improved QC content and no (or only a small fraction) of the β -AlMnSi phase.

The casting for each alloy composition was examined at two locations – in the middle and near the top (roughly 3.5 cm from the bottom and 1.5 cm from the top respectively). The microstructure was examined using a light optical microscope (LOM) as well as a scanning electron microscope (SEM). The chemical compositions of the synthesized alloys were checked using energy dispersive spectroscopy (EDS) using an accelerating voltage of 20 kV. The SEM images were used to determine the fraction of QC content for each alloy using publicly available open-source software – FIJI. Hardness measurements were also performed using a Vickers hardness tester with a load of 1 kg (HV1) and a load time of 15 s.

Our analysis indicates that we managed to increase the QC content in our optimised alloy almost two-fold - the original alloy contained roughly 11 % of QC phases whereas our optimised compositions contained around 20 %. Hardness measurements varied depending on the phases that were being analysed – generally, it increased with an increasing fraction of QC phase.

Keywords: Aluminium alloys, Quasicrystals, Metallography, Vickers hardness

QUALITY ASSESSMENT OF THE $\text{AlSi}_9\text{Cu}_3(\text{Fe})$ ALLOY WITH REGARD TO RECYCLING RATIO

Zdenka Zovko Brodarac¹, Franjo Kozina¹, Davor Stanić², Martin Folta³

e-mail: zovko@simet.unizg.hr, fkozin@simet.unizg.hr

1- University of Zagreb Faculty of Metallurgy, Sisak, Croatia

2-CIMOS – CIMOS – TCH Group P.P.C. Buzet d.o.o., Buzet, Croatia

3- Škoda Auto Vysoká Škola o.p.s., Mladá Boleslav, Czech Republic

The most common aluminum alloy in automotive industry is the $\text{AlSi}_9\text{Cu}_3(\text{Fe})$ alloy due to relatively high mechanical properties, fluidity and resistance to hot cracking. The mechanical properties of aluminum cast alloys are strongly dependent from the microstructure developed on the base of chemical composition and casting process with the emphasis on solidification sequence. Green production is an imperative in nowadays industry. Usage of secondary charge material comprehend to the green production certification with 0% CO_2 footprint when own return has been used. Maintaining the alloy quality with regard to functional properties while promoting green production makes economical production imperative. In this research, modeling of the solidification sequence in equilibrium and non-equilibrium conditions was carried out and compared with the obtained microstructure for a conventional $\text{AlSi}_9\text{Cu}_3(\text{Fe})$ alloy and alloy made of 100% return charge material. Modelled equilibrium phase diagram enables solidification sequence prediction which can be easily confirmed using metallographic analysis. Modeling of solidification sequence revealed some differences in solidification path. Microstructural investigation of obtained samples confirms the presence of following phases: primary aluminum evolution (α_{Al}), iron phases (Al_5FeSi , $\text{Al}_x(\text{Fe},\text{Mn})_y\text{Cu}_w\text{Si}_z$), primary eutectic phase ($\alpha_{\text{Al}}+\beta_{\text{Si}}$) as well as other intermetallic phases as $\text{Al}_7\text{Cu}_2\text{Mg}$, $\text{Al}_5\text{Mg}_8\text{Si}_6\text{Cu}_2$, Al_2Cu and $\text{Al}_{15}\text{Cu}_2\text{Mg}_8\text{Si}_6$. The comparison of the obtained results indicates a lowering of the main alloying element Si and thus an influence on the displacement of the solidification sequence equilibrium. Despite the observed differences in the solidification path, the comparison of the microstructures for both cases (conventional and 100% return alloy) does not reveal a degradation in the quality of the alloy. The quality assessment thus indicates that production with 100% recycled $\text{AlSi}_9\text{Cu}_3(\text{Fe})$ charge material meets all functional properties in the automotive industry.

Keywords: recycling potential, $\text{AlSi}_9\text{Cu}_3(\text{Fe})$ alloy, solidification sequence, modeling, microstructure

PREDICTING THE MODULUS OF ELASTICITY OF BIOCOMPATIBLE TITANIUM ALLOYS USING MACHINE LEARNING

Gordana Marković¹, Vaso Manojlović¹, Miroslav Sokić¹, Jovana Ružić², Dušan Milojkov¹, Aleksandra Patarić¹

e-mail: g.markovic@itnms.ac.rs

1-Institute for Technology of Nuclear and Other Mineral Raw Materials, 11000 Belgrade, Serbia,

2-Faculty of Technology and Metallurgy, University of Belgrade, 11000 Belgrade, Serbia

Titanium alloys find their application in various fields, among others in biomedical engineering. The properties characteristic of these alloys are mechanical and corrosion resistance combined with good biocompatibility. What distinguishes this group of materials, compared to other materials used for the same purpose, is the modulus of elasticity. A database containing about 300 titanium alloys free of toxic elements was created. The influence of different factors (such as the masses of alloy elements, density, specific heat, etc) on the modulus of elasticity is predicted using four methods: support vector machine, XGBoost, Neural Network, and Random Forest. The Random Forest method's mean absolute error (MAE) (7.33 GPa) enters the range of experimentally obtained absolute errors in the literature (up to about 11 GPa). A strong correlation ($R^2=0.72$) was found between experimental and predicted data. Finally, the modulus of elasticity areas of specific alloying elements was determined, which can be used to design new biocompatible titanium alloys in the future.

Keywords: titanium alloys, modulus of elasticity

CAVITATION EROSION RESISTANCE OF SOME ENGINEERING MATERIALS

Tatjana Volkov-Husović¹, Sanja Martinović², Ana Alil², Milica Vlahović²

e-mail: tatjana@tmf.bg.ac.rs

1 - University of Belgrade, Faculty of Technology and Metallurgy, Belgrade, Serbia

2 - University of Belgrade, Institute of Chemistry, Technology and Metallurgy - National Institute of the Republic of Serbia, Belgrade, Serbia

The cavitation erosion behavior of engineering materials is very important for various applications, such as industrial/military/power station equipment and parts, such as pump impellers, delicately balanced high-speed propellers, and turbine blades. Cavitation erosion could cause pits and fractures, which usually lead to a potential risk of failure, as well as potential injury for workers and others; loss of revenue, due to equipment downtime, and the extra costs of failure analysis, repair, and replacement of parts.

In this paper cavitation erosion of different engineering materials will be investigated. Selected materials are metals (austenitic stainless steel 316L and CuAlNi shape memory sample), ceramics (cordierite), and polymer composite (epoxy-carbon composite). The cavitation erosion behavior of selected samples will be compared using mass loss results and image analysis results applied on pits (damage) analysis in order to calculate the damage level.

Keywords: cavitation erosion, steel, ceramics, composite material, image analysis

PRINCIPAL COMPONENT ANALYSIS OF MORPHOLOGICAL DESCRIPTORS FOR ASSESSMENT OF SURFACE DEFECTS INDUCED BY EXTREME CONDITIONS

Sanja Martinović¹, Ana Alil², Aleksandar Savić³, Dragomir Glišić⁴, Dragana Živojinović⁴, Tatjana Volkov-Husović³

e-mail: s.martinovic@ihtm.bg.ac.rs

1-University of Belgrade, Institute for Chemistry, Technology and Metallurgy, Belgrade, Serbia,

2-University of Belgrade, Innovation Center of the Faculty of Technology and Metallurgy, Belgrade, Serbia,

3-University of Belgrade, Faculty of Civil Engineering, Belgrade, Serbia,

Implementation of statistical analysis for monitoring the formation and propagation of defects that occurred due to exposure of the material to thermal shock and cavitation is presented in this study. Results of two groups of materials are shown: refractory concrete sintered at three temperatures and ceramic materials based on cordierite and zircon. Alumina-based refractory concretes were subjected to thermal stability test, while the surface morphological changes regarding the defects were monitored after a certain number of thermal shock cycles. Cordierite and zircon based materials were initially subjected to cavitation testing while after specified time periods, morphological changes in the surface defects were analyzed. Besides the standardized methods, non-destructive testing of materials was applied for monitoring the surface changes. Cameras, microscopes, and numerous software enable recording images of sample surface with appropriate magnifications as well as monitoring and quantifying the surface damage degree with various image analysis tools. Thus, this approach offers the quantification of selected morphological descriptors regarding the defects. Obtained values were then subjected to pattern recognition method, the principal component analysis that can provide information on extracted morphological descriptors by describing the main difference among observed defects and identifying variations among them. As a result, a better insight into surface degradation that occurs during exposure to extreme conditions was obtained. The multivariate analysis provided grouping and thus reduction of the descriptors while maintaining the highest percentage of variance unchangeable. More precisely, the analysis extracted the most informative descriptors that should be observed for monitoring changes in surface defects and determining a degradation mechanism.

Keywords: Thermal shock, Cavitation erosion, Surface defects, Morphological descriptors, Principal component analysis (PCA)

CIRCULAR ECONOMY AND DIGITALIZATION IN CAST PRODUCTION

Radomir Radiša¹, Srećko Manasijević¹, Nedeljko Dučić², Aleksandar Jovičić²

e-mail: radomir.radisa@li.rs

1-Lola Institute Ltd, Kneza Visislava 70a, 11030 Belgrade, Serbia,

2-Faculty of Technical Sciences Cacak, Svetog Save 65, 32102 Cacak, Serbia,

Foundries are known to be enormous consumers of energy since the production of only a ton of castings requires an average of 2,000 kilowatt hours of energy. An adequate implementation and usage of modern software tools for the design and simulation of the casting processes enables engineers in foundries to conduct precise and optimum technology-based projections of the casting system prior to starting the casting process. Energy efficiency and closely correlated environmental protection benefits, as complemented by described potential savings of energy and materials that are unquestionably achievable through the use of digital tools, make the acquisition and inclusion of these tools in foundries mandatory. This paper proposes an innovative casting technology for each new casting, verified on a computer. Such an approach will enable the application of the circular economy in the foundries of Serbia in the true sense.

Keywords: Digital tools; energy; circular economy casting; Foundry

**STUDY OF THE CHANGES IN MECHANICAL PROPERTIES OF
THE COPPER-ZIRCONIUM ALLOYS INFLUENCED BY MINOR BORON ADDITION**

Jovana Ružić¹, Marko Simić¹, Dušan Božić¹, Nikolay Stoimenov², Stanislav Goshev², Jelena Stašić¹

e-mail: jruzic@vin.bg.ac.rs

1-Department of Materials, "Vinča" Institute of Nuclear Sciences - National Institute of the Republic of Serbia, University of Belgrade, PO Box 522, 11001 Belgrade, Serbia,

2-Institute of Information and Communication Technologies, Bulgarian Academy of Sciences, Sofia, Bulgaria

Copper-zirconium alloys with the minor addition of boron were manufactured using powder metallurgy, combining mechanical alloying, hot pressing, and sintering. The influence of boron on the mechanical properties of the Cu-Zr alloys was studied on Cu-1Zr (wt.%) and Cu-1.1Zr-0.3B (wt.%) systems. Scanning electron microscopy (SEM), laser nanoparticle sizer, computed tomography, and X-ray diffraction were employed for the observation of morphological and structural *changes* in the copper microstructure during manufacturing, as well as the development of CuZr phase and ZrB₂ particles. Based on SEM and X-ray diffraction analysis, it can be seen that finely dispersed reinforcing ZrB₂ particles in copper matrix occur only after the hot pressing process, and not during mechanical alloying. Mechanical properties of the copper-zirconium alloys were investigated using nanoindentation and microhardness tests. It was shown that the presence of a small boron amount, less than 3 wt.%, increases dislocation density in Cu-Zr-B powder mixture with increasing the duration of mechanical alloying. Moreover, analysis of the obtained results revealed that the addition of boron induces a higher hardening effect in Cu-Zr-B alloy compared to Cu-Zr alloy, which results in more stable hardness values during thermomechanical treatment.

Keywords: Cu-Zr-B alloy, Mechanical alloying, Hot pressing, Hardness

UNDERSTANDING THE ELECTRONIC STRUCTURE AND TRANSPORT PROPERTIES OF A-SITE CO-SUBSTITUTED SrTiO_{3-δ} CERAMICS WITH ENHANCED CONFIGURATION ENTROPY

Nikola Kanas¹, Reshma Madathil², Annu Sharma³, Vladimir Srdić⁴, Bojan Miljević⁴, Srđan Rakić⁵,
Subramshu Bhattacharya³, Stevan Armaković⁵

e-mail: nikola.kanas@biosense.rs

1-Institute BioSense, University of Novi Sad, 21000 Novi Sad, Serbia,

2-Department of Chemistry, University of Oslo, FERMIØ, 0349 Oslo, Norway,

3-Nanofunctional Materials Technology Centre, Department of MME, IIT Madras, India,

4-Department of Materials Engineering, Faculty of Technology, University of Novi Sad, 21000 Novi Sad, Serbia,

5-Department of Physics, Faculty of Sciences, University of Novi Sad, 21000 Novi Sad, Serbia

Since SrTiO₃ is interesting due to its versatile electronic properties ranging from dielectric to semiconducting behavior, the effect of systematic co-substitution of various A-site cations on the electronic structure and transport properties of oxygen deficient SrTiO_{3-δ} bulk ceramics was analyzed.

The materials were synthesized by reverse co-precipitation method and densified by spark plasma sintering (SPS) in vacuum, followed by a post-thermal treatment in reducing atmosphere (5% H₂ in Ar). The obtained ceramics possess different microstructures, revealed by Scanning Electron Microscopy (SEM), and all of them were characterized in terms of electrical conductivity (σ_{el}), Seebeck coefficient (S) and ionic conductivity (σ_{io}). DFT calculations were applied to obtain information on band structures, the projected density of states (PDOS) and electron density differences (EDD). Band structures indicated that the oxygen deficiency essentially changed the conductive nature of the studied materials by shifting the Fermi level to the conductive zone, while PDOS plots helped determining the role of each element located at the A site. The electronic structure is affected to the greatest extent in the cases when Nd and Pr are present at the A site. EDD plots indicated the charge distribution near Ti atoms whose oxidation state was manipulated by oxygen deficiencies. When Nd was present at the A sites, the physical shift of other oxygen atoms is significantly higher, thus affecting the oxidation state of Ti.

This work reports on the relation between the degree of entropy at A-site followed by the influence of oxygen vacancies and the electronic transport properties in the selected perovskites.

Keywords: Co-substitution, oxygen deficiency, conductivity, electronic bandgap, Density Functional Theory (DFT)

ELECTROCHEMICAL SENSORS FOR DETECTION OF BISPHENOLS

Jelena Vujančević¹, Špela Trafela¹, Neža Sodnik^{1,2}, Zoran Samardžija¹, Kristina Žagar Soderžnik^{1,3}

e-mail: jelena.vujancevic@ijs.si

1-Department for Nanostructured Materials, Jožef Stefan Institute, Jamova cesta 39, SI-1000 Ljubljana, Slovenia

2-University of Ljubljana, Faculty of Chemistry and Chemical Technology, Večna pot 113, SI-1000 Ljubljana, Slovenia

3-Jožef Stefan International Postgraduate School, Jamova cesta 39, 1000 Ljubljana, Slovenia

The endocrine-disrupting chemicals (EDCs) are chemicals of very high concern that have hazards with serious consequences on human health. It influences on development of metabolic disorders, reproduction and respiratory problems. They can be found in our everyday life, from food, and personal care products to medical devices, dental products, special lenses and baby drink cans. Among several EDCs, banned Bisphenol A (BPA) and his substitute Bisphenol S (BPS) have attracted attention due to high usage during the manufacturing process for water and food packaging, in the production of epoxy resins, lacquer coating and can even be found in receipt. Due to that, there is a need for the fast, reliable and commercial detection of Bisphenols in everyday life. The gold standard for the detection of Bisphenols is chromatography and enzyme-linked immunosorbent assay, expensive and robust methods. Electrochemical sensors are a new approach to the detection of EDCs in very small quantities in complex environments. The aim of this research was to study commercial screen-printed electrodes (SPEs) as receptor elements in electrochemical sensors for the detection of BPA and BPS. Scanning electron microscopic (SEM) and Fourier transform infrared spectroscopy (FT-IR) were employed for examining the surface of the SPEs working electrodes. SPE electrodes showed very good voltammetric responses toward BPA and BPS oxidation with linear ranges between 0.5 and 50.0 μM and lower limits of detection of 0.15 μM and 0.37 μM , respectively.

Keywords: electrochemical sensor, bisphenol S, carbon screen printed electrode, cycle voltammetry, electrochemistry.

**ANTICORROSIVE IMPROVEMENTS OF WATERBORNE POLYURETHANE
COATINGS USING EGGSHELLS BIOWASTE
FOR MILD STEEL IN ARTIFICIAL SEAWATER**

Wan Mohamad Ikhmal Wan Mohamad Kamaruzzaman¹, Muhamad Syaizwadi Shaifudin¹,
Nursabrina Amirah Mohd Nasir¹, Nur Aiman Syafiq Mohd Hamidi¹, Nusaibah Yusof¹, Azila Adnan¹, Lee Oon Jew¹,
Wan Mohd Norsani Wan Nik², Mohd Sabri Mohd Ghazali¹

e-mail: ikhmal007@gmail.com

1-Faculty of Science and Marine Environment, University Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia,
2-Faculty of Ocean Engineering Technology and Informatics, University Malaysia Terengganu, 21030 Kuala Nerus, Terengganu,
Malaysia

Eggshells (ES) waste is produced daily globally due to massive consumer demands. Even though several studies have highlighted the waste's excellent properties, most are discarded without any beneficial application. Hence, to further highlight the potential application of ES waste, the study was performed to emphasize its usage in the coatings industry. This study loaded the ES waste as an effective filler in waterborne polyurethane coatings. The coatings were applied on mild steels and exposed to a corrosive medium, artificial seawater, for 20 days. Different characterizations were performed, including water contact angle measurement, electrochemical impedance spectroscopy (EIS) and scanning electron microscopy (SEM). Based on the findings, the increment of ES concentrations until 20 wt.% (P4) produced the best efficiency in reducing the corrosion rate of mild steels. Moreover, the surface of P4 coatings immersed for 20 days was smooth, indicating low defects and superior anticorrosive behavior.

Keywords: Corrosion; Eggshells; Waterborne polyurethane; Mild steel; Seawater

GEOPOLYMERS BASED ON FLY ASH FROM THE STANARI THERMAL POWER PLANT

Nadira Bušatlić¹, Ilhan Bušatlić¹, Dženana Smajić-Terzić¹

e-mail: ilhan.busatlic@unze.ba

1-Faculty of Metallurgy and Technology, University of Zenica, Bosnia and Herzegovina

The biggest global problem today is environmental pollution. The environment is polluted by the release of various gases and small particles into the atmosphere, which are the result of the production of various materials.

During the last 20 years, geopolymers, which are also known as mineral polymers or inorganic polymers, have attracted a lot of attention as a promising new form of inorganic polymer material that could be used as a replacement for conventional or ordinary Portland cement (OPC), for plastics, and many other mineral-based products. The development of geopolymer cement is an important step towards the production of environmentally friendly cements.

In this paper, geopolymer samples based on fly ash from the Stanari thermal power plant were tested. Fly ash from the Stanari thermal power plant, 12M NaOH solution and commercial water glass were used as materials. The samples were made with an alkali to fly ash ratio of 0.8. The $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio ranged from 2; 2.5 and 3. The samples were thermally treated at geopolymerization temperatures of 60, 70 and 80 °C. After that the compressive strengths of the samples were tested after 2, 7 and 28 days.

Keywords: geopolymers, environmental pollution, industrial waste, compressive strength, cement

THE OPTIMIZATION OF METALS AND METALLOIDS EXTRACTION FROM FLY ASH

Milica Stojković¹, Sofija Dedić¹, Maja Đolić¹, Mirjana Čujić², Vladimir Pavićević¹,
Mirjana Ristić¹, Aleksandra Perić-Grujić¹

e-mail: mstojkovic@tmf.bg.ac.rs

1-Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4
2-Vinča Institute of Nuclear Sciences, University of Belgrade, 522 P.O. Box, Serbia

One of the byproducts of burning coal in thermal power plants is fly ash, which large quantities deposited in landfills can lead to soil and groundwater pollution. Fly ash contains many elements in its composition, including heavy metals and metalloids whose mobility poses a hazard to the environment and human health due to its toxicity, carcinogenicity, persistence, high solubility, mobility, etc. On the other hand, the complex structure allows its usage for numerous purposes. Most often it is reused for various applications such as the production of concrete and other construction materials. However, in Serbia, where electricity is mainly obtained by coal combustion, the percentage of reuse is at a very low level. Scientific research in the field of "alternative mining" is significant, where heavy metals and rare earth elements would be recovered from waste materials such as fly ash using the various extraction methods. Representative samples of fly ash used in this work were taken from the electrostatic precipitators of the thermal power plant in Kostolac. The aim of this paper is to determine the optimal extraction process of the examined elements as well as the assessment of their potential mobility in the environment. The following metals and metalloids were analyzed: V, Cr, Mn, Co, Ni, Cu, Zn, As, Cd, and Pb. In the experimental work, two media were used for the extraction process, sulfuric acid in two concentrations (1M and 5M) and acetic acid in a concentration of 5M. Two sets of experiments were done; in the first set the fly ash was immersed immediately in acids, and in the second set, this process was preceded by the addition of sodium hydroxide (NaOH), which effectively destroys the alumina-silicate matrix. Several different parameters were also monitored to determine the optimal conditions for both sets of experiments, namely: ratio of fly ash and acid/acid mixture (1:5 and 1:30), temperature (25 and 45°C), duration of the process (4 and 24 h), with and without stirring. Determination of the content of heavy metals and metalloids was done by mass spectrometry with induced coupled plasma (ICP-MS). For the geometric mean of concentration of all elements (M_{tot}), the solid: liquid ratio proved to be the most important parameter, while for all other elements roasting is the least significant. For the extraction Pb - H₂SO₄, temperature and time are significant, for Cd only H₂SO₄ is significant, for As the acetic acid, ratio and time are significant, for Zn, Cu, Co, Mn, and V only the ratio is significant, for Ni the ratio and time, and for Cr, in addition to the ratio and time, there is also the temperature.

Keywords: fly ash, extraction, leaching, (heavy) metals, metalloids

CRYSTALLIZATION CONTROL IN FOAM GLASS PREPARATION FROM CONTAINER GLASS WASTE

Sonja Smiljanić¹, Uroš Hribar¹, Matjaž Spraitzer¹, Jakob König¹

e-mail: sonja.smiljanic@ijs.si

¹-Advanced Materials Department, Jožef Stefan Institute, Jamova cesta 39, 1000 Ljubljana, Slovenia

Waste glass is commonly used as a raw material in foamed glass production. The use of container glass in foaming process is often interfered with its tendency toward crystallization. Glass crystallization is undesirable in foamed glass production, since it can be detrimental for the properties and the quality of the final product. We studied the influence of different types of additives (foaming agents, flux agents, crystallization inhibitors and nucleating agent) on the crystallization of waste container glass and properties of the sintered samples. We found that waste container glass manifests complex crystallization, which can be inhibited with the addition of fluxing agents (B_2O_3 and borax) and in their combination with Al_2O_3 . Quantitative XRD analysis confirmed that the crystalline content is significantly decreased to a value below 15%. The thermal conductivity of the glass can be importantly decreased by the inhibition of crystallization and careful selection of the additives. We prepared foamed glass samples from container glass cullet and foaming/oxidizing couple (carbon/ Mn_3O_4) with the addition of different amounts and combinations of the crystallization inhibitors, fluxing agents (B_2O_3 and borax), their combination with Al_2O_3 , and phosphates under oxygen free atmosphere. We determined the densities, porosity, crystallinity and thermal conductivity of foamed samples. By decreasing the content of the fluxing agent densities of the foamed samples were decreased. With the addition of phosphates to the mixtures crystalline content and apparent densities in the foamed samples were decreased. We obtained foamed samples with thermal conductivity from 56 to 66 mW m⁻¹K⁻¹.

Keywords: Foam Glass, Crystallization, Thermal conductivity

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BENTONITE CLAYS AS AFLATOXIN B₁ ADSORBENTS

Marija Marković¹, Aleksandra Daković¹, Danijela Smiljanić¹, Milena Obradović¹, Milica Ožegović¹

e-mail: m.markovic@itnms.ac.rs

¹Institute for Technology of Nuclear and Other Mineral Raw Materials, 86 Franchet d'Esperey St., 11000 Belgrade, Serbia

Aflatoxins (AFs), a class of mycotoxins produced by fungal species, mainly *Aspergillus flavus* and *Aspergillus parasiticus*, are present in feed or food. AFs, until a few years ago, have not been recognized as contaminants of big concern for primary production in Europe. However, recently, situation is different due to the climate change and in the years 2003 and 2012 in Italy and South Europe, the significant contamination of corn with AFs was reported. There are more than twenty different forms of naturally occurring AFs, among which aflatoxin B₁ (AFB₁) is the most toxic. AFB₁ is acutely and chronically toxic to humans and animals, causing liver damage, liver cirrhosis, induction of tumors, and teratogenic effects. Various techniques such as physical treatment, chemical detoxification and biological degradation have been adopted to remove, destroy or reduce AFB₁, and subsequently protect animal and human health. At present, adsorption approach is considered to have the most potential in treating mycotoxins contaminated feed. Among various adsorbents, clay minerals such as bentonites have drawn global attention due to their high efficiency, natural abundance, and low cost. Bentonites are composed primarily of montmorillonite which is dioctahedral 2:1 layer aluminosilicate. The mineralogical, chemical, and physico-chemical characteristics of bentonites have an influence on adsorption of AFB₁. Recently, it was reported that structural configuration of the octahedral sheet in bentonite clays also affects AFB₁ adsorption. The *cis*-vacant bentonites showed higher AFB₁ adsorption than the *trans*-vacant bentonites. It is obvious that the bonding of AFB₁ by bentonites is very complex and influenced by many factors, and so far exact mechanism relevant to AFB₁ adsorption by these clays is not yet fully defined.

Bentonite, in the form of dioctahedral montmorillonite, is authorized in the European Union (European Union Regulation 1060/2013) as a technological additive - substance for the reduction of the contamination of feed by AFs, since 2013. According to this Regulation, bentonite should contain $\geq 70\%$ smectite (dioctahedral montmorillonite), $< 10\%$ opal and feldspar, and $< 4\%$ quartz and calcite (based on XRD analysis). The adsorption capacity of the specific bentonite for AFB₁ needs to be above 90%.

In this study two bentonites - bentonite from deposit Beretnica in the Republic of Serbia and commercial bentonite supplied from Fagron Co, were tested as adsorbents for AFB₁. Adsorption of AFB₁ was studied according to the method described in the European Union Regulation: in buffer solution at pH 5, AFB₁ concentration 4.0 mg/L and with 0.02% (w/v) of each adsorbent. Results were compared with results of AFB₁ adsorption by previously studied gray clay from the Šipovo deposit in Bosnia and Herzegovina. A single adsorption experiment showed that all three bentonites possess high efficiency for AFB₁ (over 90%). The adsorption of AFB₁ by three clays was further followed with the same initial concentration of AFB₁ and with different amounts (0.2, 0.1, 0.05, and 0.02 g/L) of each adsorbent in suspension, at pH 3. It was found that adsorption of AFB₁ increased with increasing the amount of each adsorbent.

The structure of the octahedral sheet of bentonites can be determined by the dehydroxylation peak temperature obtained by thermal analysis (DTA or DSC). *Cis*-vacant bentonites dehydroxylate around 650-700°C, while dehydroxylation of *trans*-vacant bentonites occurred at around 500-550°C. Thermal analysis of bentonites performed at NETZSCH STA 449 F5 Jupiter instrument showed that Beretnica bentonite and commercial bentonite are considered as *cis*-vacant bentonite, while previously studied gray clay is *trans*-vacant bentonite. Although gray clay is *trans*-vacant bentonite and should have lower adsorption of AFB₁, results of adsorption of AFB₁ by three bentonites showed that among all investigated samples, the lowest adsorption of AFB₁ was achieved with commercial bentonite which is *cis*-vacant bentonite. Since the adsorption of AFB₁ by bentonites is very complex, further experiments are needed in order to define differences in the adsorption of this toxin by three clays (if any).

Keywords: mycotoxins, aflatoxin B₁, bentonites, adsorption

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Poster presentations

RAW KAOLINITIC-ILLITIC CLAYS FOR THE PRODUCTION OF REFRACTORY CERAMICS

Milica Vidak Vasić¹, Lidija Radovanović², Zagorka Radojević¹

e-mail: milica.vasic@institutims.rs

1-Institute for Testing of Materials IMS, Bulevar vojvode Mišića 43, 11000 Belgrade, Serbia,

2-Innovation Centre of the Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4, 11000 Belgrade, Serbia,

The study makes a unique effort to characterize all the necessary characteristics of refractory clays and products fired at temperatures between 1100 and 1300 °C on a laboratory level. For 19 composite samples from the recently opened reservoir, thorough instrumental and ceramic-technological data are presented. The potential for using Serbian raw refractory clays from the recently discovered deposit is investigated. The composites had a variety of qualities and varied in composition, but they were majorly composed of kaolinite and illite and had a significant share of fluxes. The samples had high quantities of clay minerals and organic matter, which is seen as the most intense mass loss in the 400–600 °C range. Low-duty and high-duty refractories could be distinguished by their refractoriness over 1581 °C. This parameter was mostly influenced by the content of alumina and kaolinite within the materials. Hydraulically-pressed ceramic tiles, following the firing stage, showed water absorption was between 9.02–1.42 % and a modulus of rupture of 16.5 – 53.6 MPa. Color is determined by spectrophotometer and correlated to the composition of the initial clay and fired products. For the lining of ceramic and glass furnaces, the majority of these clays can be used as natural refractory materials. Some of the samples' organic content has a detrimental effect on the fast-firing procedure, since the occurrence of the entrapped air (bloating).

Keywords: Kaolinitic-illitic clays; Refractory ceramics; Ceramic and glass furnaces lining

THE ADVANTAGES OF USING PELLETIZED GYPSUM COMPARED TO POWDERED GYPSUM

Vladimir Jovanović¹, Dejan Todorović¹, Branislav Ivošević¹, Dragan Radulović¹, Mladen Bugarčić¹, Sonja Milićević¹

e-mail: v.jovanovic@itnms.ac.rs

1- Institute for technology of nuclear and other mineral raw materials, 86 Franchet d'Esperey St, 11000 Belgrade, Serbia

Pelletized gypsum products were the standard for agricultural gypsum for many years, but as growers look to maximize their return on investment, they are increasingly turning toward pelletized gypsum – a premium alternative. Pelletized gypsum offers several significant advantages over other forms of gypsum such as pulverized or ultra-fine gypsum. In the first line, production of pelletized gypsum is dust-free, and product formulations are also improved. Finally, transportation of this formulation of gypsum is simplified as well as field spreading. This has led to increased grower adoption and a greater demand for fertilizer and soil amendment producers to offer gypsum in a pelletized form. Producers are also finding that they stand to gain a few benefits from offering a pelletized product as well. In the first order it is the price of this product but also it gives satisfying benefits to the cultivated plants since the calcium and sulfur release is limited. The aim of this work is to give the insight of actual approach in pelletized gypsum production technology in comparison with technology of pulverized gypsum.

Keywords: fertilizer, production management, agitation agglomeration

JAROSITE FROM ZINC HYDROMETALLURGY: THERMODYNAMIC AND KINETIC APPROACH

Sanja Jevtić¹, Vaso Manojlović¹, Milisav Ranitović², Nataša Gajić², Marija Štulović², Željko Kamberović¹

e-mail: sanja@tmf.bg.ac.rs

1-Faculty of Technology and Metallurgy, University of Belgrade, Belgrade, Serbia,

2-Innovation Centre of the Faculty of Technology and Metallurgy Ltd, University of Belgrade, Belgrade, Serbia

Jarosite is a hazardous waste whose composition is determined by the origin of the raw materials for the hydrometallurgical zinc production. In examined sample the dominant phases are hydrated ammonium jarosite $\text{NH}_4\text{Fe}_3(\text{SO}_4)_2(\text{OH})_6 \cdot \text{H}_2\text{O}$ and zinc-ferrite ZnFe_2O_4 . The thermal decomposition of an industrial sample of jarosite residue was studied using thermogravimetric analysis. The obtained data were analyzed using the Kissinger-Akahira-Sunose method to determine the decomposition process's activation energy in the air and nitrogen flow. The experiments were carried out at different heating rates of 5, 10, 15, and 20 °Cmin⁻¹ in the temperature range of 25 to 850 °C. The results showed that the sample lost mass in two steps. Deammonization, which is the first step in the decomposition of jarosite, takes place faster and at slightly lower temperatures when heated in a flow of nitrogen than in air. The maximum rate of decomposition increases with the increasing heating rate in both investigated cases. The maximum rate of deammonization in the nitrogen flow is 5.0, compared to 4.1 %min⁻¹ in air. In the second step, mass loss is attributed to desulfurization, which takes place faster in air. The maximum decomposition rate is 7.6 compared to 6.8 %min⁻¹ in nitrogen, which can be explained by the oxidation of sulfur dioxide in the presence of oxygen. This was also confirmed by determining the activation energy using the KAS method, which is higher in the air flow than in nitrogen and amounts to 235.4 and 208.8 kJmol⁻¹, respectively.

Keywords: jarosite, metal recovery, thermal decomposition, thermodynamic analysis, activation energy

**ANALYSIS OF EFFECTIVENESS OF BOTTOM STIRRING AFTER THE
MODERNIZATION ON BASIC OXYGEN FURNACE NO. 2 IN HBIS GROUP SERBIA**

Nenad Milosavljević¹, Tamara Ristić¹, Goran Jevtić¹, Mijajlo Jauković¹

e-mail: nmilosavljevic@hbisserbia.rs

1-HBIS GROUP Serbia Iron & Steel doo Belgrade

Factory HBIS GROUP Serbia Iron & Steel d.o.o. Belgrade is located in Smederevo, Radinac area, 10 km from the Danube coast. Projected max. capacity of the factory is 2.2 million metric tons of steel, including hot rolled and cold rolled sheets and plates, as well as tin plates – tinned steel is produced in Šabac. The newest modernization in our company is BOF no. 2, which contains bottom stirring of Ar and N₂ and the possibility of slag splashing with nitrogen, through the lance tip, after the tapping of Steel from the converter. The implementation of new technology was a challenge for us because we encountered it for the first time. The main goals of this modernization were obtaining lower dissolved oxygen content in steel with achieving targeted carbon at the end of the blowing process, which gives us an optimal oxidation degree of steel [%C] [%O] and reduced use of Al as a deoxidizer, higher yield, achieving low phosphorus content in steel at the end of blowing, an increased lifetime of the lining with the slag splashing and less refractory mass consumption for repairing of the lining. This analysis is based on a period of 7 months from the beginning of the campaign with new refractory lining, and the research is done on 3150 heats in this period. During the examination of the process, we came to the conclusion that certain parameters met our expectations, while certain results were not at the expected level.

Keywords: BOF, bottom stirring, slag splashing, de-phosphorization

COMPARING PURIFICATION METHODS OF CRUDE BIODIESEL OBTAINED BY TRANSESTERIFICATION OF VARIOUS OILY FEEDSTOCKS OVER CALCIUM-BASED CATALYSTS

Ivan Stojković¹, Vladimir Pavićević², Ivana Banković-Ilić³, Vlada Veljković^{3,4}

e-mail: istojkovic@tmf.bg.ac.rs

1-Innovation Center, Faculty of Technology and Metallurgy, Belgrade, Serbia,

2-Faculty of Technology and Metallurgy, University of Belgrade, Belgrade, Serbia,

3-Faculty of Technology, Leskovac, University of Niš, Serbia,

4-Serbian Academy of Science and Arts, Belgrade, Serbia

The purification methods of crude biodiesel obtained by transesterifying various oils catalyzed by calcium-based catalysts, like calcium oxide and calcium diglyceroxide, are compared by analyzing their performances regarding oily feedstocks in the so far-reported investigations. Therefore, the focus is on the decalcification of crude biodiesel obtained from various vegetable oils. The comparison includes dry washing, membrane extraction, complexation, precipitation, ion-exchanging precipitation, simultaneous reaction and ion-exchanging-caused precipitation with water washing, solvent-aided crystallization, and two-step processes. Crude biodiesels originated from vegetable oils (soybean, sunflower, palm, coconut, and jatropha), waste frying oil, and animal fats (pork lard and category-1 animal fat). The advantages and disadvantages of different purification methods are highlighted. The comparison showed that the wet washing methods were unsuitable for crude biodiesel decalcification. However, water washing after the pre-purification step consisting of dry washing with a resin, membrane extraction, or ion exchange-based precipitation resulted in a better crude biodiesel refinement. No systematic study on the combined effect of the type of oily feedstock and the purification method on the fuel properties of the purified biodiesel has been carried out. In contrast to dry washing and membrane extraction, two-step processes combining two purification methods were effective in calcium removal from crude biodiesels obtained from sunflower and waste cooking oils. In conclusion, crude biodiesel purification methods should be improved to increase their purification efficiency, and the proper method should be selected based on careful analysis and experimentation.

Keywords: biodiesel, purification, decalcification, wet washing, dry washing

THE EFFECT OF THE PLASTIC TO COKE RATIO ON THE MOST CRITICAL PROCESS PARAMETERS IN A BLAST FURNACE

Miloš Marković¹, Darija Đorđević¹, Vaso Manojlović¹, Aleksandar Vasić¹, Željko Kamberović¹

e-mail: 20190208@estudent.tmf.bg.ac.rs

1-University of Belgrade, Faculty of Technology and Metallurgy, 4 Karnegijeva St., 11120 Belgrade, Serbia

Coke is the primary source of heating and reduction in the iron and steel industry, but a major contributor to air pollution due to high carbon dioxide emissions. This study investigates an alternative reducing agent, hydrogen-rich plastic material, derived from the growing amounts of municipal waste in landfills. The increased hydrogen content in the reducing agent alters the thermodynamic and kinetic conditions of the chemical reactions, which can potentially enhance productivity, reduce material costs, and minimize the carbon footprint. Using the Blast Furnace Calculation (BFC) software, which has been employed in the Smederevo ironworks for over two decades, we simulated thermodynamic conditions, material, and energy balances to assess the impact of the coke-to-plastic ratio on the specific production coefficient, total material costs, and the degree of utilization of blast furnace gas. Our findings show that as the coke-to-plastic ratio increases from 2 to 10, the specific productivity of the blast furnace increases from 1.6 to 2.1. However, the total material costs increase from \$282 to \$302 per ton of produced iron, and the utilization rate of blast furnace gas decreases from 56.6% to 50.9%. These results highlight the potential benefits and trade-offs of using hydrogen-rich plastics as an alternative reducing agent in the iron industry. On the other hand some limitations, like chlorine and fluorine content, must be considered for the safe and effective implementation of such new raw materials.

Keywords: hydrogen-rich plastics, reductant, blast furnace

CONTRIBUTION TO LOWER COSTS BY INCREASE OF MANGANESE IN HOT METAL IN HBIS'S STEELWORKS IN SMEDEREVO

Tomislav Bradarić¹, Aleksandar Vasić²

e-mail: toma.bradaric@gmail.com

1 - HBIS GROUP Serbia Iron & Steel doo Belgrade

2 - University of Belgrade, Faculty of Technology and Metallurgy, 4 Karnegijeva St., 11120 Belgrade, Serbia

The purpose of this paper is to explore the reduction in production costs in HBIS Steelworks that can be achieved by increasing the manganese content in hot metal, compared to its current level of about 0.4%. The role of manganese in the production of hot metal and steel is well known. At the 3rd MME SEE Congress in 2017 authors proposed to HBIS' BOF shop an increase in the manganese content as a measure to reduce production costs. The optimal manganese content is determined to be the most techno-economically favorable for the specific conditions of the BF-BOF process of individual Steelworks. The lower limit of the interval in which the manganese content changes is mainly imposed by problems with the formation of slag and with the removal of sulfur from the metal, and the upper limit is determined by the maximum manganese content in the Steelworks product mix.

Previously applied and newly developed models for process management were used to calculate the most important effects of increasing the manganese content, as well as detailed material balances used for planning and monitoring the consumption of raw materials. Calculations were made for an increase in manganese content in hot metal by 0.3% (an increase from 0.4% to 0.7%). For the expected improvements of the key process indicators at the BOF, it was obtained that reduction of the share of hot metal in the metallic charge of the heat (HMF) can be reduced by about 0.4%, and at the same time, the manganese content in the steel before tap can be increased by about 0.08%.

Though an increased consumption of manganese ore and a small increase in coke consumption would increase production costs, these would be more than offset by the increased content of MnO in slag. The latter, among other effects, stabilizes the operation of the BF, thus lowering the need for 'coke reserves'.

The paper also lists examples of the most favorable levels of manganese content at several similar converter plants in the world. In conclusion, it was proposed to the HBIS' Steelworks in Smederevo to consider suggested improvements in the converter process, as well as their total financial effects. A trial period of operation with a manganese content of 0.7% in hot metal would serve as the crucial test for the change. It should also be kept in mind that every reduction in the consumption of hot metal contributes to at least a small reduction in CO₂ emissions.

Keywords: Mn content in hot metal, BF, BOF, production costs

HIGH-DENSITY GLASS-CERAMIC MATERIALS OBTAINED BY POWDER METALLURGY

Vladimir Pavkov¹, Gordana Bakić², Vesna Maksimović¹, Ivana Cvijović-Alagić¹, Marija Prekajski Đorđević¹,
Dušan Bučevac¹, Branko Matović¹

e-mail: pavkow@vin.bg.ac.rs

1-Department of Materials Science, Vinča Institute of Nuclear Sciences - National Institute of the Republic of Serbia, University of Belgrade, Belgrade, Serbia,

2-Faculty of Mechanical Engineering, University of Belgrade, Belgrade, Serbia.

In modern industry, there is an increasing demand for environmentally friendly and light structural materials with good physical and mechanical properties, produced from cheap natural raw materials available in large quantities. One of the materials that meet the mentioned criteria is basalt. Basalt is a natural igneous rock of volcanic origin, created by the pouring of magma on the Earth's surface, the amount of which is significant in the territory of Serbia. Since basalt does not exhibit toxic, carcinogenic, or mutagenic effects, it is in the true sense a non-hazardous material and belongs to the group of eco-friendly materials.

In this research, andesite basalt aggregate from the "Donje Jarinje" site, in Serbia, was used to obtain high-density glass-ceramic materials. High-density glass-ceramic materials were obtained by powder metallurgy process, which consisted of the following methods: dry grinding, homogenization, cold uniaxial and isostatic powder pressing and sintering in the air. In order to achieve a high-density of the materials, the green compacts were sintered in the temperature range from 1040 to 1080 °C. After confirming that the highest density materials were achieved at the sintering temperature of 1060 °C, the sintering time was optimized in the time interval from 30 to 240 min. After the experimental test, the optimal sintering parameters for obtaining high-density glass-ceramic material at the temperature of 1060 °C for 60 min were achieved, whose relative density is 99.50%, and hardness is 6.70 GPa.

The characterization of andesite basalt powder was performed using the laser light diffraction method, scanning electron microscopy and X-ray diffraction method, while the characterization of sintered glass-ceramic materials was performed using the Archimedes method, X-ray diffraction method, optical light microscopy and Vickers hardness test.

The results of this research confirmed that by applying powder metallurgy and sintering in the air, high-density glass-ceramic materials could be obtained for various industrial applications in the civil engineering, chemical and food industries, as well as for the making of containers for the storage of nuclear waste. Also, high-density glass-ceramic materials would be suitable for making a matrix in modern composite materials.

Keywords: andesite basalt, powder metallurgy, sintering, glass-ceramics

SURFACE EFFECTS INDUCED BY SHORT-PULSED LASERS ON PM-SYNTHEZIZED CuCrZr ALLOY

Jelena Stašić¹, Milan Trtica¹, Jovana Ružić¹, Marko Simić¹, Andrijana Žekić², Dušan Božić¹

e-mail: jelsta@vinca.rs

1-“Vinča” Institute of Nuclear Sciences - National Institute of the Republic of Serbia, Materials Department, Center of Excellence “Cextreme Lab”, University of Belgrade, P.O.B. 522, 11001 Belgrade, Serbia,

2-“Vinča” Institute of Nuclear Sciences - National Institute of the Republic of Serbia, Physical Chemistry Department, University of Belgrade, P.O.B. 522, 11001 Belgrade, Serbia,

3- University of Belgrade, Faculty of Physics, Studentski trg 12, 11001 Belgrade, Serbia

High conductivity CuCrZr alloy belongs to precipitation strengthened copper alloys, in which reinforcing precipitates are formed during heat treatment (aging), providing improved mechanical properties. This alloy has a great potential for number of applications, including heat sinks in future fusion reactors. High precipitation strengthening potential of CuCrZr comes from small amount of chromium (less than 1 wt.%) forming precipitates during aging, while even smaller amount of zirconium provides improved fatigue strength and prevents growth of Cr-rich precipitates. In the present work, CuCrZr was synthesized starting from prealloyed powder (15-53 μm), by powder metallurgy route comprising following steps: cold pressing, sintering, solution annealing with water quenching, and cold deformation. Aging treatment of synthesized compacts was conducted in argon atmosphere with subsequent water quenching. The behavior of obtained samples under the effect of high heat fluxes was examined by employing nanosecond and picosecond laser pulses. Laser-irradiated surfaces were analyzed using OM, SEM, EDX, profilometric and indentation analysis. The obtained morphological, chemical and mechanical surface effects after the action of different lasers were considered and compared.

Keywords: Copper alloy; powder metallurgy; pulsed laser; high heat fluxes; laser surface modification

INFLUENCE OF THE MILLING TIME ON THE STRUCTURAL PARAMETERS OF Cu-Zr-B ALLOYS

Marko Simić¹, Jovana Ružić¹, Jelena Erčić¹, Dušan Božić¹, Željko Radovanović², Jelena Stašić¹

e-mail: marko.simic@vin.bg.ac.rs

1-Department of Materials, "Vinča" Institute of Nuclear Sciences - National Institute of the Republic of Serbia, University of Belgrade, PO Box 522, 11001 Belgrade, Serbia,

2-Metallurgical Engineering Department, Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4, 11120 Belgrade, Serbia

A copper matrix composite, Cu-2.71Zr-2.27B (wt. %), produced using the powder metallurgy technique, was observed. High-energy ball milling was employed to produce a powder mixture with the appropriate ratio, resulting in a copper matrix reinforced with ZrB₂ ceramic particles. In this paper, the influence of milling time on the structural parameters of these alloys was investigated. Various milling times (from 2h to 40h) and ball-powder ratios (1:10 and 1:15) were chosen for analysis. The structural and morphological changes of the mechanically alloyed powder mixture were investigated using scanning electron microscopy and X-ray diffraction. Moreover, variations in Cu particle size and powder mixtures' structural parameters were analyzed in relation to the milling time. Williamson-Hall analysis was employed to calculate crystallite sizes (D , nm), lattice parameter (nm), lattice strain (ϵ , %), and dislocation density (ρ , m⁻²). Taking both ball-powder ratios into account, the size of copper powder decreases with the increase in mechanical alloying time. Different times of mechanical alloying have been taken as reference points (2h, 4h, 6h, 8h, 10h, 20h, 30h, 40h). It was determined that the lattice parameter of the Cu particles reached its maximum value after 30 hours. The obtained results show higher dislocation density of the Cu particles for the 1:15 ball-powder ratio compared to the ratio of 1:10.

Keywords: Williamson-Hall analysis, Stereological analysis, mechanical alloying, copper alloys

EVALUATION OF PARAMETERS FOR THE PRODUCTION OF Al-WO₃ COMPOSITES

Dimitrije Anđić¹, Vaso Manojlović¹, Željko Kamberović¹, Branislav Marković², Nataša Gajić³, Miljana Popović¹,
Gvozden Jovanović²

e-mail: andjicd6@gmail.com

1-University of Belgrade, Faculty of Technology and Metallurgy, 4 Karnegijeva St., 11120 Belgrade, Serbia

2-Institute for Technology of Nuclear and Other Mineral Raw Materials (ITNMS), Belgrade, Serbia

3-Innovation Centre of the Faculty of Technology and Metallurgy Ltd, University of Belgrade, Belgrade, Serbia

The paper presents the results of the analysis of the influence of the synthesis parameters on obtaining the composite system (WAl₁₂ + Al₂O₃)/Al with improved mechanical properties. The starting powders of Al and WO₃ were homogenized until reaching the required composition with 1%, 2%, 5% and 7% WO₃. After homogenization, the obtained samples were pressed under a pressure of 10 MPa. The sintering of the obtained samples was performed in an argon atmosphere at temperatures of 550 °C, 585 °C, 615 °C and 650 °C for 20, 40 and 60 minutes. As a result of the test, it was concluded that the sintering temperature, as well as the sintering time, do not have a significant effect on the increase in hardness of these composites. The main parameter that affects the increase in strength of these composites is the WO₃ content. With an increase in the WO₃ content, there is an increase in the hardness of the composite.

Keywords temperature, time, powders, content, hardness

**MICROSTRUCTURE AND ELECTRICAL PERFORMANCE OF
BARIUM TITANATE DOPED ZINC OXIDE VARISTOR CERAMICS
VIA SIMPLIFIED CITRAGE-GEL METHOD**

Muhamad Syaizwadi Shaifudin¹, Mohd Sabri Mohd Ghazali¹, Wan Mohamad Ikhmal Wan Mohamad Kamaruzzaman¹,
Wan Rafizah Wan Abdullah², Lee Oon Jew¹

e-mail: syaizwadi@gmail.com

1-Faculty of Science And Marine Environment, University Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia,

2-Faculty of Ocean Engineering Technology And Informatics, University Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia

Electronic equipment is prone to damage from overvoltage that can be caused by lightning strikes, power surges, and other electrical disturbances. The need for high-performance voltage protection devices in electronic circuits drove the development of varistor ceramics. In this study, the performance of perovskite-type barium titanate (BaTiO_3) as only a single dopant to enhance the nonlinear electrical properties of zinc oxide (ZnO) varistor ceramics has been studied. The study aims to determine the performance of perovskite-doped varistor ceramics by elucidating the processing technique, compositions, microstructure, and electrical characteristics for low-voltage varistor applications. Several characterization methods were conducted, such as Thermogravimetric/derivative thermogravimetric (TG/DTG), field emission scanning electron microscope with an energy dispersive spectrometer (FESEM), Transmission electron microscope (TEM), X-ray diffraction (XRD), density measurement, and current-voltage (I-V) measurement. The incorporation of perovskite oxide in ZnO ceramics using a simplified citrate-gel method has significantly improved the microstructure homogeneity and energy absorption capability of varistor ceramics for low-voltage surge protection devices.

Keywords: varistor; ceramic; zinc oxide; barium titanate; simplified citrate-gel method

PREPARATION AND CHARACTERIZATION OF CHITOSAN–CLAY COMPOSITES AS POTENTIAL DRUG CARRIERS

Danina Krajišnik¹, Snežana Uskoković-Marković², Aleksandra Daković³, Danijela Smiljanić³, Maja Kozarski⁴,
Vesna Lazić⁴

e-mail: snezana.uskokovic@pharmacy.bg.ac.rs

1-Department of Pharmaceutical Technology and Cosmetology, University of Belgrade – Faculty of Pharmacy, Serbia,

2-Department of Analytical Chemistry, University of Belgrade – Faculty of Pharmacy, Serbia,

3-Institute for Technology of Nuclear and Other Mineral Raw Materials (ITNMS), Belgrade, Serbia,

4-Institute for Food Technology and Biochemistry Faculty of Agriculture, University of Belgrade, Serbia

Modern pharmacy and medicine impose the need to find advanced therapeutic systems, which will be more rational, efficient, and eco-friendly at the same time. The discovery that natural clays may act as drugs or coadjuvants in pharmaceutical preparations has greatly enlarged the field of their application. Furthermore, to enhance coadsorption of organic molecules, functionalization with cation substances has been used for modification of natural clays. With that aim composites based on bentonite (from deposit Beretnica, the Republic of Serbia) and chitosan were prepared. Chitosan, a natural cationic polysaccharide, has received considerable attention as a functional, renewable, nontoxic, and biodegradable biopolymer for diverse pharmaceutical/biomedical applications. Two types of chitosan were used for the composite preparation: commercially available low molecular weight (Sigma-Aldrich Co.) and chitosan isolated from fruit bodies of commercially grown mushroom *Agaricus bisporus* (Delta Danube d.o.o., Kovin, the Republic of Serbia).

Adsorption of tetracycline onto chitosan-clay composites from aqueous media (pH 3,4) was investigated under different conditions (different initial drug concentrations and different contact time). The concentrations of adsorbed antibiotic were determined spectrophotometrically from the difference between the initial concentrations of tetracycline and their amounts remaining in the aqueous medium after the establishment of dynamic equilibrium. The characterization of chitosan-clay composites before and after adsorption of tetracycline was performed by zeta potential measurements.

Adsorption of tetracycline followed a nonlinear type of isotherm for all composites. The Langmuir equation showed the best fit of the equilibrium adsorption data over the entire concentration range (0.05 – 1 mg/ml) and the maximum adsorbed amount of the drug onto both chitosan-clay composites was up to ~ 200 mg/g. Only slight differences in adsorption of the drug were noticed between composites with commercially available chitosan and chitosan isolated from mushroom. The detected decrease of zeta potentials for the drug chitosan–clay complexes revealed the interactions of the composite mineral surface with tetracycline molecules.

The obtained results indicated the potential of the tested newly synthesized chitosan-clay composites for the development of therapeutic systems for topical application to infected and damaged skin. The study also pointed out an extremely important aspect today, which is the possibility of using fungi as a renewable source of chitosan.

Keywords: clay, chitosan, composites, drug carriers, tetracycline

Acknowledgement

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MICROSTRUCTURAL AND THERMAL CHARACTERIZATION OF THE Bi–In–Sn TERNARY ALLOYS

Ljubiša Balanović¹, Dajana Milkić², Dragan Manasijević¹, Ivana Marković¹,
Milan Gorgievski¹, Uroš Stamenković¹, Kristina Božinović¹

e-mail: ljbalanovic@tfbor.bg.ac.rs

1-University of Belgrade, Technical Faculty in Bor, VJ 12, 19210 Bor, Serbia,
2-Measuring Transformers Factory Zaječar, Stražilovska 57, 19000 Zaječar, Serbia

In this work, the microstructural and thermal characterization of three-component alloys from the Bi–In–Sn system was carried out, all to investigate the possibility of using these alloys as low-temperature lead-free solder materials complying with the European Union directives on the prohibition of the use of hazardous substances in electrical equipment and electronic devices. The composition of the investigated alloys was from three cross-sections with mass fraction ratios In/Sn=1, Bi/Sn=1, and Bi/In=1. The chemical composition and microstructure of the alloy samples were studied using scanning electron microscopy with energy-dispersive X-ray spectrometry (SEM-EDS). The acquired results of the microstructural investigation revealed good agreement with the results of the thermodynamic analysis of phase equilibria according to the calculation of the phase diagram (CALPHAD method). Thermal diffusivity was measured by the flash method using Discovery Xenon Flash, DXF-500. Based on the measured values of thermal diffusivity and calculated specific heat capacity, the thermal conductivity of the solid Bi–In–Sn alloys were determined. The results show that both the thermal conductivity and thermal diffusivity are dependent upon the prevailing temperature conditions and alloy composition to a certain extent. The variations of thermal diffusivity, specific heat capacity, and thermal conductivity with alloy composition and temperature were presented and discussed.

Keywords: Bi–In–Sn alloys, microstructure, thermal conductivity

DISTRIBUTION OF ALLOYING ELEMENTS IN FRICTION STIR WELDED AA2024-T351 ALLOY

Ljubica Radović¹, Fernando Carrasco López^{2,3}, Igor Radisavljević¹, Nenad Radović²

e-mail: ljubica.radovic@mod.gov.rs

1-Military Technical Institute Belgrade,

2-Faculty of Technology and Metallurgy, University of Belgrade

3-Universidad Politécnica de Madrid, Spain

The aim of this work was to evaluate microstructure of overlap joint by means of optical and SEM/EDS microscopy. Specimens of commercial 2024-T351 containing 4.70% Cu, 1.56% Mg, 0.65% Mn, 0.17 %Fe, 0.11 %Zn, 0.046% Si, 0.032 %Ti, 0.011% Zr, 0.006% Ni, 0.006 %Cr, rest Al, rolled plates of 6.0 mm thickness with 2.0 and 3.5mm overlaps were welded using FSW, welding parameters were: rotation rate R=750 rpm, welding speed =73 mm/min (R/ v=10,27). Welded joints were prepared by mechanical grinding, polishing and electrolytical etching and evaluated using optical microscope and/or Scanning Electron Microscope (SEM) with EDS.

In both welded joints presence of characteristic zones are identified base metal (BM), Thermomechanical affected Zone (TMAZ), Heat Affected Zone (HAZ) and Nugget zone (NZ). Apart from these zones, bright line like areas are placed between NZ and TMAZ. This zone is characterized with absence of large particles and considerable depletion of Cu, Mg and Mn. It is assumed that presence of this zone is related to presence of two plates in cross section and slower cooling after welding that enabled intensive diffusion of listed elements. Furthermore, level of iron seems to be unchanged, as diffusion of iron is slowest.

Keywords: friction stir welding, AA2024 alloy, particle distribution

ADVANCEMENT OF BIOCOMPATIBILITY AND MECHANICAL SURFACE CHARACTERISTICS OF THE Ti-13Nb-13Zr ALLOY USING ELECTROCHEMICAL ANODIZATION

Dragana Mihajlović¹, Marko Rakin¹, Đorđe Veljović¹, Bojan Međo¹, Veljko Đokić^{1,2}

e-mail: vdjokic@tmf.bg.ac.rs

1-University of Belgrade, Faculty of Technology and Metallurgy, 11120 Belgrade, Serbia,

2-Innovation Centre of the Faculty of Technology and Metallurgy in Belgrade, Karnegijeva 4, 11120 Belgrade, Serbia

The coarse-grained (CG) and ultrafine-grained (UFG) Ti-13Nb-13Zr alloy (TNZ) were used as tested materials in this study. UFG alloy was obtained using a high-pressure torsion process (HPT) at room temperature, with 5 rotations, and with 4.1 GPa pressure. After that, the surface was modified using electrochemical anodization in the 1M H₃PO₄ + NaF electrolyte, for 60 and 90 minutes.

Scanning electron microscopy (SEM) was used to characterize the morphology of the modified surface after electrochemical anodization. In order to determine whether the electrochemical anodization leads to obtaining the surface modulus of elasticity values close to the values characteristic of the human bones, a nanoindentation test was done. The nanoindentation test was performed on the device called nanoindenter *G200, Agilent Technologies*, where a diamond tip of the Berkovich type shaped as a three-sided pyramid was used as an indenter. Control of the nanoindentation test was done by nanoindentation depth, where for non-anodized materials a depth of 2000 nm was used, while for anodized materials a minimum value of 10% of the thickness of the formed nanostructured oxide layer was used. Biocompatibility of the tested titanium alloy was estimated by the tetrazolium salt colorimetric test (MTT test) using mouse fibroblasts (L-929) and human lung fibroblasts (MRC-5). The cell adhesion on the alloy surface was analyzed using SEM.

Electrochemical anodization has led to the formation of the nanostructured oxide layer on the titanium alloy surface. The obtained results show the existence of the influence of anodizing time on the nanostructured oxide layer morphology. Characterization of the titanium alloy surface using nanoindentation showed a decrease in the values of modulus of elasticity for an alloy with a nanostructured oxide layer, which is close to the values of bone tissue in the human body. The results of the MTT test showed that the titanium alloys before and after electrochemical anodization were not cytotoxic. After electrochemical anodization, a uniform nanostructured surface, with nanotubes, was formed which led to better cell viability and adhesion of L-929 and MRC-5 cells in contact with the test alloys than of cells in contact with the control material.

Keywords: High-pressure torsion, Ultrafine-grained Ti-13Nb-13Zr alloy, Electrochemical anodization, Biocompatibility, Surface modulus of elasticity

INFLUENCE OF HOMOGENIZATION ON CHEMICAL PROPERTIES OF ALUMINUM ALLOY EN AW-5083

Natalija Dolic¹, Zdenka Zovko Brodarac¹, Franjo Kozina¹

e-mail: ndolic@simet.unizg.hr

1-University of Zagreb Faculty of Metallurgy, Aleja narodnih heroja 3, 44 000 Sisak, Croatia

The aim of this paper is to estimate the chemical composition homogeneity of six aluminum alloy EN AW-5083 slabs produced by the Direct Chill Process in as-cast and homogenized state. The homogeneity assessment is based on the content of magnesium (Mg) as a main alloying element.

The experimental part of this work is based on the analysis of Mg content on previously defined positions within the cross-sectioned plates taken from the ingots' fronts and rears. The determination of chemical composition homogeneity was based on the Latin square experiment design. The statistical processing enabled the determination of the difference in the cross-section of each individual plate, while the chemical homogeneity of the blocks as a whole was evaluated by comparing the results from the ingots' fronts and rears. The significance and influence of homogenization on the magnesium content were quantified by comparison of the obtained measurement results for the as-cast and homogenized state of the tested EN AW-5083 aluminum alloy.

The obtained results indicate that the Mg content of all melts and all average values of magnesium content in as-cast and homogenized states per individual batch are within the values prescribed by the norm. A small deviation in the magnesium content was detected in the first three tested alloys in the homogenized state (3112, 3113, 3114). The line Energy Dispersive Spectrometry analysis confirmed the even distribution of Mg in the aluminum matrix for both metallurgical conditions (as-cast and homogenized conditions). However, a decrease in Mg content was observed from the melt to as-cast and homogenized ingots due to oxidation loss. The average value of Mg loss for all six observed batches is: during casting from the amount of Mg in the melt to the cast block 0.18 %, additional loss of Mg during the homogenization of 0.14 %, while the average total loss of magnesium is 0.32 %.

Keywords: aluminium alloy EN AW-5083, chemical composition homogeneity, content of magnesium, Direct Chill Process

MICROSTRUCTURE AND THERMAL CONDUCTIVITY OF THE Ag–Bi–Sn TERNARY ALLOYS

Dragan Manasijević¹, Ljubiša Balanović¹, Ivana Marković¹, Milan Gorgievski¹, Uroš Stamenković¹,
Kristina Božinović¹, Duško Minić², Milena Premović Zečević²

e-mail: dmanasijevic@tfbor.bg.ac.rs

1-University of Belgrade, Technical Faculty in Bor, VJ 12, 19210 Bor, Serbia,

2-University of Priština, Faculty of Technical Science, Kneza Miloša, 7, 4000 Kosovska Mitrovica, Serbia

The Ag–Bi–Sn ternary alloys represent an important material for development of lead-free solders. In this work, the effect of tin content on thermal properties of the Ag–Bi–Sn ternary alloys was investigated. Five Ag–Bi–Sn ternary alloys with tin content ranging from 12.8 to 75.1 mass% and nearly equal mass contents of silver and bismuth were prepared and experimentally studied. Microstructures of as-solidified alloy samples were examined using scanning electron microscopy with energy dispersive X-ray spectrometry (SEM-EDS) and X-ray diffraction (XRD) analysis. The observed constitutive phases were Ag₃Sn, (Sn) and, (Bi) phases as well as ternary (Sn)+(Bi)+Ag₃Sn eutectic. Thermal diffusivity measurements in the temperature range from 25 to 100 °C were performed using the flash method. Thermal conductivities of the solid Ag–Bi–Sn alloys were obtained using the measured values of thermal diffusivity. Thermal conductivity of the studied Ag–Bi–Sn ternary alloys considerably increases with increasing tin concentration and slightly decreases with increasing temperature. The measured thermal diffusivity at room temperature extends from 8.00 to 27.80 mm²s⁻¹ and thermal conductivity extends from 14.24 to 45.77 Wm⁻¹K⁻¹ with increasing Sn content. Chemical and phase compositions have a strong effect on thermal diffusivity and thermal conductivity of the studied Ag–Bi–Sn alloys. It was found that an increase of the phase fractions of the (Bi) solid solution phase and Ag₃Sn intermetallic compound hinders heat transfer and decreases thermal conductivity.

Keywords: Ag-Bi-Sn alloys, microstructure, thermal conductivity

MICROSTRUCTURE DEVELOPMENT DURING HEAT TREATMENT OF HIGH CHROMIUM WHITE CAST IRON

Marija Mihailović¹, Aleksandra Patarić², Snežana Aksentijević³, Branka Jordović⁴

e-mail: marija.mihailovic@ihtm.bg.ac.rs

1-University of Belgrade - Institute of Chemistry, Technology and Metallurgy - National Institute of the Republic of Serbia,
Njegoševa 12, Belgrade, Serbia,

2-Institute for Technology of Nuclear and other Mineral Raw Materials, Franchet d'Eperey St.86, 11000 Belgrade, Serbia,

3-Western Serbia Academy of Applied Studies, Užice Department, Trg Svetog Save 34, 31000 Užice, Serbia,

4-Faculty of Technical Sciences Čačak, Svetog Save 65, Čačak, Serbia

Notwithstanding the fact that it has been in use for a very long time, the high chromium white cast iron (HCrWCI) still triggers scientists due to its outstanding wear resistance. Besides its well-known as-cast usage, it can be used as a coating material, as well. HCrWCI owes this feature due to the presence of carbides in the microstructure, which depends on the composition and heat treatment regime. This investigation was conducted on two HCrWCI alloys, where in addition to chromium, the second important alloying element is molybdenum (Mo). Apart from other alloying elements, HCrWCI_1 is alloyed with 24.48% Cr and 1.32% Mo, while HCrWCI_2 contains 14.11% Cr and 2.47% Mo. The comprehensive microstructural characterization was carried out on as-cast samples and samples obtained after quenching (at -196°C) and/or quenching followed by tempering (at 250°C). Important microstructure indicators are the stability of austenite, the ratio of micro-constituents (especially different types of carbides), and their arrangement and morphology, so they are revealed and discussed in relation to the influencing mechanical properties.

Keywords: wearing; carbides; molybdenum; eutectic; austenite.

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MICROSTRUCTURE ASSESSMENT OF Co ALLOY INTENDED FOR DENTISTRY

Aleksandra Patarić¹, Gordana Marković¹, Nataša Đorđević¹, Slavica Mihajlović¹, Marija Mihailović²

e-mail: a.pataric@itnms.ac.rs

1-Institute for Technology of Nuclear and other Mineral Raw Materials, Franchet d'Eperey St.86, 11000 Belgrade, Serbia
2-University of Belgrade - Institute of Chemistry, Technology and Metallurgy - National Institute of the Republic of Serbia, Njegoševa 12, Belgrade, Serbia

Cobalt–chromium–molybdenum (CoCrMo) alloys are known for medical use due to their biocompatibility, corrosion and wear resistance. The chemical and phase composition, as well as microstructure of the alloy directly affect the mechanical properties. In this investigation, CoCrMo alloy samples were obtained by vacuum precise casting. The procedure of melting and casting process as well as their parameters are given. Molds fabricated of copper, gray iron, steel, ceramics and graphite were used during the casting process. In this way, the cooling rate influence on the obtained microstructure was examined. Besides, different casting temperatures (1400°C, 1450°C and 1500°C) were applied for each kind of mold. After metallographic preparation, the microstructure was examined on the cross section of samples by optical microscopy. The obtained results show that by increasing the cooling rate, the microstructure of samples become finer and more homogeneous.

Keywords: CoCrMo alloy, vacuum precise casting, cooling rate

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IMPROVED MECHANICAL AND BIOLOGICAL PROPERTIES OF CHITOSAN COATED SCAFFOLDS BASED ON HYDROXYAPATITE DOPED WITH Mg, Sr AND Cu

Jakov Dimitrijević¹, Julijana Tadić², Vukašin Ugrinović³, Tamara Matic³, Đorđe Janačković¹, Đorđe Veljović¹

e-mail: djveljovic@tmf.bg.ac.rs

1-University of Belgrade, Faculty of Technology and Metallurgy, Belgrade, Serbia,

2-University of Belgrade, Vinča Institute of Nuclear Sciences, Belgrade, Serbia,

3-Innovation Center of the Faculty of Technology and Metallurgy Ltd, Belgrade, Serbia

Bioceramic and biocomposite scaffolds based on doped calcium phosphates are in the focus of materials science and tissue engineering in last years, in order to process macroporous materials with a potential to enable controlled environment and enough volume for new bone tissue formation in bioreactors or *in vivo*. Different multi-ion doped calcium hydroxyapatite rod-like nanoparticles, due to the chemical and morphological similarity with human hard tissue minerals, are good starting point for scaffold processing and mimicking the properties of human hard tissue. Chitosan is a natural polymer which possesses biological properties such as antibacterial, antiviral, and fungicidal activity, and also has an immuno-stimulating effect. The aim of this study was to process bioceramic scaffolds based on hydroxyapatite, simultaneously doped with magnesium, strontium and copper cations, and further coated with chitosan, as biocompatible and biodegradable polymer, in order to improve their mechanical properties and bioactivity. Nanoparticles of triple doped hydroxyapatite, synthesized by hydrothermal method, were calcinated in order to obtain spherical-like particles of β -tricalcium phosphate with incorporated Mg, Sr, and Cu cations. Macroporous scaffolds were obtained by sponge replica method, by sintering at 1445 °C for 2 h. Phase transformation to α -tricalcium phosphate occurred and α -tricalcium phosphate became the dominant phase. Sintered scaffolds were further coated at 50 °C with chitosan of similar viscosity and different molecular weights. Soft biodegradable chitosan-based coatings showed great potential for improvement of compressive strength of scaffolds and also for filling the microcracks as a consequence of imperfect sintering of macroporous bioceramics. Bioactivity of coated scaffolds was investigated at 37 °C in simulated body fluid and morphological analysis confirmed the positive effects of chitosan coatings on the formation of new nanostructured hydroxyapatite layer at the scaffold surface, indicating the great potential of obtained materials in bone tissue engineering.

Keywords: Scaffold, Hydroxyapatite, Chitosan, Mechanical properties, Bioactivity.

**OPTIMIZING ELECTROCHEMICAL ANALYSIS FOR THE ACCURATE
CHARACTERIZATION OF ELECTRODE MATERIALS IN ENERGY STORAGE
APPLICATIONS: A COMPREHENSIVE GUIDE TO ELECTROCHEMICALLY
ANALYZING MATERIALS**

Aleksandra Popović¹, Branimir Grgur¹

e-mail: apopovic@tmf.bg.ac.rs

¹-University of Belgrade Faculty of Technology and Metallurgy Karnegijeva 4, 11120 Belgrade, Serbia

Electrochemical Energy Storage (EES) technologies have gained considerable attention in recent years due to their potential to reduce the reliance on fossil fuels and promote a sustainable society. To achieve this, researchers are actively searching for an efficient electrode material that can advance the field of EES. However, new researchers often face challenges in understanding the core concepts, procedures, and methodologies that define the electrochemical community's standards. Although various publications have provided guidance on the matter, none have offered an actual example of a step-by-step procedure to tackle the issue.

To address this gap, we present a comprehensive electrochemical analysis of polypyrrole, a commonly used conducting polymer with substantial potential as an electrode material for batteries and supercapacitors. Our approach provides a detailed procedure, including sample preparation, instrumentation, data acquisition, and analysis. The presented methodology serves as a guideline for researchers, regardless of their field, to carry out accurate and reliable electrochemical analysis.

The methodology's validity is demonstrated by the obtained results, indicating the effectiveness of polypyrrole as a potential electrode material for EES. The presented analysis provides critical insights into the properties and behavior of polypyrrole in electrochemical systems and can guide researchers in selecting suitable materials for their studies

Keywords Electrode, electrochemical analysis, energy storage, polypyrrole.

PRELIMINARY CYTOTOXICITY TESTING OF NEWLY SYNTHESISED SBA-15 MATERIAL

Maja Kokunešoski¹, Aleksandra Šaponjić¹, Zvezdana Baščarević², Ivan Jovanović¹, Jelena Filipović Tričković¹,
Ana Valenta Šobot¹

e-mail: majako@vin.bg.ac.rs

1-"Vinča" Institute of nuclear science, National Institute of the Republic of Serbia, University of Belgrade, P.O. Box 522, 11001
Belgrade, Serbia,

2-Institute for Multidisciplinary Research, University of Belgrade, Kneza Višeslava 1, Belgrade, Serbia

Material SBA-15 with all of its particles in the form of regular spheres was synthesised by template method using Pluronic P₁₂₃ (non-ionic triblock copolymer) as a surfactant and tetraethoxysilane (TEOS, 98%) as a source of silica. The synthesis of spherical SBA-15 was carried out in acidic conditions according to method proposed by Zhao et al. Instead of expensive commercial cetyltrimethylammonium bromide as a co-surfactant, a spent HCl solution obtained after the chemical treatment of clay in an aqueous solution of HCl was used. The mixture was aged at a higher temperature. After that, the spherical SBA-15 was made by drying at room temperature after washing with distilled water and then calcining in flowing air to decompose the triblock copolymer. The continual presence of spheres of diameters around 2 µm in the entire SBA-15 was confirmed by scanning electron microscopy. The Energy-dispersive X-ray spectroscopy confirmed that the spheres consisted only of SiO₂ in composition. In addition, X-ray diffraction and Fourier-transform infrared spectroscopy methods were used to characterise SBA-15 material. To assess the cytotoxic effects of newly synthesised SBA-15 material, HEK 293 cells were treated with particles and extracts obtained from particles incubated 24 hours at 37 °C in serum-free DMEM medium and DMEM with 10% serum. Cells were treated with concentrations of 100, 250, and 500 µg/mL for 72 hours. All treatments reduced cell viability in a concentration-dependent manner (p<0.001). Cytotoxicity was more pronounced in particle treatment, indicating that mechanical damage was induced in cells, whereas the extraction process influences the level of toxicity since the sera-free medium had the mildest effect. To acquire safe particles for human use, additional synthesis optimisation is required.

Keywords: Spherical SBA-15, Pluronic P₁₂₃, Cytotoxicity

DISSOLUTION OF POLYPHOSPHATE GLASS: IMPACT OF pH ON DIFFUSION COEFFICIENTS OF MODIFYING CATIONS AT LOW TEMPERATURES

Jelena Nikolić¹, Vladimir Topalović¹, Marija Đoščić¹, Veljko Savić¹, Milena Obradović¹, Srđan Matijašević¹, Snežana Grujić²

e-mail: j.nikolic@itnms.ac.rs

1-Institute for the Technology of Nuclear and other Mineral Raw Materials-Belgrade, Serbia,

2-University of Belgrade, Faculty of Technology and Metallurgy-Belgrade, Serbia

This paper shows the dissolution behavior of polyphosphate glass in demineralized water and 2% citric acid solution at temperatures: 15, 20, 30, 37 and 50 °C. The influence of the solvent type on the diffusion coefficients of the modifying cations, under static conditions, is also presented. The diffusion coefficients, determined from concentration profiles of the modifying cations, are calculated to be in the range of 0.27 to 350x10⁻¹⁴ m²/h for dissolution in demineralized water, and in the range of 3.6 to 148x10⁻¹² m²/h for the dissolution in 2% citric acid solution. The values of activation energy of cations diffusion in demineralized water are in the range from 68.32 to 105.41 kJ/mol, while for 2% citric acid solution they are in the range from 39.28 to 55.34 kJ/mol.

Keywords: polyphosphate glass, diffusion coefficient, dissolution

CHARACTERIZATION OF RAW PEACH STONES AND ITS BIOCHAR BY SEM, FTIR AND RAMAN SPECTROSCOPY

Tatjana Šošćarić¹, Stefan Kolašinac², Zorica Lopičić¹, Anja Antansković¹, Vladimir Adamović¹, Jelena Avdalović³,
Mladen Bugarčić¹

e-mail: t.sostaric@itnms.ac.rs

1-Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia,

2-University of Belgrade, Faculty of Agriculture, Zemun-Belgrade, Serbia,

3-University of Belgrade, Institute of Chemistry, Technology and Metallurgy, Belgrade, Serbia

Growing industrialization and non-renewable resource depletion have a huge impact on alternative clean up technologies development, inducing investigation of the renewable, low-cost waste materials. Recently, lignocellulosic waste biomass, generated at a large scale by different industries, appeared as an attractive feedstock worldwide, due to its abundance, availability, multi-functionality and low cost. Since the fruits production has increasing trend all over the world, generation of lignocellulosic waste such as fruit stones (that represent approximately 1/5 of the total fruit mass) became a great environmental threat. In order to minimize negative impact on the environment, avoid greenhouse gasses emissions and help in resource depletion by its renewable nature, it is necessary to explore its performances regarding its wide application potential. Thermo-chemical conversion of lignocellulosic biomass in oxygen limited conditions, in order to get biochar, has received a lot of attention recently. The biochar production requires lower energy inputs and less expensive technology compared to activated carbon (estimation is that production cost of activated carbon are six times higher than biochar costs).

In this paper, waste peach stones biomass was effectively converted into biochar at 500 °C under inert (Ar) atmosphere. Pyrolysis was performed in Nabertherm 1300 muffle furnace for residence time of 1.5 h, with a heating rate of 10 °C/min, while Ar was circulated through the sample at a rate of 100 mL/min. The raw biomass (PS) and obtained biochar (PS-B) with particle size $0.1 \text{ mm} < d_p < 0.5 \text{ mm}$, were characterized by Scanning Electron Microscopy (SEM), Fourier transform infrared spectrometry (FTIR) and Raman spectrometry to understand physicochemical changes which have been occurred after pyrolysis. SEM analysis revealed the increased surface area due to the visible augmentation of pores and roughness. FTIR analysis showed that many bands present in native biomass cannot be observed in biochar, due to the conversion or removal of the most of the functional groups. Prominent bands have confirmed presences of aromatic compounds in biochar: at 1592 cm^{-1} (C=C bond stretching derived from aromatic rings in the lignin), 1030 cm^{-1} (alcohol C-O stretching vibration) and in the region $900 \text{ to } 700 \text{ cm}^{-1}$ (originating from aromatic compounds). These finding are in accordance with results from Raman analysis, where D and G bands (at 1350 cm^{-1} and 1580 cm^{-1} respectively), indicate the presence of poly-aromatic hydrocarbons.

Thermo-chemically modified biomass like this has a great application potential: as a pollutants sorbent, biofuel, soil amendment, biocarriers, and in construction and electronic industries.

Keywords: peach stones; pyrolysis; biochar; SEM; FTIR; Raman.

**EFFECT OF CHEMICAL COMPOSITION ON THE MICROSTRUCTURE, HARDNESS,
AND ELECTRICAL CONDUCTIVITY OF THE TERNARY Bi-Cu-Ge SYSTEM**

Aleksandar Đorđević^{1,*}, Milena Zečević¹, Duško Minić¹

e-mail: aleksandar.djordjevic@pr.ac.rs

¹-University of Prishtina in Kosovska Mitrovica, Faculty of Technical Science, Department of Technological engineering, Knjaza Miloša 7, 38220 Kosovska Mitrovica, Serbia

This paper presents the results of experimental and analytical testing of the ternary Bi-Cu-Ge system. The results include the characterization of the tested alloys, in terms of the dependence of hardness and electrical conductivity on the composition of alloy components. The prepared alloys were tested using X-ray powder diffraction (XRD) method, scanning electron microscopy with energy dispersive spectrometry (SEM-EDS), hardness and electrical conductivity tests. Used analytical method was Calphad method and the corresponding thermodynamic program (Pandat ver. 8.1). Based on the obtained results, structural characteristics, phase composition, mechanical properties and electrical conductivity were determined. A good general agreement was obtained between the experimental and calculated values. For the obtained values of hardness and electrical conductivity, a mathematical model was used in order to determine the properties of the alloy in the entire range of the composition.

Keywords: Bi-Cu-Ge system; hardness measurement; electrical conductivity measurement; mathematical model.

**DIRECTIONS AND CHALLENGES OF THE CIRCULAR ECONOMY:
MOVEMENT OF MUNICIPAL SOLID WASTE IN CITY OF PARAĆIN**

Uroš Stojaković¹, Aleksandar Jovanović², Ivana Mikavica², Branislav Marković², Vladimir Pavićević³

e-mail: a.jovanovic@itnms.ac.rs

1-Hemofarm A. D. STADA Group, Prote Mateje 70, Belgrade, Serbia,

2-Institute for Technology of Nuclear and Oterr Mineral Raw Materials, Franš d' Eperea 86, Belgrade, Serbia,

3-University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, Belgrade, Serbia

This paper aims to show a short economic analysis of the profitability of recycling management on the territory of Paraćin municipality. Consumption habits of modern society and socioeconomic conditions have made it difficult to implement sustainable waste management strategies. Industry, economics, sociocultural and political activities must work together to solve the multifaceted problem of waste management. The municipality of Paraćin is located in the central part of the Republic of Serbia, Pomoravlje region. Annually, this municipality generates about 18,000 t of waste, while the mass of recyclable waste is approximately 7,000 t. This small industrial city have been constantly facing with an insufficient number of available bins and containers for the disposal of Municipal Solid Waste (MSW). Moreover, this city has been challenging by an increasing number of illegal dumps along the roads. Together with surrounding municipalities, the local government was planning to create Regional Recycling Center and Landfill with all necessary operations. Sorting of the waste at the source of its origin, and adequately disposing it in containers intended for different types of waste, would provide precisely insight into the movement of recyclable waste. In addition, with these actions by the local community, it could produce a habit among citizens for proper deposition and disposal of household waste and reduce the negative impact on the environment. This article shows the categories of waste and their percentages, as well as the resulting amount of recyclable waste, which should be a starting point for establishing a recycling center with surrounding municipalities.

Keywords: Municipal Solid Waste (MSW); recyclable materials; recycling center; economy analysis

COMPARATION OF CORROSION AND MECHANICAL PROPERTIES OF COMMERCIAL AND RECYCLED 6060 AND 6082 ALUMINIUM ALLOYS

Silvana Dimitrijević¹, Stevan Dimitrijević², Aleksandra Ivanović¹, Marija Korac³, Milisav Ranitović²

e-mail: silvana.dimitrijevic@irmbor.co.rs

1-Mining and Metallurgy Institute Bor, Zeleni bulevar 35, 19210 Bor, Serbia,

2-Innovation Center Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4, 11000 Belgrade, Serbia,

3-University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, 11120 Belgrade, Serbia

Although the same quality of various metals from the recycling process is considered for granted, obtaining aluminium of high purity and aluminium alloys with identical properties as from production from pure raw materials is a very challenging process, especially if the sources for recycling are numerous materials that contain different impurities. In this study, recycled alloys were produced by melting of aluminium scrap with the addition of the required components. This paper aims to compare two commercial Al alloys with their analogues from the recycling process. Both of the alloys belong to the aluminium series 6000, with designations 6060 and 6082. The results of comparing corrosion and physical characteristics are presented in this study. Physical characterization was performed by measuring the alloys' hardness and electrical conductivity. The electrochemical investigation used several corrosion methods, OCP (open circuit potential), measuring polarization curves in apparent Tafel region with extrapolation of results, and LRP (linear polarization resistance). The corrosion environment was acidulated 0.5M Na₂SO₄ with pH=2.5. The received results have confirmed insignificant differences between the properties of the commercial and alloys produced by the recycling. Recycled alloys have achieved almost identical mechanical properties and even slightly better corrosion characteristics, which was the consequence of the lower concentration of impurities in recycled alloys or higher concentration of microalloying components.

Keywords: recycling, aluminum alloys, corrosion, Tafel extrapolation, linear polarization resistance

INFLUENCE OF WPCBS MECHANICAL PRE-PROCESSING ON BASE METALS LEACHING EFFICIENCY

Milislav Ranitović¹, Željko Kamberović², Marija Korać², Dragana Ivšić¹, Nikola Jovanović¹

e-mail: mranitovic@tmf.bg.ac.rs

1-Innovation Center Faculty of Technology and Metallurgy in Belgrade Ltd., University of Belgrade, Karnegijeva 4, 11120 Belgrade, Serbia,

2-University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, 11120 Belgrade, Serbia

The exploitation period of various electronic devices is significantly shortened by technological advancements and market expansion. Because of this, discarded electronics have become the fastest-growing solid waste stream in the world. Printed circuit boards (PCBs) are an essential part of almost all electric and electronic devices. These particular components sometimes have a concentration of metals like copper, gold, and silver that is more than ten times higher than ones in conventional raw materials. Therefore, efficient management is crucial when dealing with this particular type of waste, not only from the perspective of reducing pollution but also in the context of resource efficiency. Up until recently, waste PCB recycling operations were concentrated on the recovery of only specific valuable metals from waste PCBs, such as copper and gold. Other valuable metals like tin, lead, iron, or nickel have suffered significant losses as a result of this mass-balanced recycling model. However, recent developments in the electrification of the transportation sector have changed the emphasis to a model that is more product-centric, putting metals like iron, nickel, and cobalt in the spotlight. In this study, the effectiveness of various base metals (BM) leaching from mechanically pre-processed waste PCBs using various mechanical and/or chemical operations was evaluated. Leaching was done using the sulfuric acid solution with the addition of hydrogen peroxide as an oxidant at different temperatures. The influence of system variables, like acid concentration, amount of oxidant, particle size, time, and temperature on BM recovery was investigated. The obtained data led to the conclusion that mechanical preparation has a significant impact on BM leaching efficiency, not only in terms of metals liberation for contact with the leaching agent but also in terms of various hydrodynamic properties of waste PCB particles.

Keywords: PCBs, mechanical processing, leaching, copper

DEALLOYING OF PdNi₅ ALLOY IN 1.0 M NITRIC ACID

Aleksandra Ivanović¹, Silvana Dimitrijević¹, Stevan Dimitrijević², Renata Kovačević¹

e-mail: aleksandra.ivanovic@irmbor.co.rs

1-Mining and Metallurgy Institute Bor, Zeleni bulevar 35, 19210 Bor, Serbia,

2-Innovation Center Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4, 11000 Belgrade, Serbia,

Binary palladium alloys with nickel are known to have catalytic activity and are also used as catalyst-trap in the synthesis of nitric acid. High Pd alloys are corrosion resistant in many solutions, including highly alkaline and highly acidic. Although generally not prone to corrosion in low concentrations of mineral acids due to the high content of palladium in it, these alloys are still prone to the dealloying effect under specific conditions. The dealloying process was researched under anodic polarization in 1.0 M nitric acid solution. Optical microscopy, scanning electronic microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS), inductively coupled plasma atomic emission spectroscopy (ICP-AES), and electrochemical methods were used to study the susceptibility of PdNi₅ alloy to dealloying.

Keywords: PdNi₅ alloy, corrosion, dealloying, ICP-AES, SEM-EDS

INFLUENCE OF ZINC ADDITION ON ANTI-TARNISH SILVER ALLOYS IN FOUR DIFFERENT SYSTEMS

Marija Korać¹, Stevan Dimitrijević², Kemal Delijić³, Silvana Dimitrijević⁴, Željko Kamberović¹

e-mail: marijakorac@tmf.bg.ac.rs

1-Faculty of Technology and Metallurgy, University of Belgrade, Serbia

2-Innovation Center Faculty of Technology and Metallurgy in Belgrade Ltd, University of Belgrade,

3-Faculty of Metallurgy and Technology, University of Montenegro, Podgorica, Montenegro,

4-Mining and Metallurgy Institute Bor, Serbia

The presented paper investigates the influence of zinc addition on the corrosion characteristics of different sterling silver alloy systems. Open circuit potential measurements, linear polarization resistance method, and potentiodynamic polarization tests were employed to determine the corrosion characteristics of the alloys. The materials were tested in an Artificial Sweat, 0.9% NaCl, and 0.01M Na₂S solutions. Based on the presented results, it could be concluded that zinc has the most significant direct impact. The benefits of zinc addition are especially noticeable in aluminum-containing alloy systems, but only up to 3.7 % of Zn. The best results are achieved for alloy AgCu_{1.9}Zn_{3.7}Al_{1.6}Si_{0.3}.

Keywords: atmospheric corrosion, silver, alloys, anti-tarnish; alloy

INFLUENCE OF COLD ROLLING AND ANNEALING ON THE MECHANICAL AND CORROSION PROPERTIES OF AN AA5182 Al-Mg ALLOY

Miljana Popović¹, Ana Alić², Bojan Gligorijević³, Endre Romhanji¹

e-mail: miljana@tmf.bg.ac.rs

1-Dept. Metallurgical Engineering, Faculty of Technology and Metallurgy, University of Belgrade, Belgrade, Serbia

2-Institute of Chemistry, Technology and Metallurgy, University of Belgrade, Belgrade, Serbia

3-Innovation Center, Faculty of Technology and Metallurgy, University of Belgrade, Belgrade, Serbia

Wrought Al-Mg alloys are an important and promising material for application in the transportation industry primarily due to their favorable strength-to-weight ratio. As a result of their usage in the field of transportation, the weight of vehicle constructions become reduced, resulting in a decrease in fuel consumption and carbon dioxide emissions. In addition to these advantages, one of the important demands on the application of Al-Mg alloys as a lightweight structural material is providing high strength, corrosion resistance, and good surface quality without relief caused by yield point phenomena and serrated flow. Such a good combination of properties can be achieved by a variation and optimization of thermomechanical processing parameters including a degree of deformation and annealing conditions.

In this study, we investigated the effect of cold rolling reductions and different annealing conditions, on the corrosion resistance and mechanical properties of an AA5182 type Al-Mg alloy. The material was industrially produced and supplied by Impol-Seval Aluminium Rolling Mill, Sevojno, Serbia, as hot rolled 12 mm thick plates. Further lab-processing of hot rolled plates included cold rolling with a 40-85% reduction in thickness, and annealing in the range of temperatures 280-350°C, in a different periods of time. Annealed specimens were used for room temperature tensile testing while corrosion testing was performed on the annealed and sensitized specimens. Intergranular corrosion (IGC) susceptibility was determined by nitric acid mass loss test (NAMLT) for the selected states.

The results showed that corrosion resistance, tensile strength, and yield point elongation were mostly affected by a degree of cold deformation. It was found that an increase in cold rolling reduction from 40-85% before annealing causes an improved strength level due to grain size refinement. However, yield point elongation became more pronounced and a tendency towards Lüdering as an undesirable surface appearance was increased. NAMLT testing showed that the material was corrosion-resistant in as-annealed conditions while it became susceptible to intergranular corrosion after sensitization treatment. IGC susceptibility of sensitized specimens increases with cold rolling reduction and a raise of annealing temperature.

Keywords: Al-Mg alloy, degree of deformation, strength, yield point elongation, corrosion resistance

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CORROSION OF CERAMIC-METAL COMPOSITES IN ARTIFICIAL ACID RAIN

Milovan Stoiljković¹, Vladimir Pavkov¹, Gordana Bakić², Aleksa Luković¹, Vesna Maksimović¹

e-mail: vesnam@vinca.rs

1-Vinča Institute of Nuclear Sciences - National Institute of the Republic of Serbia, University of Belgrade, Belgrade, Serbia,
2-Faculty of Mechanical Engineering, University of Belgrade, Belgrade

In this work, the basalt deposits site from southern Serbia was used as a matrix for the composite materials. The corrosion behavior of basalt matrix composites with various ratios of commercial stainless steel 316L (5, 10, 15, and 20 wt.%) and bulk basalt produced by sintering in the air ($t=1060^{\circ}\text{C}$ and $\tau=60$ min) were studied by immersion corrosion technique. The samples were immersed in a solution of artificial acid rain $\text{H}_2\text{SO}_4:\text{HNO}_3 = 9:1$ at $\approx \text{pH } 3$ to measure the concentration of released metal ions using inductively coupled plasma-optical emission spectrometry (ICP-OES). The elements Fe, Cr, Mn, and Ni were monitored, while the others were below the limit of quantification. The concentrations of the metal ions in the solution were studied over 15 weeks. The ions release rate from all the sintered samples was found to follow the Weibull cumulative distribution function (CDF). This means that the release rate changes over time and allows us to calculate the characteristic times of ion release. The changes in the samples were monitored by light microscopy (LM), scanning electron microscopy (SEM), X-ray diffraction (XRD), and weight loss. Sintered basalt shows high resistance to corrosion with a characteristic time of about 18 years for Fe and more than 25 years for Cr and Mn. The weight difference between the initial and after 15 weeks of exposure to the solution (sintered basalt) was not observed. On the other hand, the presence of 5 wt.% steel powder in the sintered basalt accelerates the release of metal ions by about six times. When the steel powder content in the composite materials increases, the metal release rate does not change further, but a weight loss is observed. The characteristic times for Fe, Cr, Ni, and Mn in composite materials are about 5 years. These composite materials can be investigated as materials for using in an acid environment.

Keywords: composite materials, basalt, artificial acid rain, corrosion, Weibull distribution

INFLUENCE OF FUNCTIONAL GROUPS ON THERMODYNAMIC PROPERTIES OF BINARY MIXTURES

Milana Zarić^{1,2}, Mirjana Kijevčanin³, Ivona Radović³

e-mail: milana.zaric@ihm.bg.ac.rs

1-Institute of Chemistry, Technology and Metallurgy, University of Belgrade,

2-Centre of Excellence in Environmental Chemistry and Engineering – ICTM, University of Belgrade,

3-Faculty of Technology and Metallurgy, University of Belgrade

The thermodynamic properties of liquid mixtures depend on the geometry, actually on the shape of the molecules and their ability to pack. In addition, the properties also depend on non-covalent interactions like Van der Waals, electrostatic interactions, hydrogen bonding and π - π interactions. Each functional group in a molecule influences the geometry of the molecule and the intermolecular interactions between molecules. In this work, the functional groups of double bond and -OH group has been investigated and their influence on the properties of mixtures. The properties of mixtures (1) n-hexane + 1-hexanol and (2) n-hexane + cis-3-hexen-1-ol have been investigated both with experimental measurement and with computation molecular modeling. Experimental measurement considered measurements of density, viscosity, refractive index and speed of sound at a range of temperature from (288.15 to 318.15) K and at complete composition range. Excess molar volume and deviations of viscosity, refractive index and speed of sound has been calculated and Redlich-Kister equation has been used for fitting the data. Considering the molecular modeling, the quantum chemical calculations and molecular dynamic simulations have been performed. Different model systems are used order to investigate the -OH group energy interaction and double bond / single bond intermolecular energy interactions. The molecular dynamic simulations have been performed with the liquid mixtures.

Keywords: binary mixtures, themodynamic properties, quantum chemical calculations, molecular dynamic simulations

INFLUENCE OF MOLD PREHEATING ON RAILWAY ALUMINOTHERMIC WELDING CASTING SIMULATION

Gvozden Jovanović¹, Vaso Manojlović², Miroslav Sokić¹, Alen Delić³, Milorad Gavrilovski⁴

e-mail: g.jovanovic@intms.ac.rs

1-Institute for Technology of Nuclear and other Mineral Raw Materials, Belgrade, Serbia

2-University of Belgrade, Faculty of Technology and Metallurgy, Belgrade, Serbia

3-TTU energetik d.o.o., Tuzla, Bosnia and Herzegovina

4-University of Belgrade, Faculty of Technology and Metallurgy, Innovation Center, Serbia

Railway rails have been linked using the aluminothermic welding process for over a century. This method has several advantages, including flexibility, compactness of the weld, and ease of execution. It is not necessary to employ outside energy to complete the procedure. It is provided by the exothermic effect of the chemical reactions of the aluminothermic combination's elements. The design of the mold with the pouring system, which should ensure even pouring of thermal steel without turbulence, then even heat dissipation or cooling in order to obtain an appropriate micro and macro structure of steel, free of internal and external defects, is an important factor in producing the required quality welded joint. As a result, the mold's design was constantly evolving, necessitating the use of costly experimental procedures in industrial settings. In this iteration of the model, the casting cavity was improved by adding hoes in the sand and putting rails on both sides in order to more accurately simulate heat transfer. Software programs are being used to simulate conventional casting procedures that can be used in the casting of thermite steel during the fabrication of welded railway connections in order to prevent costly and time-consuming industrial experimentation. The NovaFlow & Solid CV software package was used to simulate casting thermite steel in the mold cavity, i.e. in the weld joint, for the 49E1 rail.

Keywords: aluminothermic welding, simulation modeling, Novacast, welded joint, preheating influence

NANOSCALE METAL OXIDES AS MATERIALS USED FOR MODIFICATION OF CRBORN-BASED ELECTRODES IN ELECTROCHEMICAL SENSORS

Miloš Ognjanović¹, Biljana Dojčinović², Bratislav Antić¹, Dalibor Stanković^{1,3}

e-mail: miloso@vin.bg.ac.rs

1-University of Belgrade, VINČA Institute of Nuclear Sciences-National Institute of the Republic of Serbia, Belgrade, Serbia,
2-Institute of Chemistry, Technology and Metallurgy, National Institute of the Republic of Serbia, University of Belgrade, Belgrade, Serbia,
3-Faculty of Chemistry, University of Belgrade, Belgrade, Serbia

Nanostructured metal oxides used as modifiers of various carbon-based working electrodes serve as the basis for designing sensitive electrochemical sensors to detect desired analytes. The sensors we develop are distinguished by low detection limit (*LOD*), high analyte selectivity, sensitivity, and versatile real-world sample use case. In this work we present the design of two based on metal oxides as modifiers of carbon paste working electrode (CPE) and their applications in the electrochemical determination of levodopa and adrenaline. The physicochemical properties of designed materials were analyzed by complementary experimental technics (XRPD, TEM, SEM, EDS, electrochemical measurements) to determine their (micro)structural properties and correlate them with electroanalytical performance. Europium has been considered a significant lanthanide element with higher redox reaction behavior. We conducted a hydrothermal synthesis of $\text{Eu}_2\text{O}_3@\text{Cr}_2\text{O}_3$ and used them for CPE modification. The proposed $\text{Eu}_2\text{O}_3@\text{Cr}_2\text{O}_3/\text{CPE}$ electrode was used to develop an analytical procedure quantifying L-Dopa in a wide micromolar linear range (1-100 μM), high sensitivity of $1.38 \mu\text{A} \mu\text{M}^{-1} \text{cm}^{-2}$ and a low detection limit ($LOD = 0.72 \mu\text{M}$). On the other side, we investigated the physicochemical properties of the gallium/bismuth mixed oxides and studied the influence of different $\text{Ga}_2\text{O}_3:\text{Bi}_2\text{O}_3$ ratios on the electrochemical detection of adrenaline. Square wave voltammetry was optimized, and the best electrode showed a wide linear working range of 7-100 μM , under optimized conditions. The *LOD* for the proposed sensor was calculated to be 1.9 μM , with a low limit of quantification ($LOQ = 5.8 \mu\text{M}$). The total performance of the sensors, particularly their performance on real-world samples and their potential for commercialization, had to be carefully evaluated during the sensor construction. Our team is devoted to developing highly selective electrochemical sensors based on nanomaterials to be potentially used as the basis for the fabrication of high-performance miniature devices with exceptional sensitivity to specific analytes, like adrenaline and L-Dopa, in this research.

Keywords Europium/chromium oxide, gallium/bismuth oxide, electrochemistry, levodopa, epinephrine

DEVELOPMENT OF SUSTAINABLE METHOD FOR METAL RECOVERY FROM OLD FLOTATION TAILINGS (MAJDANPEK, SERBIA) USING *Aspergillus niger* FUNGUS

Dušan Milojkov¹, Miroslav Sokić¹, Angelina Mitrović¹, Danijela Smiljanić¹, Jelena Petrović¹, Marija Simić¹,
Vukosava Živković-Radovanović²

e-mail: d.milojkov@itnms.ac.rs

1-Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia,

2-Faculty of Chemistry, University of Belgrade, Belgrade, Serbia

The accumulation of mine tailings in the mining area of Majdanpek represents a serious environmental problem. A strong impetus for the development of sustainable methods for the recovery of metals from mine tailings is given by the importance of heavy metal recovery and the economic benefits of precious and base metals. Currently, researchers are working to develop metals recovery method from tailings through bioleaching, which is a more sustainable approach compared to traditional metallurgical methods. Biotechnologies for leaching heavy metals from old flotation tailings (Majdanpek, Serbia) with the fungus *Aspergillus niger* are discussed in this paper. The subject of this research is iron ions since the tailings are the richest in this metal. With a simple bioleaching process, which is based on the cultivation of *A. niger* in the basic growth medium and then challenging it with tailings, 15% iron was released in 35 days. Processes by which *A. niger* extracts metals from tailings include acidolysis, complexation, alkalolysis, and biosorption. Also, in addition to metabolites, these fungi produce spores and mycelium that can destroy material and transform it into soil. Given that Serbia is abundant with agricultural waste, as well as waste from the food industry, in future studies cheap waste should be used as an energy input source for *A. niger* and the transformation of tailings.

Keywords: bioleaching, flotation tailings, *Aspergillus niger*, sustainable development, Majdanpek.

APPLICATION OF SUSTAINABLE MATERIALS IN THE NO_x REDUCTION OF AIR POLLUTION

Nemanja Barac¹, Katarina Dimić-Mišić², Monireh Imani², Petar Uskoković³, Ernesto Barceló^{2,4}, Patrick Gane^{2,3},
Dorđe Janačković³

e-mail: nbarac@tmf.bg.ac.rs

1-Innovation Center of Faculty of Technology and Metallurgy in Belgrade Ltd., Karnegijeva 4, 111200 Belgrade, Serbia,
2-School of Chemical Engineering, Department of Bioproducts and Biosystems, Aalto University, 00076 Aalto, Helsinki, Finland,
3-Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4, 111200 Belgrade, Serbia,
4-Environmental, Social and Governance, Gestamp Automoción S.A., Torre Ombú n°3, Planta 1, 28045 Madrid, Spain

According to the World Health Organisation (WHO), in 2019, 4.2 million premature deaths worldwide were estimated as a consequence of ambient (outdoor) air pollution. Of major interest, especially in city environments, and increasingly inside vehicles or industrial plants, is the drive to reduce human exposure to oxides of nitrogen (NO_x). A possible solution to achieve proof of principle in the development of filter element structure models may be based on the chemical reaction between Calcium Carbonate (CaCO₃) and ambient NO_x. Calcium Nitrate (Ca(NO₃)₂), as a product of a chemical reaction, can be further used as a fertilizer that provides plants with both calcium and nitrogen, two essential nutrients for plant growth. The filter element (sheets) is formed by pin/needle coating ground CaCO₃ (GCC-produced from Norwegian marble, wet ground chemical-free Omya Hustadmarmor AS, Molde, Norway), on a porous carrier. The active component carrier (substrate) designed for the filter application is based on forming a paper-like sheet from 'over-recycled' cellulose fibre, *i.e.* paper and board waste in which the fibre content has already been weakened due to manifold previous recycling. It is known that the heterogeneous reaction of NO_x with CaCO₃ depends on the presence of moisture, so, to ensure that, some humectant material that absorbs and stores moisture in excess of its own matrix dispersion needs to be included. Such a material is conveniently sourced from the same feed material used as the substrate, *i.e.* cellulose fibers, in this case in the form of micro nanofibrillated cellulose (MNFC). After use, the nitrate-rich GCC-cellulose-based filter, proposed as a soil fertiliser and micronutrient, represents sustainable material sourcing pre and post-initial use as one of the key targets for industrial practice today within the circular economy. This application of air mining technology has the potential to provide a sustainable solution for both, air pollution reduction and agriculture.

Keywords: NO_x mitigation, Calcium Carbonate, Filter Sheets, Micro-nanofibrillated Cellulose

DESIGN OF A PILOT PLANT FOR CHEMICAL TREATMENT OF THE SPENT MINERAL OIL-IN-WATER EMULSION FROM NON-FERROUS METAL PROCESSING

Vesna Lazarević¹, Ivan Stojković², Ivana Banković-Ilić³, Vlada Veljković^{3,4}

e-mail: istojkovic@tmf.bg.ac.rs

1-Military Hospital Niš, Centre for Preventive Medical Care, Niš, Serbia,
2-Innovation Center, Faculty of Technology and Metallurgy, Belgrade, Serbia,
3-Faculty of Technology, Leskovac, University of Niš, Serbia,
4-Serbian Academy of Science and Arts, Belgrade, Serbia

A pilot plant having a capacity of 200 m³ per year was designed to chemically treat the spent mineral oil-in-water emulsion (SMOWE) from non-ferrous metal processing. The process involves spent emulsion equalization, secondary oil separation, coagulation/flocculation with metal ion precipitation, sedimentation of flocculated particles (clarification), filtration of clarified liquid, sludge conditioning, sludge dehydration by filtration, neutralization of treated wastewater, and solid waste disposal. First, the spent emulsion is decomposed through a combined effect of a flocculating agent (hydrated aluminum(III) sulfate, 1 kg/m³ SMOWE) that destabilizes the emulsion and a base (slaked lime, 3 kg/m³ SMOWE) that favors the formation of aluminum(III) hydroxide and precipitates sulfate and some metal (zinc and copper) ions. After the flocculated particle sedimentation, the clarified liquid is separated and filtered through a sand filter while the sludge is conditioned by adding a filter aid and wood sawdust (1 and 5 kg/m³ SMOWE, respectively), filtered under pressure (3 bar) to remove excess water, dries in the open air, and disposed of. Finally, the clarified liquid from sedimentation and the filtrate from sludge conditioning are mixed and then diluted (1:10) with an acid wastewater stream to reduce the pH to 6.5–8.5. By balancing the overall treatment process at the pilot level, it was determined that the quantities of the purified wastewater and solid waste (wet filter cake with about 50-55% water) amount to about 98% and 2% of the amount of SMOWE, respectively.

Keywords: balancing, chemical treatment, non-ferrous metal processing, spent oil-in-water emulsion, wastewater

EFFECTS OF MULTIPLE WELDING DEFECTS ON MECHANICAL PROPERTIES OF WELDED JOINT

Stefan Dikić¹, Mihajlo Arandelović^{2,3}, Simon Sedmak², Radomir Jovičić², Dragomir Glišić¹

e-mail: sdikic@tmf.bg.ac.rs

1-Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4, 11120 Belgrade,

2-Innovation Centre of Faculty of Mechanical Engineering, Kraljice Marije 16, 11120 Belgrade,

3-Faculty of Civil Engineering, Transilvania University of Braşov, Turnului Street 5, 500152 Braşov, Romania

The aim of this work was to establish the influence of multiple welding defects on the structural integrity of low-carbon low-alloyed steel welded joints. Defects in welded joints (misalignments, incomplete root penetrations, excess weld metal and undercuts) were made intentionally in order to simulate the most common defects that occur in everyday welding practice. Samples were made of S275JR carbon steel plates, welded with MAG technology, with VAC60 used as filler material. Welded joints with and without defects were investigated by means of tensile testing, metallographic examination and hardness testing. Also, both surface and volume non-destructive testing methods were performed. Metallographic observation of welded joints has shown uniform microstructure of the base metal (BM), as well as uniform microstructure in the heat-affected zone (HAZ) and weld metal (WM). Hardness testing results were analyzed in this paper in detail and were determined to be in accordance with observed microstructures. Hardness values in HAZ show no significant deviation from the face to the root side of the weld. Welding defects do not significantly affect the microstructure of HAZ and obtained hardness values in HAZ. Despite homogeneous microstructure and hardness distribution, tensile testing results revealed a significant influence of the welding defects on stress distribution over the welded joint which leads to a decrease of structural integrity.

Keywords: welded joint defects, carbon steel, strength, hardness

CHARACTERIZATION OF FRICTION STIR WELDING JOINTS IN 7075-T6 ALLOY PLATES

Nebojša Tadić¹, Mitar Mišović¹, Žarko Radović¹, Ratka Petrović¹

e-mail: nebojsa@ucg.ac.me

¹-Faculty of Metallurgy and Technology, University of Montenegro, Podgorica, Montenegro

The paper presents the results of testing macro and microstructure, hardness, and impact toughness of 7075-T6 alloy plate joints welded by the Friction Stir Welding (FSW) process. Characteristic zones are identified in the cross-sectional macrostructure of the FSW joint. As a result of dynamic recrystallization in the nugget zone (NZ), a fine equiaxial microstructure was formed. In the thermomechanically affected zone (TMAZ) there are unrecrystallized deformed grains elongated in the direction of metal flow. The microstructure in the heat-affected zone (HAZ) is close to the base metal. The hardness in the nugget zone is reduced to between 70% and 84% of the hardness of the base metal, while the impact toughness in the nugget zone is 2.5 times higher than that of the base metal.

Keywords: 7075-T6 plate, friction stir welding, microstructure, hardness, impact toughness

SEQUENCING BATCH REACTOR SYSTEMS FOR THE TREATMENT OF WASTEWATER

Aleksandar Jovanović¹, Mladen Bugarčić¹, Marija Stevanović², Miroslav Sokić¹, Aleksandar Marinković³

e-mail: a.jovanovic@itnms.ac.rs

1-Institute for Technology of Nuclear and Other Mineral Raw Materials, Franš d' Eperea 86, Belgrade, Serbia,

2-Institute of Pesticides and Environmental Protection, Banatska 31b, Belgrade, Serbia,

3-University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, Belgrade, Serbia

Releasing untreated or inadequately treated effluents into recipients (rivers, lakes, and oceans) can endanger aquatic ecosystems and drinking water sources. In order to eliminate harmful pollutants, wastewater must be treated, especially industrial wastewater enriched with nutrients. Phosphorus contamination can occur through a variety of different routes, such as wastewater treatment plant discharge (municipal and industrial), agricultural fertilizer losses from erosion, drainage, etc. The establishment of suitable wastewater treatment techniques is essential. The subject of this paper is the biological removal of nutrients (phosphorus) from wastewater, using unconventional methods of wastewater treatment - sequencing batch reactors (SBR) systems. Their modifications and the advantages such processes offer, compared to the standard activated sludge (AS) treatment that is the most often used, will be presented. Enhanced biological phosphorus removal methods that employ various microorganisms have been observed from the perspective of the method's effectiveness. Of all applied treatments for phosphorus removal, the most effective and the most often used was the accumulation of polyphosphates in the biomass itself. The purpose of this paper is to offer a brief review and theoretical essentials of environmental engineering techniques that can be applied in real industrial wastewater treatment plants

Keywords: environmental pollution, biological treatment, nutrient removal, sequencing batch reactors

Mg/Fe-PYRO-HYDRO CHAR DERIVED FROM CORN COB AS EFFECTIVE ADSORBENT OF LEAD REMOVAL FROM AQUEOUS SOLUTIONS

Marija Simić¹, Jelena Petrović¹, Marija Ercegović¹, Marija Koprivica¹, Jelena Dimitrijević¹, Aleksandar Jovanović¹, Dušan Milojković¹

e-mail: m.petrovic@itnms.ac.rs

¹Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia

The corn cob was investigated as available agro-waste material for the production of potentially efficient material for heavy metals removal. The hydrothermal carbonization (HTC) technology is one of the appropriate methods for biomass transformation into high-value carbonaceous products that can be utilized as adsorbents. In this study, modified pyro-hydrochar derived from corn cob (MCC) was effectively prepared by modification with Mg-Fe solution and pyrolysis. This material was used for Pb ions removal from aqueous solutions. The effect of solution pH, contact time, and initial Pb concentration were examined in a batch system. The achieved results revealed that the most effective Pb adsorption takes place at pH 5. The experimental results were fitted to Langmuir, Freundlich, Sips, and Redlich-Peterson isotherm models. The best data fit was achieved with the Sips isotherm model with maximum adsorption capacity for Pb removal of 214.9 mg/g. Additionally, the experimental results from kinetic study were fitted by pseudo-first and pseudo-second order models. According to kinetic parameters, the Pb removal using MCC follows pseudo-second order model, which assumes that chemical interaction between Pb ions and functional groups on the MCC surface was involved in metal adsorption. According to data from this investigation and in comparisons to other adsorbents can be concluded that investigated material can be used as potentially suitable adsorbent of Pb from aqueous solutions.

Keywords: hydrothermal carbonization, modification, corn cob utilization, heavy metals removal, adsorption.

THE ADSORPTION OF ARSENATE IONS FROM AQUEOUS SOLUTIONS BY COMPOSITE PARTICLES OF NATURAL POLYMERS AND METAL OXIDES

Janko Živanić¹, Mladen Bugarčić², Anja Antanasković², Zorica Lopičić², Miroslav Sokić², Aleksandar Jovanović², Milan Milivojević¹

e-mail: mmilan@tmf.bg.ac.rs

1-Department of Chemical Engineering, Faculty of Technology and Metallurgy, University of Belgrade, Karnegijeva 4, Belgrade, Serbia,

2-Institute for Technology of Nuclear and Other Mineral Raw Materials, 86 Bulevar Franš d'Eperea Street, Belgrade, 11000, Serbia

The presence of arsenic in drinking water above the permissible limits is one of the current problems facing modern engineers in the field of separations and it has not yet been satisfactorily resolved, because the permissible concentrations of arsenic in drinking water are constantly reduced by the World Health Organization (WHO). Among the various processes for arsenic removal, adsorption has a special place as one of the most efficient and cheap process. Many natural and artificial materials are tested for adsorption processes, and adsorption on metal powders is particularly interesting because they represent adsorbents with fast kinetics and relatively high adsorption capacities. However, the application of powder adsorbents is difficult due to their separation from the mixture after adsorption. In order to solve that problem, metal powders may be immobilized into particles obtained by gelling natural polymers. In this way, it is easier to separate the adsorbent from the mixture, however this must be achieved without losing the adsorption capacity and rate compared to pure metal powders.

In this study, the use of metal oxide (iron oxide nanoparticles) immobilized within the alginate gel showed good potential for arsenic (V) ions removal. The obtained results showed that arsenic adsorption by obtained composite has fast kinetic, following pseudo-second-order model, and that it is conducted in several steps, according to the Weber-Morris model. It was suggested that external diffusion is the dominant mechanism at the beginning of the arsenic adsorption, followed by intraparticle diffusion. The obtained results confirmed that gelling with alginate did not significantly affect the adsorption process, that is, it does not interfere with the adsorption capacity of metal oxides, which is a very important factor. Arsenic (V) removal was successfully performed using the investigated composite.

Keywords: arsenate removal, alginate, goethite, nanoparticles, composite.

CLAY-CHITOSAN-SURFACTANT COMPOSITES AS EFFICIENT ADSORBENTS OF ZEARALENONE

Danijela Smiljanić¹, Aleksandra Daković¹, Milena Obradović¹, Marija Marković¹, Milica Ožegović¹, Milena Pantić²,
Danina Krajišnik³

e-mail: d.smiljanic@itnms.ac.rs

1-Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia,

2-Institute for Food Technology and Biochemistry, University of Belgrade, Faculty of Agriculture, Belgrade, Serbia,

3-Department of Pharmaceutical Technology and Cosmetology, University of Belgrade – Faculty of Pharmacy, Belgrade, Serbia

Mycotoxins are secondary metabolites of fungi commonly found in cereal grains and animal feed. The most frequently found mycotoxins are: aflatoxins, ochratoxin A, zearalenone, and fumonisins. Consumption of contaminated feed and food can cause serious health problems in animals and humans. Various chemical, physical, and biological methods have been used to remove mycotoxins from contaminated animal feed. Among physical methods, adsorption has been widely used for decontaminating animal feed from mycotoxins. Natural aluminosilicates, like zeolites and/or clays, are commonly utilized as adsorbents because they are abundant, inexpensive, and environmentally acceptable. Among clays, bentonites have proven to be excellent adsorbents, but only for aflatoxin B1. However, the adsorption of other less polar mycotoxins - ochratoxin A and zearalenone (ZEN) by natural minerals is low, so to increase their adsorption, these minerals are usually modified with long-chain cationic surfactants. Recently, composites of natural biopolymers (chitosan and/or alginate) and clays have been extensively studied as adsorbents for many weakly polar molecules, dyes, phenols, and even aflatoxin B1. Chitosan is a particularly interesting biomaterial due to its biocompatibility, biodegradability, antimicrobial activity, and adsorption properties. Nanocomposites based on chitosan exhibit significant structural and functional properties, which, when combined with their biocompatibility and biodegradability, could find application in animal nutrition, wastewater treatment, and a variety of biomedical fields.

In this work, a non-swelling Ca-bentonite (clay) from the Beretnica deposit in the Republic of Serbia (BENT) was modified with commercial low molecular weight chitosan (Sigma-Aldrich Co.) and chitosan isolated from fruit bodies of commercially grown mushroom *Agaricus bisporus* (Delta Danube d.o.o., Kovin, Republic of Serbia). The obtained materials were subsequently modified with long chain organic cation (surfactant) – hexadecyltrimethyl ammonium bromide. The amount of surfactant used for modification of clay-chitosan composites was equal to 50% of CEC of starting clay. Clay-chitosan composites as well as clay-chitosan-surfactant composites were characterized by Fourier transform infrared (FTIR) spectroscopy. Results of FTIR analysis confirmed presence of surfactant in both clay-chitosan-surfactant composites. Clay-chitosan composites and clay-chitosan-surfactant composites were preliminary tested as adsorbents for ZEN, under *in vitro* conditions. The adsorption of ZEN was investigated with starting ZEN concentration of 4.0 mg/L, with 10 mg/10 mL of each adsorbent, at pH 3. Suspensions were stirred for 30 min, then centrifuged at 13 000 rpm for 3 min. ZEN concentrations in supernatants were determined by high performance liquid chromatography (HPLC). Results indicated that both clay-chitosan-surfactant composites showed high adsorption of ZEN, while adsorption of this mycotoxin by clay-chitosan composites was low. Adsorption of ZEN by clay-chitosan-surfactant composites was further studied with different amounts of each adsorbent (20, 10, 5, and 2 mg/10 mL), at pH 3. It was shown that adsorption of ZEN increased with increasing the amount of both composites, indicating that although composites contain surfactant ions onto which ZEN is adsorbed, materials also possess biocompatible and biodegradable substance – chitosan with known antimicrobial activity. The fact that adsorption of ZEN by composites containing commercial chitosan and chitosan isolated from mushroom is similar, opens the possibility for using the fungi in preparation of clay-chitosan-surfactant composites as new animal feed additives.

Keywords: clays, chitosan, surfactants, zearalenone, adsorption

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HYDROCHAR-DERIVED HIGHLY EFFICIENT ADSORBENT FOR COPPER IONS REMOVAL

Jelena Petrović¹, Marija Ercegović¹, Marija Simić¹, Marija Koprivica¹, Marija Marković¹, Jelena Dimitrijević¹

e-mail: j.petrovic@itnms.ac.rs

¹Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia

Within this study, the valorization of waste grape pomace hydrochar was investigated. Emphasis will be directed toward the potential application of hydrochar as an adsorbent of copper ions from aqueous solutions. In order to further enhance the adsorption potential, the obtained hydrochar was doped with Mg and Fe salts, followed by pyrolysis at 300°C. The resulting material, a novel Mg/Fe-pyro-hydrochar was applied in further adsorption tests and compared with native hydrochar. Preliminary adsorption results revealed that the modified pyro-hydrochar show significantly higher adsorption capacity (71 mg/g) compared to the unmodified (14 mg/g). In order to gain insight into the maximum adsorption capacity and the nature of the interaction, Mg/Fe-pyro-hydrochar was subjected to an examination of the influence of the initial pollutant concentration, isothermal study, and spectroscopic analysis. The obtained results revealed that the isotherm equilibriums were determined by the Sips isotherm model, with a maximum theoretically achieved capacity of 75 mg/g. Besides, binding of copper ions to the surface of the Mg/Fe-pyro-hydrochar was reached by a complex mechanism that includes electrostatic interaction, π - π interaction, as well as surface complexation with oxygen-containing functional groups. Summarized results from this study reveal that one-step hydrothermal carbonization followed by pyrolysis is a promising method for the production of efficient adsorbents for copper removal.

Keywords: Copper removal, Hydrothermal carbonization, Pyrolysis, Hydrochar modification

INNOVATIVE INDUSTRIAL SYMBIOSIS APPROACH FOR ACIDIC MINE DRAINAGE NEUTRALIZATION

Nela Vujović¹, Vesna Alivojvodić², Miroslav Sokić¹, Gvozden Jovanović¹, Branislav Marković¹, Željko Kamberović³

e-mail: n.petronijevic@itnms.ac.rs

1-Institute for Technology of Nuclear and Other Mineral Raw Materials (ITNMS), Belgrade, Serbia,

2-The Academy of Applied Technical Studies Belgrade,

3-Faculty of Technology and Metallurgy, University of Belgrade, 11000 Belgrade, Serbia

Following the principles of sustainable development and circular economy, an innovative industrial symbiosis approach has been developed that uses already disposed voluminous waste. The research was focused on the utilization potential of vast amounts of flotation tailings, fly ash and acidic mine waters. Numerous previous case studies have pointed out the harmful, long-lasting environmental damage from waste disposal. In this case, the emphasis is on finding a practical approach for using industrial waste, specifically thermal power plant fly ash, in order to neutralize acidic mine waters resulting from tailings disposal. The proposed treatment gives waste a practical value, eliminates its harmful effect on the environment and redefines (lowers) regular waste management costs. In addition, it is possible to obtain valuable metals by exploiting and reprocessing acidic mine drainage.

Keywords: acidic mine drainage, flotation tailing, circular economy, fly ash

REMOVAL OF MESOTRIONE BY HETEROGENEOUS PHOTOCATALYTIC TREATMENT USING UV-VIS LAMP AS LIGHT SOURCE

Jovana Bošnjaković¹, Nataša Knežević², Aleksandar Marinković³, Srećko Manasijević¹, Andrija Savić², Milena Milošević⁴

e-mail: jovana.bosnjakovic@li.rs

1-Research and Development Institute Lola L.T.D., Kneza Višeslava 70A, 11030 Belgrade, Serbia,

2-University of Belgrade, "VINČA" Institute of Nuclear Sciences - National Institute of the Republic of Serbia, Mike Petrovića Alasa 12-14, 11351 Belgrade, Serbia,

3-University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, 11120 Belgrade, Serbia,

4-University of Belgrade, Institute of Chemistry, Technology and Metallurgy, National Institute of the Republic of Serbia, Njegoševa 12, 11000 Belgrade, Serbia

Population growth, the development of agriculture, industry, and mining caused the creation of an increasing amount of wastewater. Due to the discharge of wastewater, without prior treatment, the quality of water resources is impaired. Polluting substances, such as pesticides, have a negative impact on human health and the environment. Heavy pollution of both surface and underground water is one of the biggest problems associated with the use of pesticides. They reach human organisms indirectly via agricultural products. Most pesticides are more or less toxic, and some of them are highly soluble in water. In recent years, special attention has been paid to the development of methods for the treatment of wastewater contaminated with pesticide residues, in order to partially reduce or eliminate their further impact on humans, plants, and the environment. Among many processes, the photocatalytic degradation process has proven to be a very effective and inexpensive process for the removal of pesticides from wastewater. Photocatalytic degradation implies the breakdown of various types of toxic organic substances into simpler molecules, such as ions, water, etc.

In this paper, the photocatalytic degradation of the pesticide mesotrione (MS) using ZnO photocatalyst is presented. The degradation of the pesticide compound was studied using Shimadzu 1800 analytical UV-VIS spectroscopy. As a replacement for UVC radiation, a solar-imitated Ultra Vitalux (UV) lamp (300W) was used. Degradation kinetics follows pseudo-first order. After 240 minutes, the MS was completely degraded.

Keywords: mesotrione, wastewater, UV-VIS, photocatalytic degradation, environment

TESTING OF THE ADSORPTION-DEGRADATION CAPACITY OF CRYSTAL VIOLET DRY WITH OXIDIZED COTTON LINTERS

Nataša Knežević¹, Jovana Bošnjaković², Marija Vuksanović¹, Jovana Milanović³, Ljiljana Janković-Mandić¹,
Adela Egelja¹, Aleksandar Marinković⁴

e-mail: natasa.knezevic@vin.bg.ac.rs

1-University of Belgrade, “VINČA” Institute of Nuclear Sciences - National Institute of the Republic of Serbia, Mike Petrovića

Alasa 12-14, 11351 Belgrade, Serbia,

2-Research and Development Institute Lola L.T.D., Kneza Višeslava 70A, 11030 Belgrade, Serbia,

3-Innovation Center of the Faculty of Technology and Metallurgy in Belgrade d.o.o, Karnegijeva 4, 11120 Belgrade, Serbia,

4-University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, 11120 Belgrade, Serbia

The textile and dyeing industries are considered to be major industries that use water and produce a huge amount of toxic effluents in the final processing and dyeing processes. In order to prevent negative consequences on the environment and human lives, it is necessary to apply technological and ecologically acceptable methods of disposing of pollutants.

In this work, functionalized cotton linters were chosen as a renewable source for the removal of crystal violet dye (CV, $C_0=20 \text{ mg L}^{-1}$) using a batch technique. The efficiency of oxidation on cotton linters by 2,2,6,6-tetramethylpiperidin-1-yl-oxyl (TEMPO radical) in the adsorption of CV was carried out as a function of pH value, adsorbent dose, contact time, mixing and initial pollutant concentration. Adsorption isotherms such as Langmuir, Temkin, and Freundlich were investigated with the aim of understanding the mechanism of adsorption of the colored molecule. The obtained maximum adsorption capacity was $107,025 \text{ mg g}^{-1}$, where a pseudo-second-order kinetic equation with an R^2 value of 0.9953 was found. After desorption dye was subjected to photocatalytic degradation accelerated by adding ZnO as a catalyst. The whole process takes place under the action of an Ultra Vitalux (UV) lamp (300W) that imitated solar irradiation. Under optimal conditions (0.05 gL^{-1} of catalyst), the degradation of CV (97%) takes place for 130 min. Quantum yield measurement and UV-Vis analysis results helped to understand the degradation pathways. To identify the chemical and morphological characteristics, the modified cotton linters (TOCott) were characterized using emission scanning electron microscopy (SEM) and Fourier transform infrared spectrophotometry (FTIR).

The obtained adsorption results indicate that the prepared TOCott can be used as an environmentally acceptable material for removing colored adsorbent from aqueous solutions that are easily photodegraded after desorption.

Keywords: TEMPO-oxidized cotton, adsorption, crystal violet, azo reactive dye, photodegradation

MECHANOCHEMICAL SYNTHESIS AND CHARACTERIZATION OF THE ADSORBENTS BASED ON NATURAL ZEOLITE AND HYDROXYAPATITE

Katarina Sokić¹, Jelena Dikić², Đorđe Veljović¹, Jovana Đokić³, Zoran Anđić³, Sanja Jevtić¹

e-mail: djokic@chem.bg.ac.rs

1-Faculty of Technology and Metallurgy, University of Belgrade, Belgrade, Serbia,

2-Innovation Centre of the Faculty of Technology and Metallurgy Ltd, University of Belgrade, Belgrade, Serbia,

3-Innovative Centre of the Faculty of Chemistry Ltd, University of Belgrade, Belgrade, Serbia

Natural zeolite is a good sorbent for many cations due to its specific porous structure. The degree of sorption efficiency depends on the cation type and the availability of exchange positions in the zeolite lattice. The aim of this work is to examine the synthesis possibility of a new adsorbent based on natural zeolite and hydroxyapatite. The adsorbent was prepared mechanochemically by using clinoptilolite-rich zeolite tuff from the Slanci deposit (Serbia) and hydroxyapatite obtained by the hydrothermal process. The milling process was performed in a ball mill and optimized regarding contact time and milling speed. For the synthesis of zeolite/hydroxyapatite adsorbent (ZHAp), optimal values for these two parameters were 10 min and 250 rpm, respectively. The resulting sample ZHAp was characterized by X-ray powder diffraction, thermal analysis and scanning electron microscopy. Diffraction analysis of the ZHAp confirmed the presence of the most abundant mineral in zeolite tuff - clinoptilolite and hydroxyapatite. Needle-like crystals of hydroxyapatite on the clinoptilolite surface are observed in SEM images. Zeolite/hydroxyapatite sample is thermally stable at 800 °C. Adsorption properties of ZHAp were examined for nickel and chromium ions, towards which natural zeolite shows a low affinity. The presence of hydroxyapatite on the zeolite surface led to an increase in the adsorption capacity more than two times for both tested ions compared to the initial zeolite tuff.

Keywords: mechanochemical synthesis, natural zeolite, hydroxyapatite, adsorption

NICKEL RECOVERY FROM STATIC RINSING IN THE ELECTROPLATING PROCESS

Borislav Malinović¹, Anđela Bojić¹, Draženko Bjelić¹, Tijana Đuričić¹

e-mail: borislav.malinovic@tf.unibl.org

1-University of Banja Luka, Faculty of Technology, Stepe Stepanovića 73, 78000 Banja Luka, Bosnia and Herzegovina

Wastewater from the electroplating process can contain heavy metals in high concentrations and, if released into the environment, can cause serious consequences for the environment and human health, due to its toxicity and biological accumulation. High amounts of Ni²⁺ ions are produced during drag-out and rinsing in the nickel plating process and end up in wastewater. Recovery (recycling) of nickel from such water is desirable both from an economic and ecological point of view. In this research, nickel was recovered from static rinsing wastewater after nickel plating by the cathodic electrodeposition process. The concentration of nickel in this water was 34.3 g/L. Using the Hull cell, the most important parameters affecting the process were determined: current density and temperature. Also, the influence of stirring, the type of anode material and the surface ratio of the anode and cathode were examined. The electrodeposition process was performed in a batch laboratory electrochemical reactor with the possibility of constant stirring. During different treatment times, the mass yield of nickel on the cathode was monitored by the gravimetric method and process parameters were calculated: current efficiency and specific energy consumption. It was found that with an increase in the treatment time, the yield on the cathode increases linearly. The results showed that the highest current efficiency was at a current density of 2.5 A/dm². For 60 minutes of electrodeposition treatment, 2.59 g of nickel was recovered per liter of wastewater, on the cathode surface of 0.5 dm², while the energy consumption was 6.71 kWh/kg of recovered nickel.

Keywords Environmental protect, metal recovery, cost benefits

**PHOTOCATALYTIC DEGRADATION OF CONGO RED DYE USING
UiO-66 MOF-METAL OXIDES COMPOSITES**

Dimitrije Petrović¹, Marija Egerić¹, Yi-nan Wu^{2,3}, Aleksandar Vukadinović¹, Aleksandar Devečerski¹,
Ljiljana Matović^{1,2}, Radojka Vujasin¹

e-mail: dimitrije.petrovic@vin.bg.ac.rs

1-Vinča Institute of Nuclear Sciences, National Institute of the Republic of Serbia, University of Belgrade, Mike Petrović Alasa 12-14, Vinča, Belgrade, Serbia

2-College of Environmental Science and Engineering, State Key Laboratory of Pollution Control and Resource Reuse, Tongji University, Shanghai 200092, P.R. China

3-Shanghai Institute of Pollution Control and Ecological Security, Shanghai 200092, P.R. China

This study aimed to investigate the degradation efficiency of Congo red dye using metal-organic frameworks (MOFs), metal oxides (MeOs), and their composites. The possibility of reusing the composites multiple times was also evaluated. Congo red is a commonly used dye in the textile industry and its release into the environment can have harmful effects on aquatic life and human health. MOF used in this study was UiO-66, which has a high surface area and tunable pore size, making it a promising candidate for environmental remediation applications. Microstructure and morphology of as received materials and a composite which performed the best degradation performance were characterized using XRD, FTIR and BET. Removal efficiency of Congo red dye was evaluated using UV-vis spectroscopy. Co_3O_4 and CuO oxides were chosen because of their suitable band gaps (2.07 and 1.4 eV, respectively) which falls within the favorable band gap range for visible light absorption, contrary to the UiO-66, whose band gap is ≈ 4 eV. Co_3O_4 -MOF composite demonstrated better catalytic activity than CuO-MOF composite. Results showed that Co_3O_4 used alone had the least effect on the degradation of the dye, while the composite of MOF and Co_3O_4 had the highest percentage of dye degradation. The composite degraded 80% of the dye within 2 hours of the reaction, whereas MOF alone degraded only 47% of the dye. When Co_3O_4 was used alone, no degradation of the dye was observed within the same period. The possibility of reusing the composite was also evaluated, and the degradation of the dye using Co_3O_4 -MOF composite was observed through three cycles. This study provides insight into the potential of MOF- Co_3O_4 composites for environmental remediation applications, specifically for the degradation of Congo red dye. These findings suggest that the MeO-MOF composites could be an effective alternative to UiO-66 alone for the degradation of dyes in wastewater treatment. The use of MOFs and their composites with metal oxides could provide a promising approach for the removal of harmful pollutants from wastewater, contributing to the preservation of our environment and the protection of human health.

Keywords MOF, UiO-66, metal oxide, dye degradation, composite, wastewater treatment

DETECTION AND SENSING OF BENZENEDIOLS USING MODIFIED SCREEN PRINTED ELECTRODES

Abhilash Krishnamurthy^{1,2}, Špela Trafela¹, Kristina Žagar Soderžnik^{1,2}

e-mail: abhilash.krishnamurthy@ijs.si

1-Department for Nanostructured Materials, Jožef Stefan Institute, Jamova cesta 39, 1000 Ljubljana, Slovenia,

2-Jožef Stefan International Postgraduate School, Jamova cesta 39, 1000 Ljubljana, Slovenia

Volatile toxic Organic compounds (VOC) have started to pick up public awareness as one of the leading causes of common acute and chronic illnesses in people. Conditions ranging from relatively simple ailments to the extremes of potentially lethal conditions have been linked to repeated and/or continuous exposure to VOC's. While they are easily detected at the lab scale, real time detection in dynamic environments presents a challenge that requires the robustness and reliability of the lab instruments to be replicated in smaller, more cost- and power-efficient and technically simpler packages. Screen Printed Electrodes promise a viable alternative for detecting and sensing VOC's in real time. The particular focus VOC's will be the three benzenediol isomers, catechol resorcinol and hydroquinone. Electrochemical techniques such as cyclic voltammetry (CV) for detection of single isomer, differential pulse voltammetry (DPV) for, detection of multiple isomers and chronoamperometry (CA) for sensing in real time were performed. Sensing ranges were obtained for all isomers and practical limits of quantification were obtained as follows: Catechol: 1 μ M, Resorcinol: 100 nM and Hydroquinone: 1 μ M

Keywords: electrochemical sensor, screen printed electrode, benzenediol, catechol, resorcinol, hydroquinone, carbon-supported catalyst

MACHINABILITY OF Ti₆Al₄V IN HIGH SPEED MICRO TURNING UNDER DRY CONDITION

Prince Anand¹, Arnab Das¹, Vivek Bajpai¹

e-mail: 9550anand@gmail.com

¹-Department of Mechanical Engineering, Indian Institute of Technology (ISM) Dhanbad, Jharkhand, 826004, India

The abstract, Ti6Al4V alloy is increasingly being used in biomedical, aerospace, and automotive applications due to its outstanding corrosion resistance and increased specific strength. Due to high chemical reactivity, lower thermal conductivity, and low elastic modulus, the machinability of Ti6Al4V is very poor causing high tool wear as well as surface roughness. Micro turning is a technology to fabricate miniaturized cylindrical components however the poor material removal rate promotes the requirement of high-speed micro turning. In this work, high-speed micro turning in dry conditions have been performed on workpiece of 3mm diameter with a constant feed rate (2 μm/rev), constant depth of cut (50 μm), and two levels of cutting speeds (10000, 15000 rpm). Two different combinations of cutting speed have been used because during high-speed operation, rapid spark was generated as the heat completely accumulated in the chip tool interface carried out by the chips and the tendency of spark has been increased at a higher cutting speed which might damage the machine tool. Investigations have been performed on the topography of the surface, the burr formation, and tool wear. A surface roughness of 0.592 microns has been achieved during the experiment at a lower cutting speed however when we increase the cutting speed the surface roughness has been increased due to rapid heat accumulation. Additionally, rapid crater and flank wear have been observed on the cutting tool. The tendency of exit burr formation has been increased at a higher cutting speed. All of these phenomena are expediting the requirements of cooling technologies during high speed-micro turning of Ti6Al4V.

Keywords: High speed micro turning, Ti6Al4V, Tool wear, Surface Roughness

ANALYSIS OF HOLE QUALITY IN MICRO DRILLING OF METAL-OXIDE NANOSTRUCTURED CFRP COMPOSITES

Abhipsa Kar¹, Vivek Bajpai¹, Ravi Shankar Rai¹

e-mail: 22dr0005@iitism.ac.in

1-Indian Institute of Technology (Indian School of Mines), Dhanbad

Micro drillings are being used in various industries, such as aerospace, automobiles, and electronics. These industries also use carbon fiber-reinforced composites extensively. There is a growing need for micro-drilling in carbon fiber-reinforced composites, which is a challenging process. In this paper, we will analyze holes made by a 400-micron drill bit in carbon fiber-reinforced composite and metal-oxide nanostructured CFRP composites. Holes are made with three different spindle speeds and three different feed rates. The depths of holes are taken as 1 mm. Delamination damage, burr formation, fiber pull-out, and roundness error will be analyzed for different process parameters. The condition of the holes is to be compared for different process parameters as well as carbon fiber composites with and without nanostructures.

Keywords: Composites, Micro-drilling, Delamination damage, Nanostructures

APPROACH TOWARDS GREEN MANUFACTURING IN MAGLEV EDM USING DIFFERENT BIO-DIELECTRICS AT VARIABLE DISCHARGE CONDITIONS

Rajesh Sahoo¹, Vivek Bajpai¹, Nirmal Kumar Singh¹

e-mail: rajeshsahoo246@gmail.com

1-Department of Mechanical Engineering, Indian Institute of Technology (ISM) Dhanbad, Jharkhand, India-826004

Sustainable advanced manufacturing is a great alternative to the conventional approach to minimize (or) eliminate the hazardous effects of commonly used dielectrics in EDM. The thermo-electric nature of EDM process leads to the sparking phenomena which releases various toxic gases and non-biodegradable residuals as a result of hydrocarbon oil burning. To overcome such setbacks, several bio-dielectrics have been implemented as an economical replacement to the conventional (hydrocarbon oils such as kerosene, n-decane, etc.) dielectrics. In the current research, the machining characteristics of Ti-6Al-4V alloy have been investigated using neem oil and canola oil as bio-dielectrics in Maglev EDM. The newly developed Maglev EDM uses an electromagnet and two permanent magnets to provide better discharge gap control than conventional servo-gap control. The machining operation is conducted on a Ti-6Al-4V alloy workpiece (15mm × 15mm × 3mm) using a cylindrical mild steel tool ($\phi = 3.3$ mm). Performance measures such as workpiece erosion rate (WER), electrode erosion rate (EER) and roughness parameters were evaluated for both the bio-dielectrics. The results were compared for respective bio-dielectric at variable discharge voltages i.e. 22V, 25V and 28V, whereas the discharge current varied within (200-240 mA). The machining operations were performed at a duty cycle of 95% for every experimental repetition. Furthermore, the analysis of voltage-current characteristics waveforms depicts stable and consistent pulses with negligible irregularities. It also illustrates the absence of arcing and short-circuiting phenomena leading to better energy utilization as compared to conventional system. Additionally, the obtained results show a maximum MRR of 337 $\mu\text{g}/\text{min}$ in the case of canola oil and 306 $\mu\text{g}/\text{min}$ in the case of neem oil at 28V. Similarly, a maximum TWR of 79 $\mu\text{g}/\text{min}$ was achieved using canola oil and 63 $\mu\text{g}/\text{min}$ using neem oil at 28V. Canola oil is recommended over neem oil for better MRR and vice-versa for lower TWR. Lowest roughness (R_a) of 0.863 μm was achieved at 22V in neem oil as compared to 1.12 μm in canola oil. Moreover, the SEM micrographs help analyze the machined workpiece's surface characteristics by observing the surface anomalies such as micro-cracks, recast layers, blow holes, and embedded debris for respective bio-dielectrics. The elemental analysis of machined workpiece surface through EDX reports depicts the recast formation and material migration phenomena from the tool and dielectric for the respective bio-dielectric oils.

Keywords: MagLev EDM, Discharge, Roughness, Bio-dielectric, Titanium

GEOMETRICAL INSPECTION OF TOOL WEAR EFFECT IN MAGLEV MICRO-EDM PROCESS

Rajesh Sahoo¹, Sakshit Wankhede¹, Vivek Bajpai¹

e-mail: rajeshsahoo246@gmail.com

¹-Department of Mechanical Engineering, Indian Institute of Technology (ISM) Dhanbad, Jharkhand, India-826004

The current research inspects the geometrical deviation of tool interacting surface subsequent to the machining operation in MagLev EDM. The tool degradation phenomena in EDM, negatively impacts the dimensional accuracy of manufactured features which can be vividly explored through comparative analysis of tool tip geometry before and after the machining operation. The precise evaluation of tool erosion effect has been executed through extensive assessment of tool wear length at the bottom face and the edge i.e. both longitudinal and lateral direction to the tool tip. The investigation has been performed using a tungsten micro-rod ($\phi = 500 \mu\text{m}$) tool for machining Ti-64 alloy plate. The machining operation has been executed at a discharge voltage of 28V and current of 1.2A using deionized water as dielectric. The machining operations were performed at a duty cycle of 95% for every experimental repetition. The performance outcomes such as average electrode erosion rate (EER), lateral erosion length (LaEL) and longitudinal erosion length (LoEL) have been acquired at variable machining time. Moreover, the eroded tool surface has been inspected using a 3D surface profiler to acquire the roughness parameters at variable machining time. Furthermore, the analysis of voltage-current characteristics waveforms depicts stable and consistent pulses with negligible irregularities. It also illustrates the absence of arcing and short-circuiting phenomena leading to better energy utilization as compared to conventional system. Moreover, the SEM micrographs help analyze the tool surface characteristics by observing the crater orientation, distribution and surface anomalies such as micro-cracks, recast layers, blow holes, and embedded debris.

Keywords: MagLev EDM, Tool wear, Roughness, Surface profile, Titanium

AUTOR INDEX

A

Abdul Mu'iz Aniq Aiman Mohd Suhaimi	5
Abhilash Krishnamurthy.....	93
Abhipsa Kar.....	95
Adam Zaky.....	22, 23, 24
Adela Egelja.....	89
Aleksa Luković.....	73
Aleksandar Devečerski.....	92
Aleksandar Jovanović.....	67, 82, 83, 84
Aleksandar Jovičić.....	29
Aleksandar Marinković.....	82, 88, 89
Aleksandar Savić.....	28
Aleksandar Vasić.....	18, 46, 47
Aleksandar Vukadinović.....	92
Aleksandra Daković.....	37, 53, 85
Aleksandra Ivanović.....	68, 70
Aleksandra Patarić.....	26, 59, 60
Aleksandra Perić-Grujić.....	35
Aleksandra Popović.....	62
Aleksandra Šaponjić.....	63
Alen Delić.....	75
Alexander Birich.....	11
Ana Alil.....	27, 28, 72
Ana Valenta Šobot.....	63
Andrej Resnik.....	22
Andrija Savić.....	88
Andrijana Žekić.....	49
Andela Bojić.....	91
Angelina Mitrović.....	77
Annu Sharma.....	31
Anja Antanasković.....	65, 84
Arnab Das.....	94
Axel Schippers.....	12
Azila Adnan.....	33

B

Bernd Friedrich.....	11
Biljana Dojčinović.....	21, 76
Blaž Leskovar.....	22, 23, 24
Bojan Gligorijević.....	72
Bojan Medo.....	56
Bojan Miljević.....	31
Borislav Malinović.....	91
Boštjan Markoli.....	22, 23, 24
Branimir Grgur.....	10, 62
Branislav Ivošević.....	42
Branislav Marković.....	20, 51, 67, 87
Branka Jordović.....	59
Branko Matović.....	13, 48
Bratislav Antić.....	21, 76

C

Chen Soo Kien.....	5
--------------------	---

Corby Anderson.....	3
---------------------	---

D

Dajana Milkić.....	54
Dalibor Stanković.....	21, 76
Danijela Smiljanić.....	37, 53, 77, 85
Danina Krajišnik.....	53, 85
Darija Đorđević.....	46
Davor Stanić.....	25
Dejan Todorović.....	42
Dimitrije Anđić.....	51
Dimitrije Petrović.....	92
Dragan Manasijević.....	54, 58
Dragan Radulović.....	42
Dragana Ivsic.....	69
Dragana Mihajlović.....	56
Dragana Randelović.....	20
Dragana Živojinović.....	28
Dragomir Glišić.....	28, 80
Draženko Bjelić.....	91
Dušan Božić.....	30, 49, 50
Dušan Bučevac.....	48
Dušan Milojkov.....	26, 77, 83
Duško Minić.....	58
Dženana Smajić-Terzić.....	34
Đorđe Janacković.....	61, 78
Đorđe Veljović.....	56, 61, 90

E

Endre Romhanji.....	72
Ernesto Barceló.....	78

F

Fernando Carrasco López.....	55
Franjo Kozina.....	25, 57

G

Goran Jevtić.....	44
Gordana Bakić.....	48, 73
Gordana Marković.....	26, 60
Grozdanika Bogdanović.....	19
Gvozden Jovanović.....	20, 51, 75, 87

H

Horst Hahn.....	6
-----------------	---

5th Metallurgical & Materials Engineering Congress of South-East Europe 2023 – Trebinje, BIH

I	
Igor Radisavljević.....	55
Ilhan Bušatlić.....	34
Ivan Jovanović.....	63
Ivan Stojković.....	45, 79
Ivana Banković-Ilić.....	45, 79
Ivana Cvijović-Alagić.....	48
Ivana Marković.....	54
Ivana Marković ¹	58
Ivana Mikavica.....	67
Ivona Radović.....	74
Iztok Naglič.....	22, 23, 24
<hr/>	
J	
Jakob König.....	36
Jakov Dimitrijević.....	61
Janko Živanić.....	84
Jelena Avdalović.....	65
Jelena Dikić.....	90
Jelena Dimitrijević.....	83, 86
Jelena Erčić.....	50
Jelena Filipović Tričković.....	63
Jelena Nikolić.....	64
Jelena Petrović.....	77, 83, 86
Jelena Stašić.....	30, 49, 50
Jelena Vujančević.....	32
Jovana Bošnjaković.....	88, 89
Jovana Đokić.....	90
Jovana Milanović.....	89
Jovana Ružić.....	26, 30, 49, 50
Jovica Stojanović.....	23
Julia Glaum.....	4
Julijana Tadić.....	61
<hr/>	
K	
Katarina Dimić-Mišić.....	78
Katarina Sokić.....	90
Kemal Delijić.....	71
Kristina Božinović.....	54, 58
Kristina Žagar Soderžnik.....	32, 93
<hr/>	
L	
Lee Oon Jew.....	33, 52
Lidija Radovanović.....	41
Ljiljana Janković-Mandić.....	89
Ljiljana Matović.....	92
Ljubica Radović.....	55
Ljubiša Balanović.....	54, 58
<hr/>	
M	
Maja Đolić.....	35
Maja Kokunešoski.....	63
Maja Kozarski.....	53
Malia Athirah Badruddin.....	5
Marija Došić.....	64
Marija Egerić.....	92
Marija Ercegović.....	83, 86
Marija Koprivica.....	83, 86
Marija Korać.....	68, 69, 71
Marija Marković.....	37, 85, 86
Marija Mihailović.....	59, 60
Marija Mirković.....	21
Marija Prekajski Đorđević.....	48
Marija Simić.....	77, 83, 86
Marija Stevanović.....	82
Marija Štulović.....	43
Marija Vuksanović.....	89
Marko Rakin.....	56
Marko Simić.....	30, 49, 50
Martin Folta.....	25
Matjaž Spraitzer.....	36
Mihajlo Arandelović.....	80
Mijajlo Jauković.....	44
Milan Gorgievski.....	54, 58
Milan Milivojević.....	84
Milan Trtica.....	49
Milana Zarić.....	74
Mile Đurđević.....	17
Milena Milošević.....	88
Milena Obradović.....	37, 64, 85
Milena Pantić.....	85
Milena Premović Zečević.....	58
Milica Ožegović.....	37, 85
Milica Stojković.....	35
Milica Vidak Vasić.....	41
Milica Vlahović.....	27
Milislav Ranitović.....	43, 68, 69
Milorad Gavrilovski.....	75
Miloš Marković.....	46
Miloš Ognjanović.....	21, 76
Milovan Stoilković.....	73
Miljana Popović.....	51, 72
Miljana Radivojević.....	9
Mirjana Čujić.....	35
Mirjana Kijevčanin.....	74
Mirjana Ristić.....	35
Miroslav Sokić.....	20, 26, 75, 77, 82, 84, 87
Mitar Mišović.....	81
Mladen Bugarčić.....	42, 65, 82, 84
Mohd Sabri Mohd Ghazali.....	5, 33, 52
Monireh Imani.....	78
Muhamad Syaizwadi Shaifudin.....	5, 33, 52
<hr/>	
N	
Nadira Bušatlić.....	34
Natalija Dolić.....	57
Nataša Đorđević.....	60
Nataša Gajić.....	43, 51
Nataša Knežević.....	88, 89
Nebojša Tadić.....	81
Nedeljko Dučić.....	29
Nela Vujović.....	87
Nemanja Barać.....	78
Nenad Milosavljević.....	44
Nenad Radović.....	55
Neža Sodnik.....	32
Nikola Jovanović.....	69

Book of Abstracts

Nikola Kanas	31
Nikolay Stoimenov	30
Nirmal Kumar Singh	96
Nur Aiman Syafiq Mohd Hamidi	33
Nursabrina Amirah Mohd Nasir	5, 33
Nusaibah Yusof	5, 33

P

Patrick Gane	78
Petar Uskoković	78
Prince Anand	94

R

Radojka Vujasin	92
Radomir Jovičić	80
Radomir Radiša	29
Rajesh Sahoo	96, 97
Ratka Petrović	81
Ravi Shankar Rai	95
Renata Kovačević	70
Reshma Madathil	31

S

Sakshit Wankhede	97
Sanja Jevtić	43, 90
Sanja Martinović	27, 28
Sanja Petrović	19
Sanja Vranješ-Đurić	21
Silvana Dimitrijević	68, 70, 71
Simon Sedmak	80
Slavica Mihajlović	60
Snežana Aksentijević	59
Snežana Grujić	64
Snežana Uskoković-Marković	53
Sofija Dedić	35
Sonja Miličević	42
Sonja Smiljanić	36
Srđan Matijašević	64
Srđan Rakić	31
Srđan Stanković	12
Srećko Manasijević	17, 29, 88
Stanislav Goshev	30
Stefan Dikić	80
Stefan Kolašinac	65
Stefanie Hetz	12
Stevan Armaković	31
Stevan Dimitrijević	68, 70, 71
Subramshu Bhattacharya	6, 31
Špela Trafela	32, 93

T

Tamara Matić	61
Tamara Ristić	44

Tatjana Šoštarić	65
Tatjana Volkov-Husović	27, 28
Tijana Đuričić	91
Tomislav Bradarić	47

U

Uroš Hribar	36
Uroš Stamenković	54, 58
Uroš Stojaković	67

V

Vaso Manojlović	26, 43, 46, 51, 75
Veljko Đokić	56
Veljko Savić	64
Vesna Alivojvodić	87
Vesna Lazarević	79
Vesna Lazić	53
Vesna Maksimović	48, 73
Vivek Bajpai	94, 95, 96, 97
Vlada Veljković	45, 79
Vladimir Adamović	65
Vladimir Jovanović	42
Vladimir Malbašić	18
Vladimir Pavičević	35, 45, 67
Vladimir Pavkov	48, 73
Vladimir Srdić	31
Vladimir Topalović	64
Vukašin Ugrinović	61
Vukosava Živković-Radovanović	77

W

Wan Mohamad Ikhmal Wan Mohamad Kamaruzzaman	5, 33, 52
Wan Mohd Norsani Wan Nik	33
Wan Rafizah Wan Abdullah	52

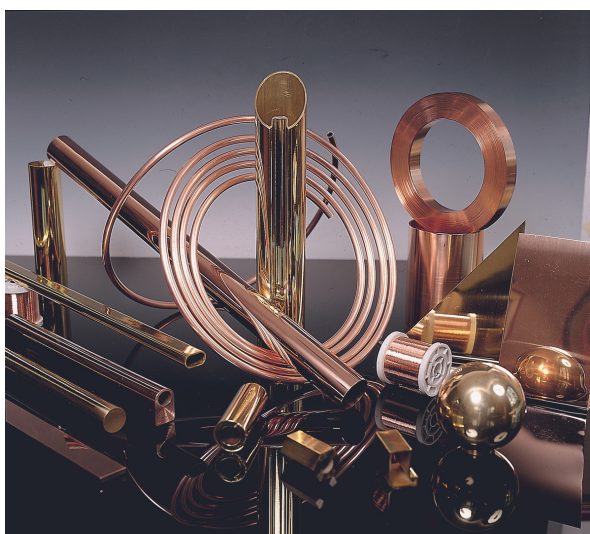
Y

Yi-nan Wu	92
-----------------	----

Z

Zagorka Radojević	41
Zdenka Zovko Brodarac	25, 57
Zoran Anđić	90
Zoran Samardžija	32
Zorica Lopičić	65, 84
Zvezdana Bašćarević	63
Žan Kresnik	22
Žarko Radović	81
Željko Kamberović	23, 43, 46, 51, 69, 71, 87
Željko Radovanović	50

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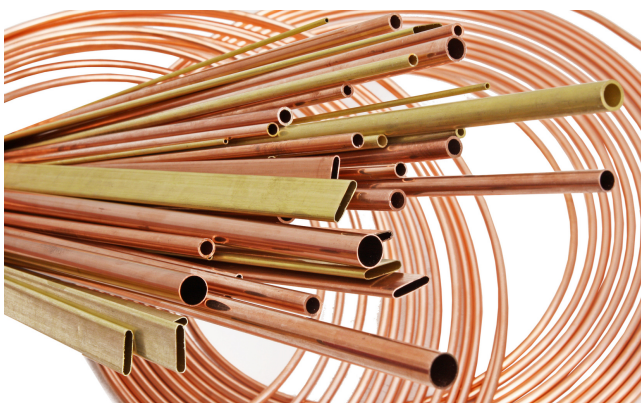


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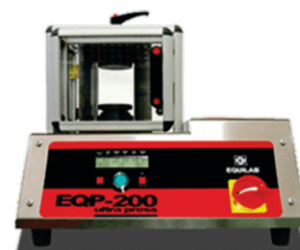
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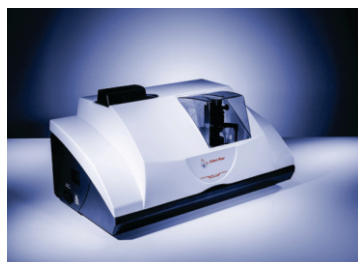
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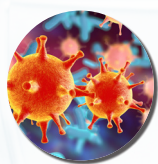


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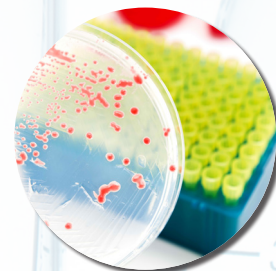
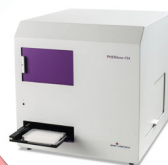
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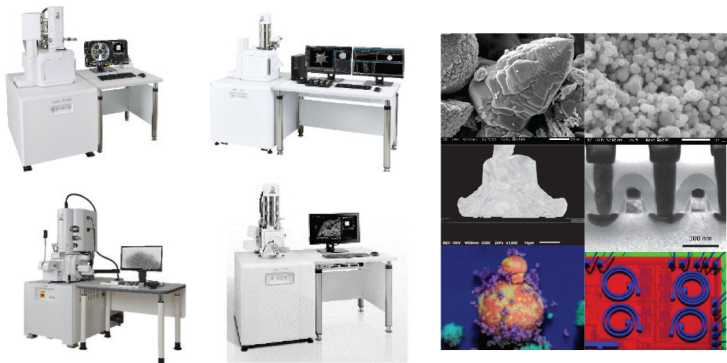


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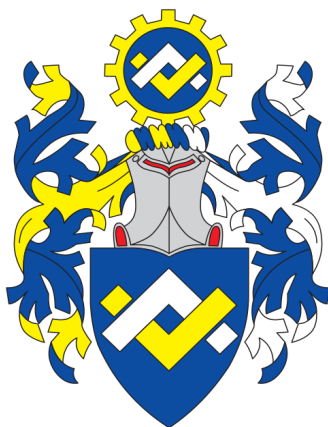


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