

Association of Metallurgical Engineers of Serbia  
Faculty of Technology and Metallurgy, University of Belgrade  
Institute for Technology of Nuclear and Other Mineral Raw Materials  
Institute of Chemistry, Technology and Metallurgy  
Vinca Institute of Nuclear Sciences  
Serbian Foundrymen's Society

**MME SEE**

**2019**

Metallurgical & Materials  
Engineering Congress  
of South-East Europe

**BOOK OF ABSTRACTS**

June, 5<sup>th</sup> - 7<sup>th</sup> 2019, Belgrade, Serbia  
[www.mme-see.org](http://www.mme-see.org)

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Dragomir Glišić  
Branislav Marković  
Vaso Manojlović

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**Technical editor:**

Department of Printing Engineering

Faculty of Technology and Metallurgy, University of Belgrade

**Published by:**

Association of Metallurgical Engineers of Serbia (AMES)

**Circulation:**

120 copies

**Printed by:**

Department of Printing Engineering, Faculty of Technology and Metallurgy

Karnegijeva 4, POB 35-03

11 120 Belgrade, Serbia

Tel: +381 11 3370 492

ISBN 978-86-87183-30-8

Supported by:  
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## **PREFACE**

The Fourth Metallurgical & Materials Engineering Congress of South-East Europe (MME SEE 2019) 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 the knowledge in the field of metallurgical and materials engineering.

The Congress is organized jointly by the Association of Metallurgical Engineers of Serbia, Faculty of Technology and Metallurgy, University of Belgrade, Institute for Technology of Nuclear and Other Mineral Raw Materials, Institute of Chemistry, Technology and Metallurgy, Vinca Institute of Nuclear Sciences and Serbian Foundrymen's Society.

The Editors would like to thank the Scientific and the Organizing Committee, the Congress Secretariat - CONGREXPO d.o.o. and all those who helped in making the Congress a success.

Special thanks are due to the Ministry of Education, Science and Technological Development of the Republic of Serbia and sponsors for the financial support of the Congress.

*Editors*





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# **Plenary Lectures**



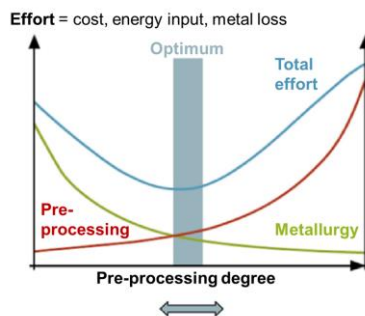
## INNOVATIVE RECYCLING OF POLYMETALLIC EOL-PRODUCTS - CHALLENGES AT THE INTERFACE OF THE PROCESS CHAIN

Dr.-Ing. Elinor Rombach<sup>1</sup>, Prof. Dr.-Ing. Dr. h.c. Bernd Friedrich<sup>1</sup>

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Due to the increasing complexity of modern secondary raw materials and the limited cycle guidance in the metallurgical aggregates used, innovative metal recycling can only be achieved by multi-metal approaches. The current challenges for a meaningful design of suitable process routes are manifold and interdisciplinary. Against the background of a long-term securing of the raw material basis for high-tech products, current research work focuses not only on bulk metals such as aluminum, copper, lead and zinc, but also on economically strategic metals such as cobalt, tantalum, tungsten, etc.



Due to the close interlinking, efficiency potentials can be found, especially at the interface between pre-processing and metallurgy. For example, a mechanical treatment process that is optimized from a scientific-technical point of view and is not adapted to the requirements of the subsequent metallurgy can drastically reduce its efficiency. The aim here is to determine the techno-economically sensible processing depth and thus the optimum for both consecutive process steps.

The starting point for the contribution presented here to the optimization of the recycling route for complex waste streams is the idea of a resource-efficient circular economy. The chances and the difficulties of an intensified cooperation at the interface pre-processing - metallurgy are worked out and qualitatively clarified by the example of Li-Ion battery scrap. It will be shown that interface optimization can only be achieved if more communication takes place between the two fields of technology. Here the more flexible system must always adapt to the upstream/downstream unit.

**Keywords:** circular economy, recycling, pre-processing, metallurgy, interface optimization.

## **APPLICATION AND PRACTICE OF MULTI-POLLUTANT COOPERATIVE CONTROL TECHNOLOGY FOR FLUE GAS IN IRON AND STEEL INDUSTRY**

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Coking and sintering flue gas pollutant discharge status and the current iron and steel industry of environmental protection policies are analyzed in this paper. The gas emissions of pollutants characteristics to a large iron and steel enterprises of sintering and coking are analyzed. In view of the typical pollutants sulfur, nitrate, the existing of dust removal technology, analyzes its advantages and disadvantages and put forward the technical routes which can realize ultra-low emission. Finally, the application practice of these techniques in a large iron and steel enterprise is emphatically introduced.

**Keywords:** iron and steel industry, multi-pollutant control, flue gas.

## **PRESENT DEVELOPMENT AND TENDENCY ABOUT THE TREATMENT OF SECONDARY RESOURCE IN CHINA**

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The investigations of present development and tendency about the treatment of secondary resource in China includes four part. The first part is about metallurgy technology status quo of nonferrous metal. Metallurgy technology of several kinds of nonferrous metals will be introduced. The second part is about smelting solid waste disposal technology. The recovery technology of solid waste from copper smelting plant (including arsenic pressure filtered cake, black copper slime, lead pressure filtered cake, soot, etc.), coal ash with high aluminum fly ash and cyanide tailings will be presented. The third part is about secondary resource recycling and regeneration technology. The recovery and treatment process of exhaust catalyst and waste lithium-ion power batteries will be introduced. The final part is about the development tendency of collaborative smelting technology. Several examples of collaborative smelting technology will be shared, including “collaborative smelting technology of waste circuit board and low-grade waste copper”, “collaborative smelting technology of waste circuit board and copper concentrate” and “collaborative smelting technology of waste circuit board, electroplating sludge, waste catalyst and other materials containing copper” etc.

**Keywords:** secondary resources, China, solid wastes, recycling, smelting.

## **CORSON ALLOYS: EFFECT OF MICROSTRUCTURAL FEATURES ON THE PROPERTIES**

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Precipitation hardened copper alloys are heat treatable alloys and the uniform distribution of fine precipitates in the copper matrix increases the strength. In several applications, these alloys are preferably used due to their high strength, excellent conductivity and low cost. To design a matrix having high strength and high electrical conductivity, both alloying and thermomechanical routes directly affect the final properties.

Nowadays, Corson (CuNiSi) alloys have created considerable interest due to their beneficial combination of high strength and high electrical conductivity. The strengthening in these alloys is achieved due to the precipitation of finely distributed second phase particles known as metal-silicides. Several investigations on Corson alloys reveal that the simultaneous optimization of electrical conductivity and hardness can be obtained as a function of alloying, thermomechanical routes and powder metallurgical (PM) techniques.

In this study, several Corson alloy compositions are designed with the addition of Al, Mg, Cr, Co, and Zr, and the alloys are produced by several manufacturing techniques (casting, forging, powder metallurgy). The effects of both precipitation kinetics and microstructural features on their properties are discussed in detail.

**Keywords:** Corson alloy, alloying, heat treatment, kinetics, microstructure.

# **Invited lectures**





## PROPERTIES OF CAST ALUMINUM ALLOYS SUITABLE FOR PRODUCTION OF E-MOBILITY COMPONENTS

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The global automotive industry is nowadays moving towards electrifications. Economy, air pollution and global warming are factors that considerably changed automotive strategy in recent years. This paper analyses the currently used cast alloys and summarizes the alloy requirements to fulfill the specifications for e-mobility automotive-mobility parts. Five cast alloys have been recognized to have the potential to be applied for serial production in casting processes such as: High-Pressure Die Casting, Low-Pressure Die Casting and Core Package Sand Casting. Specific requirements such as dimensional stability, corrosion resistance, electromagnetic compatibility and crashworthiness are discussed in this paper.

**Keywords:** e-mobility parts, castings, properties.

## ELECTRODEPOSITION OF LANTHANUM IN ROOM TEMPERATURE IONIC LIQUID ELECTROLYTE<sup>1</sup>

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Electrochemical techniques are of vital importance for development of pyrochemical fuel treatment processes for separation of actinides from the fission products (lanthanides) contained in spent nuclear fuel. Using pyroprocessing technology, actinides can be recovered from the spent fuel and prepared for recycle, as fuel, while the fission products can be encapsulated in durable, leach resistant waste forms destined for storage. Extensive research has and continues to be performed in this area under the US Department of Energy's Fuel Cycle Research and Development program with the ultimate goal of developing efficient, sustainable and environmentally responsible nuclear energy systems. Although termed as pyroprocessing technology, the underlying electrochemical separation of actinides and lanthanides is in essence electrometallurgy, where eutectic inorganic molten salts are typically utilized as the electrolyte. The inorganic molten salts should be viewed as the high temperature ionic liquids.

For processing at low temperatures, separation of electronegative metals by electrodeposition, such as those from lanthanide series of elements, requires the use of hydrogen ion free (non-aqueous) electrolyte solutions. This precondition is met by using the so-called room temperature ionic liquids, a relatively new class of electrolytes. In this research, the electrodeposition of lanthanum was studied in <sup>1</sup>ethyl-3-methylimidazolium dicyanamide (EMIM-DCA) and 1-butyl-3-methylimidazolium dicyanamide (BMIM-DCA). The electrochemistry was studied by using the rotating disk technique, in which the working electrode was Pt, and respective counter and reference electrodes were graphite and silver/silver chloride. Cyclic voltammetry and chronoamperometry were the primary electrochemical techniques used in these studies. The parameters studied were the effect of scanning potential range, scanning rate, cathodic and anodic vertex potentials. The role of absorbed water, and the presence of alcohols (ethanol), was also examined. The rotation speed parameter was of importance for calculation of reaction rate and mass transfer rate constants. The electrode surface characterization was performed by using optical and scanning electron microscopy.

**Keywords:** Lanthanum, room temperature ionic liquids, electrodeposition, rotating disk

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<sup>1</sup> Acknowledgment

This work was supported by the US Department of Energy- Nuclear Engineering University Program (NEUP), Contract No. DE-NE00008556.

## HYDROMETALLURGICAL TREATMENT OF PRIMARY AND SECONDARY MATERIALS IN THE PRODUCTION OF THE CRITICAL METAL OXIDES

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The new list of critical raw materials from 2017 features 27 raw materials: Antimony, Beryllium, Borates, Cobalt, \*Coking Coal, Fluorspar, Gallium, Germanium, Indium, Magnesium, Natural Graphite, Niobium, Phosphate Rock, Silicon Metal, Tungsten, Platinum Group Metals, Light Rare Earths and Heavy Rare Earths, Baryte, Bismuth, Hafnium, Helium, Natural Rubber, Phosphorus, Scandium, Tantalum, and Vanadium. It is hoped that the list will help incentivize the European production of critical raw materials through enhancing recycling activities and when necessary to facilitate the launching of new mining activities. It is very difficult to replace these critical metals by other metals. Because of the high application, the demands of these metals are increased, but the production cannot follow its increased consumption in electronics, catalysis, and medicine. The selective production of rare earth element oxides is the most important aim in the processing of raw materials. The hydrometallurgical treatment (dissolution of ores, concentrates and waste materials under atmospheric and high pressure, purification of solution through neutralization, filtration, precipitation) and thermal decomposition of the oxides were mostly applied for the selective metal oxide production from ores and secondary materials. The hydrometallurgical treatment makes possible to limit the environmental impacts like residual waste producing, energetic expenditure and reagent consumption. Hydrometallurgy offers an alternative to pyrometallurgical treatment, but the combination of pyrometallurgical and hydrometallurgical treatment offers the best research strategy for the formation of critical metal oxides. In this study, the role of a hydrometallurgical treatment in the production of the critical metal oxides shall be presented mostly for the recovery of rare earth elements, and partially for cobalt.

**Keywords:** hydrometallurgy, rare earth elements, cobalt, oxides.

## CATIONIC SURFACTANTS MODIFIED KAOLIN – EFFICIENT ADSORBENTS FOR MYCOTOXINS

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The natural kaolin from a plant for the production of quartz sand in Rgotina, Serbia (KR) was modified with two cationic surfactants - octadecyl dimethyl benzyl ammonium chloride (O) and hexadecyltrimethylammonium bromide (H). Both surfactants were added in amounts equal to 90% of kaolin cation exchange capacity (CEC). The obtained materials were denoted as OKR and HKR. Characterizations of KR, OKR, and HKR were done by Fourier transform infrared spectroscopy (FTIR) spectroscopy and zeta potential measurements. FTIR spectra and zeta potential measurements confirmed the presence of both surfactants at the kaolin surface.

Adsorption of mycotoxins – zearalenone (ZEN) and ochratoxin A (OCHRA) was studied by the natural kaolin and organokaolines under *in vitro* conditions. The effects of the amount of the adsorbent, the initial ZEN and OCHRA concentrations, and pH, on adsorption of mycotoxins, were investigated. Preliminary results of adsorption of ZEN and OCHRA by KR showed that natural kaolin had no affinity to adsorb these mycotoxins. However, the presence of both organic cations in the kaolin structure significantly increased adsorption of ZEN and OCHRA. Adsorption of the mycotoxins by OKR and HKR increased with increasing the amount of each adsorbent in suspension. Slightly higher adsorption of ZEN than OCHRA by both adsorbents was observed at both pHs.

ZEN and OCHRA adsorption increased for both OKR and HKR as the concentration of the initial toxins increased at pH 3 and 7. OCHRA and ZEN are hydrophobic molecules that possess different functional groups and may exist in various forms at different pH values. OCHRA adsorption by OKR and HKR followed nonlinear isotherms at pH 3 and 7, and higher adsorption capacity was obtained for OKR. Also, OCHRA adsorption capacity for OKR and HKR was much higher at pH 3. The obtained results suggest that adsorption of OCHRA was dependent on the form of OCHRA in solution and that type of surfactant had an influence on OCHRA adsorption. Adsorption of ZEN by organokaolines also showed nonlinear isotherms at pH 3 and 7 and similar amounts were adsorbed at both adsorbents at both pH values. The obtained adsorption capacities suggest that adsorption of ZEN was practically independent of the form of the toxin in solution and also on the type of the organic cations in the kaolin structure. Based on the obtained results, kaolin modified with surfactants may be effective materials for adsorption of ZEN and OCHRA.

**Keywords:** kaolin, surfactants, mycotoxins, adsorption.

# **Oral presentations**



## INITIAL STEPS ON THE ROAD TO THE DIGITALISATION OF THE IMPOL ALUMINIUM INDUSTRY

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The following examples of digitalisation in the Impol Aluminium Industry are presented: (i) the use of artificial intelligence in the modelling of production processes, (ii) the use of self-learning algorithms in the development of improved properties for end-products, and (iii) the use of predictive artificial intelligence in quality control and certification.

The first example is the modeling of the processing path for the production of extruded semis with the required mechanical properties. For this purpose a powerful and cost-effective algorithm based on a neural network was developed and learned to (i) **extract structured data** (process parameters, concentrations of alloying elements, and mechanical properties), (ii) **find the correlations** between the individual processing paths and the end-product properties, and (iii) **make predictions** about the compositions of new alloys and the processing parameters for matching the required mechanical properties.

The improved end-product performance achieved with the use of the self-learning algorithm is illustrated by the development of more intergranular corrosion-resistant wrought aluminum alloys. The purpose of the self-learning algorithm, developed for that particular case, was to compare and correlate the results of the corrosion resistance measured by two different methods. In the first set of experiments, the corrosion resistance was determined by a metallographic measurement of the intergranular corrosion. In the second, the numerical data of the corrosion resistance were exported directly from an electrochemical cell. The entire process resulted in the accumulation of the appropriately filtered and structured data for high-quality data-driven predictions of the most stress-corrosion-resistant compositions of wrought aluminum alloys.

The use of predictive artificial intelligence in quality control and certification was highlighted by a new method for the data-driven inspection of inclusions in wrought aluminum alloys. A promising approach for the rapid inspection of inclusions in wrought aluminum alloys is optical emission spectroscopy (OES). However, to separate the peaks corresponding to particular inclusions from the peaks obtained from various microstructural features in the matrix, advanced filtering of the OES spectrum is necessary. The developed methodology is based on big-data-driven predictions of whether the online analyzed sample is good or bad. By following a machine-learning process, an algorithm was developed that enables the online division of the samples into good and bad, based on criteria received from the casting house.

**Keywords:** digitalization, aluminum industry, modeling, predictions, correlations.



## **X-RAY DIFFRACTION ANALYSIS ON MECHANICALLY ALLOYED ALUMINUM COMPOSITE POWDERS CONSISTING OF NANO ALUMINA PARTICLES AND MULTIWALL CARBON NANOTUBES**

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In this work, the aluminum alloy (Al-4Cu) based composite powders bearing nano alumina particles and multiwall carbon nanotubes were produced via mechanical alloying in argon gas environment for different periods up to 20 h. The process was carried out in the attritor mill using a stainless-steel chamber with 10 mm diameter stainless steel balls. The ratio of the balls to the powder weight was selected to be 10:1 while the rotational speed was kept as 250 rpm. The composite powders in the slurry form, which were spread within ethanol completely, were processed during the mechanical alloying to get more uniform powder distribution and prohibit powder agglomeration. After milling the composite powders for various periods up to 20 h, the X-ray diffraction (XRD) analysis of the nanocomposite powders was performed after the operation of every 5 h. The crystallite sizes of the powders and micro-strains induced during the operation were determined by various available methods with the aid of XRD patterns. The results brought out that, a significant reduction in the crystallite size of powders was observed only after a milling time of 5 hours and a further increase in the milling time led to a little change.

**Keywords:** mechanical alloying, multiwall carbon nanotube, composite powder, XRD analysis.

## INVESTIGATION OF EQUILIBRIUM AND NON-EQUILIBRIUM SOLIDIFICATION OF Al-2.2Mg-2.1Li ALLOY

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The investigation of equilibrium and non-equilibrium solidification of aluminum (Al) alloy containing 2.2 wt. % magnesium (Mg) and 2.1 wt.% lithium (Li) was conducted in order to estimate the influence of chemical composition and thermodynamic parameters on microstructure development.

Predictions of solidification sequence under equilibrium and non-equilibrium conditions were obtained using Computer Aided Thermodynamic Diagram Calculation (CALPHAD). Equilibrium predictions comprehended transformations and precipitations in liquid and solid state. Prediction in non-equilibrium conditions, based on Scheil-Gulliver solidification, covered only diffusion based processes in liquid state. Solid state processes were not predicted due to the low diffusion rate. Developed microstructure of Al-2.2Mg-2.1Li alloy was analyzed using metallographic and thermal analysis, respectively.

Equilibrium solidification begins with transformation of  $\alpha_{Al}$  dendritic network followed by diffusion based solid state precipitations. Stable AlLi ( $\delta$ ) phase precipitates first as a result of reduced solubility of Li in  $\alpha_{Al}$  and high Li/Mg ratio. Ternary Al<sub>2</sub>LiMg (T) phase precipitates from Mg rich banded  $\alpha_{Al}$ . Equilibrium solidification ends with precipitation of Al<sub>3</sub>Mg<sub>5</sub> ( $\beta$ ) phase.

Prediction under non-equilibrium conditions covered precipitation of  $\alpha_{Al}$ , stable  $\delta$  and ternary T phase directly from the Liquid. Precipitation of  $\beta$  phase was not predicted.

However, variations in the solidification sequence were identified by microstructural analysis, respectively. According to microstructural analysis, solidification of Al-2.2Mg-2.1Li alloy begins with transformation of  $\alpha_{Al}$  dendritic network followed by precipitation of metastable Al<sub>3</sub>Li ( $\delta'$ ) phase inside the grains of  $\alpha_{Al}$ . Metastable precipitation is caused by reduced solubility of Li in  $\alpha_{Al}$ . Reduced solubility of Li in  $\alpha_{Al}$  is additionally affected by the Mg. During rest of solidification, the  $\delta'$  phase is used as a nuclei for precipitation of both  $\delta$  and T phase. Nucleation and growth of  $\delta$  and T phase causes formation of precipitation free zones (PFZ) near the grain boundaries. Solidification ends with precipitation of irregular and coarse  $\beta$  phase at the grain boundaries.

The Al-2.2Mg-2.1Li alloy investigation indicated intensive influence of chemical composition and thermodynamic parameters correlation on the solidification sequence. This correlation caused variations between equilibrium and non-equilibrium solidification.

**Keywords:** Al-2.2Mg-2.1Li alloy, equilibrium and non-equilibrium solidification, microstructure.

## ON THE COMPRESSIBILITY BEHAVIOR OF ALUMINUM COMPOSITE POWDERS BEARING VARIOUS FRACTIONS OF TITANIUM DIBORIDE PARTICULATES<sup>2</sup>

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At the beginning of this study, the powder characterizations for aluminum and titanium diboride particles were made to determine their powder size and shape. And then, pure Al matrix composite powders containing different proportions of titanium diboride (TiB<sub>2</sub>) ceramic particles were prepared by mixing them for 2 h in a 3D mixer. After the mixing operation, they were cold-pressed at various pressures up to 800 MPa to see the effect of compaction pressure and reinforcement content on the green and relative densities of the investigated composite powders. Next, the variations in the relative densities for the composite powders concerning compaction pressure were obtained. Finally, the data gained experimentally in this research was processed with different powder compressibility models to clarify the densification behavior of powders. The experimental results showed an excellent agreement with most of the models used in this work.

**Keywords:** aluminum composite, compressibility, titanium diboride.

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### <sup>2</sup> Acknowledgment

This study was supported by Osmaniye Korkut Ata University, Scientific Research Projects Unit (Project No: OKÜBAP-2018-PT3-011). The authors would like to thank Osmaniye Korkut Ata University for its financial support.

## VALORIZATION OF LEACHING RESIDUES OF LATERITES FOR THE PRODUCTION OF INORGANIC POLYMERS

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In this study, the residues obtained after heap leaching of Greek low-grade laterites using 147 g/L H<sub>2</sub>SO<sub>4</sub> as leaching solution for the production of IPs is investigated. The effect of the molarity of alkaline solution (6-10 M), curing temperature (60 and 80 °C), aging period (7 and 28 days) and the addition of Na<sub>2</sub>SiO<sub>3</sub> on the compressive strength of the final IPs was studied. The results indicate that the produced specimens cannot be alkali activated as their compressive strength reached the maximum value of 1.3 MPa. To improve their alkali activation potential, the residues were calcined at 800 and 1000 °C and the addition of metakaolin, produced from calcination of kaolin, as alkali activated precursor was explored. The results show that the addition of 10% metakaolin increases the compressive strength of the specimens to 39 MPa. The structural integrity of the produced IPs when immersed in distilled water or exposed to high- temperatures (200-1000 °C) was determined. The characterization of the raw materials and the produced IPs was performed by using X-ray fluorescence analysis (XRF), X-ray diffraction (XRD), Fourier-transform infrared (FTIR) spectroscopy and differential scanning calorimetry (DSC/TG).

**Keywords:** inorganic polymers, leaching laterites, compressive strength.

## INTEGRATED RECYCLING OF THE CRITICAL RAW MATERIALS FROM WASTE ELECTRONICS

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Materials scarcity and supply risks have become significant issues. Accordingly, high efficient material valorization from secondary sources has emerged as a response to the resource pressures and demand for more sustainable production models.

Among others, waste electrical and electronic equipment (WEEE) represent a secondary material which contains a significant amount of critical materials. However, considering the established industrial practice which is mostly focused on the recovery of Cu and Au, this waste is not processed correctly, which eventually hinders its true potentials. A range of techniques and operations are currently applied for materials recovery from WEEE. The essential features of these systems generally follow the scheme of independent pre-processing (disassembly, size reduction, separation) and end-processing (reductive smelting, pyro-hydro-electro refining) operations. However, in this way, both functional and sustainable recovery of materials is not implemented correctly, which leads to lower recoveries and losses of the metal values. Additionally, due to the high material diversity, different elements, with different metallurgical properties and in different concentrations are introduced, why process control is much more difficult. Therefore, the development of the highly efficient process for selective metals recovery is crucial for overall improvements.

This paper presents experimental results for selective metals recovery from WEEE combining the different pyro-hydrometallurgical operations. Experimental results showed that due to the complex metallurgical reactions, pure pyrometallurgical treatment leads to the distribution of metals in melting products, deviating from the theoretical and expected. However, these results led to a more comprehensive understanding of the reaction systems and contributed to an optimized design of processing operations. This will allow recycling companies to become competitive in the regional market and beyond, which is particularly essential for small and medium enterprises with lower operating capacities.

Successful implementation of the developed state-of-art technological process, guarantee a more efficient approach to recycling processes, production of new materials which supports the concepts of sustainable development and circular economy.

**Keywords:** recycling, critical materials, WEEE, circular economy.

## THE INFLUENCE OF THE ADDITION OF DIFFERENT TYPES OF LIME ON THE SINTERING PROCESS

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Sintering process represents a necessary step before hot metal production in a blast furnace, mixing mainly iron ore with fluxes and coke. Optimal composition and metallurgical properties of the sinter must be achieved to enable the production of high-quality hot metal and finally steel. This research was initiated to analyze the impact of lime addition into sinter mix and partially replacing a portion of fluxes (limestone) by lime on the productivity of the sintering process and potential fuel savings. The main objective of the research was to assess the impact of different types of lime on qualitative and quantitative indicators of the sinter. Experiments were carried out in a sintering pan. Addition of different types of lime and coke were added in the pre-pelletizing phase. Lime as an intensifier of the sintering process leads to improved agglomeration and permeability of the sinter mix. Lime addition into sinter mix indicates a positive effect on the heat content in the sinter bed leading to shortening sintering time and possible fuel savings. Based on experimental results, partial usage of lime in the sinter mix brings a positive impact on productivity and positive environmental influence.

**Keywords:** sinter, sintering, lime, productivity, fuel savings.

## SCIENTIFIC COOPERATION THROUGH JOINT LABORATORY AND THE PRESENT DEVELOPMENT AND TENDENCY ABOUT NICKEL LATERITE METALLURGY PROCESS

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Development and tendency on nickel laterite metallurgy process are investigated. Several processes for treating laterite-nickel ore will be introduced, including the RKEF smelting process, BF smelting process and HPAL process. The flowsheet and characteristics of each process are presented, and the main technical and economic indicators are compared. A method called "reverse leaching" that can deal with both limonite laterite and saprolite laterite developed by BGRIMM is proposed. The process splits the conventional high acid pressure leaching process (HAPL) into two stages to realize leaching and iron removal. Reverse leaching process can treat two typical kinds of laterite in one process, solve the problem that only one type of laterite can be treated in the existed process. It is especially suitable for processing transitional laterite nickel ore that that stockpile in the mines and ports. It's thought to be of great potential.

Scientific Cooperation Between BGRIMM and the University of Belgrade is achieved through Joint laboratory within Belt and Road Partnership Program studying Jarosite Treatment Process Development.

**Keywords:** joint laboratory; Nickel Laterite; reverse leaching; HAPL.

## **CERTIFICATION PROCESS FOR THE MANUFACTURE OF METALLIC PARTS AND COMPONENTS USING ADDITIVE MANUFACTURING 3D PRINT TECHNOLOGY**

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3D printing has the potential to offer substantial value through its ability to offer personalized products to consumers and to make several parts of today's manufacturing processes more efficient.

Recognizing that Additive Manufacturing (AM) is a unique and evolving manufacturing process with limited standards and best practice, LR has been actively involved in research to provide guidance to the industry in the adoption of AM in place of traditional manufacturing processes.

Manufacturers who are considering using 3D print technology need to understand the steps and the requirements involved. This paper will outline what additive manufacturing is and what are the challenges. It will give an overview of 3D printing technologies and important steps towards its certification process.

The presentation will outline current projects which represent some of the earliest and most significant efforts to undertake a dedicated technical approach to certify AM products. The first additively manufactured component for the Oil & Gas Industry has already been certified by LR and we are working together with experts in the ISO / ASTM /ASME Additive Manufacturing standards committee to update regulations and standards, resolve challenges and accelerate industry adoption.

**Keywords:** 3D print, materials, research, manufacturing, certification



## STATISTICAL ANALYSIS OF SHEAR STRENGTH OF WELDS IN WELDED FABRIC FOR CIVIL ENGINEERING WITH APPLICATION OF NEW TOOL DESIGN

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Many new requirements in the field of testing of metals, emphasize traceability and accuracy of test results as of the end goal. These requirements have hidden context with emphasis on energy efficiency too., which is initiated by mass productions and increasing market of end products based on steels for civil engineering applications. The noted above move focus on improvements of existing test methods. The key point in the verification of improvements is a statistical analysis of test results. This paper describes one such analysis, based on results obtained by application of new insert tool for testing of shear strength of welded fabric for civil engineering. This paper presents an extensive series of experimental results to verify the efficiency of the application of the new insert tool. The obtained results justify applications of a new tool insert to reduce data scatter during testing of welded fabric for civil engineering.

**Keywords:** prestressing steel; welded fabric; shear strength; statistical analysis.

## THE EFFECT OF INCONEL 718 ADDITION ON THE CAVITATION EROSION OF NICKEL MATRIX COLD SPRAYED COATINGS

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Cavitation erosion is a critical problem commonly encountered in hydraulic machinery such as turbine blades, valves, propellers, and pipelines. It is commonly caused by a rapid drop of fluid pressure and the generation of steam bubbles which may subsequently collapse on the metal surface. The released energy can lead to local plastic deformation of the material and mass removal, which although obtained in microscale, its extended action can result in malfunction or even failure of the component.

The current study aims to investigate the efficiency of metallic coatings to the mitigation of cavitation erosion damage by using the Gas Dynamic Cold Spraying technique. Pure nickel and mechanically blended nickel-Inconel 718 powders, with 1:1 ratio, were used as feedstock while a custom-made cold spray system was employed to accelerate the particles to supersonic velocity by using nitrogen as propellant gas. Duplex stainless steel 1.4462 was used as a substrate in the form of cylindrical billets with 30 mm diameter.

After the cold spray process, samples of appropriate dimensions were extracted and examined in terms of porosity and microhardness. The cavitation erosion conditions were simulated according to ASTM G32 by using a vibratory apparatus. During the testing, the progressive mass loss of the specimens was measured, and image analysis on the eroded surfaces was performed at predetermined time intervals. Also, Scanning Electron Microscopy was utilized to reveal the characteristics and morphology of the accumulative cavitation damage. The analysis of the experimental results focused on the effect of Inconel 718 addition on the cavitation erosion resistance of pure nickel matrix. The obtained results showed better cavitation resistance for samples with Inconel 718 addition.

**Keywords:** cavitation erosion, cold spray, nickel, Inconel 718.

## COMPOSITION DEPENDENCE OF INTERFACE ENERGY AS A DRIVING FORCE FOR DIFFUSION BONDING OF CERAMICS

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Diffusion bonding of ceramics with a metallic interlayer can deliver a variety of joint microstructures including a seamless bond. When applied to ZrC with Ti interlayer such procedure can deliver the seamless joint, depending on the parameters of the process, of which the thickness of the interlayer is particularly relevant. Experiments indicate the existence of the critical interlayer thickness, below which the seamless homogeneous joint is obtained, and above which the joint does not homogenize.

We analyze the thermodynamics and kinetics of the diffusion bonding process with metallic interlayer, uncover the driving forces for the diffusion and phase transformations, and, explain the critical thickness of the interlayer. We begin with the sharp interface thermodynamics, then develop the phase field model which predicts the observed behavior.

During the bonding procedure (heating-hold-cooling), Ti first undergoes a phase transformation from hcp to bcc. This and the thermal expansion mismatch between Ti and ZrC results in the compressive stresses in Ti. At hold temperature, the key process is the diffusion of carbon from ZrC into Ti, which, when the critical carbon concentration is reached, initiates the phase transformation of bcc Ti (with interstitial C) to B1 structure (TiC) identical to ZrC structure. The binary Zr/Ti diffusion is then driven by entropy and results in a seamless Zr(Ti)C joint.

Analysis of phase diagrams of Zr-C and Ti-C systems and elastic strain energies indicate that the changes in bulk free energies resulting from small changes in carbon concentrations oppose the diffusion of carbon. We show that the only component of the total system energy that decreases with carbon transfer from ZrC to Ti is the interface energy. Specifically, the interface energy must depend on the jump in the carbon concentration across the interface, in such a way that a lower concentration jump produces lower interface energy.

The critical film thickness is then estimated as the ratio of the change in interface energy with the concentration jump to the change in bulk energy density (elastic and/or chemical). The sharp interface model yields an estimate for the critical thickness of 10 microns, while the phase field simulations of the diffusion bonding process with a range of film thicknesses predict the value of 32 microns, both in good agreement with experimental findings (10-50 microns).

The analysis and methods apply to the broad range of transition metal carbides.

**Keywords:** interface energy, composition dependence, carbon diffusion, phase field model

## INVESTIGATION OF METAL POWDERS USING X-RAY COMPUTED TOMOGRAPHY

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X-ray Computed Tomography (CT) is a modern method for nondestructive testing of various materials which enables its wide application in both research and industry. Therefore, the CT scanning is a powerful tool for observation of the external and internal structures providing a comprehensive 3D image analysis of the observed sample. Investigation of the metal powders using CT scanning is a new approach based on inserting particles in a transparent matrix without damaging and/or changing particle morphology and shape. The CT scanning was performed on a Nikon XT H 225 system with reflection head. The reconstructed object data is in a voxel format, where the minimum resolution (voxel size) is 3 microns. In this study, changes in the morphology of metal powders obtained during mechanical alloying were investigated using scanning electron microscopy (SEM), laser particle size analyzer and CT scanning. It was observed that with increasing duration of the mechanical alloying the particle shape becomes flattered due to ball-particle-ball collisions. Particle size distribution results displayed that particle size has been changing during mechanical alloying, in the way where with increasing the time of mechanical alloying the amount of submicron particles increases up to the certain time of mechanical alloying, and particle size distribution becomes wider. 3D images obtained using CT scanning are in the excellent agreement with SEM images.

**Keywords:** X-ray computed tomography, metal powders, mechanical alloying,

## THERMODYNAMIC MODELLING OF 3C-6Si-1W-1Al DUCTILE CAST IRON

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In this study, a novel cast iron composition 3C-6Si-1W-1Al was designed to develop ferritic ductile cast iron for high temperature applications. In the designed alloy, Si content was kept at a higher value than the conventional ductile cast irons and aluminum was added to the composition since they both increase the  $A_1$  temperature. Initially, solidification sequence and possible phases of the designed alloy were determined by thermodynamic calculations using TCFE6 database for phase transformations and Scheil module for crystallization. The alloy was then produced according to ASTM E8 standard by sand mold casting. The cast alloy was characterized by microscopic examination, thermal analysis and X-ray diffraction to verify the ThermoCalc data. Calculations indicated that the stable phases at room temperature were ferrite,  $W_6C$  and graphite. Experimental results verified the room temperature phases, critical temperatures and also revealed that the graphite morphology deteriorated due to aluminum content.

**Keywords:** Ductile cast iron, alloy design, solidification, ThermoCalc, high temperature material.

# **Poster presentations**



## EFFECT OF POLYHEDRAL OLIGO SILSESQUIOXANES (POSS) PARTICLES ON CAVITATION RESISTANCE OF HYBRID COMPOSITE FILMS

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The objective of this study is to investigate the influence of the polyhedral oligo silsesquioxanes (POSS) structure on cavitation erosion of hybrid acrylate composite films. Cavitation is used as a method for estimation of adhesion quality of a composite film on a metal substrate. The hybrid composite films consist of UV-cured Bisphenol A glycidyl methacrylate/triethylene glycol dimethacrylate (Bis-GMA/TEGDMA) as matrix and reactive POSS structures as adhesion enhancers. POSS structure is established in dependence of content of hydroxyl groups and cross-linkable functionalities that affect the system compatibility. Composites are made with 1, 3, and 5 wt. % of three different types of POSS reagents containing: i) hydroxyl groups (POSS-M), ii) both hydroxyl and allyl groups (POSS-M-A), and iii) methacrylate groups (POSS-M-Cl). Morphology of composite films is examined by scanning electron microscope (SEM) and atomic force microscopy (AFM). The roughness after cavitation erosion of hybrid films with POSS-M-A determined by AFM was decreased for ~86% when compared to the polymer matrix. The loss of mass after cavitation erosion is significantly reduced by the addition of 5 wt. % of POSS particles. The best resistance to cavitation evaluated as the mass loss has a composite film with POSS-M-A particles and it exhibits ~94% lower mass loss when compared to the neat polymer matrix. Likewise, hybrid composite films with POSS-M-Cl and POSS-M particles decreased mass loss after cavitation test by ~91% and ~89%, respectively. The results indicate that improved mechanical properties and enhanced adhesion to the metal substrate contributed to improved resistance to cavitation erosion. Methods used in this study for estimation of adhesion quality and wear resistance clearly indicate that the best compatibility with Bis-GMA/TEGDMA matrix is achieved with POSS reagent containing both hydroxyl and allyl functional group.

**Keywords:** composite materials, atomic force microscopy, surfaces, electron microscopy.



## CAVITATION EROSION BEHAVIOR OF THE CUALNI SHAPE MEMORY SAMPLES

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Cavitation erosion testing of Cu-12.8Al-4.1Ni (wt. %) shape memory alloy samples was investigated. Samples for analysis were produced by continuous casting procedure obtaining CuAlNi bar of 8 mm in diameter. Microstructural and cavitation erosion testing were carried out on samples in as-cast state and after solution annealing at 885 °C for 60 minutes following room temperature water quenching. Metallographic analysis using optical microscope (OM) and scanning electron microscope (SEM) was performed. Examination of microstructure reveals martensite in both investigated samples.

Cavitation erosion resistance testing was applied using standard ultrasonic vibratory cavitation set up with stationary specimen. Weight loss and image analysis were used for determination of the effects of cavitation erosion.

Based on obtained results, very low values of mass loss were measured for both samples, after an exposure time of 420 minutes. Further image analysis of the samples and pits characteristics at the end of experiment pointed out very low cavitation erosion.

**Keywords:** cavitation erosion testing, CuAlNi, shape memory alloy, image analysis, microstructure

## GLASS- CERAMICS OBTAINED FROM COPPER MINE TAILINGS AND GLASS CULLETS

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Copper mine tailings are one of the most used secondary raw materials. The most common use is in the cement industry and as filler in road construction. The area of secondary raw materials utilization offering the highest potential is in development of glass-ceramic compositions, where the waste is melted to form a glass, fabricated using conventional glass forming techniques, and subsequently nucleated and devitrified in a controlled manner through heat treatment. The resultant, partially crystallized material offers improved strength, chemical and abrasion resistance.

Copper mine tailings were mixed with 30% glass cullets and melted in electric furnace BLF 17/3 at  $T=1450$  °C during  $t=0.5$  h. The obtained glass sample was black, without visible residual gas bubbles. X-ray powder diffraction (XRD) analysis confirmed the quenched melt to be amorphous. DTA and HSM analysis were performed on the previously prepared glass powder. The glass powder samples were pressed in pellets and sintered in an electric furnace at the temperature of 1040 °C for 2h and glass- ceramics were obtained.

Applications for these glass-ceramic materials would include structural products, pipe or pipelines to transport abrasive or corrosive fluids, alkali-resistant fibers to replace asbestos in concrete products, and use as a matrix material in which radioactive wastes could be chemically incorporated in a stable, non-leachable, solid form.

**Keywords:** glass, glass- ceramics, mine tailings.

## CHARACTERIZATION OF LANTHANUM-DOPED PHOSPHATE GLASS

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Due to their potential biomedical application, different types of phosphate-based glasses were studied and evaluated for drug delivery applications and bone tissue engineering.

The addition of lanthanum to modified apatite was found to improve its biocompatibility and lower the cytotoxicity against osteoblast.

In this paper, glass-ceramic with the composition  $42\text{P}_2\text{O}_5 \cdot 40\text{CaO} \cdot 5\text{La}_2\text{O}_3 \cdot 10\text{Na}_2\text{O} \cdot 3\text{TiO}_2$  (mol %) was obtained by the standard melt-quenching method. The raw materials used for glass synthesis were reagent grade  $(\text{NH}_4)_2\text{HPO}_4$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{CaCO}_3$ ,  $\text{La}_2\text{CO}_3$ , and  $\text{TiO}_2$ . Powder X-ray diffraction analysis confirmed the quenched melt to be vitreous.

To examine the glass crystallization non-isothermal crystallization was studied using DTA. Samples were crystallized at the appropriate temperature according to the DTA analysis. The XRD technique was used to identify the phase composition of the crystallized bulk glass samples.

XRD analysis of the powdered sample showed that during sintering the glass particles crystallized and the determined crystalline phases were:  $\alpha\text{-Ca}_2\text{P}_2\text{O}_7$ ,  $\beta\text{-Ca}_2\text{P}_2\text{O}_7$ ,  $\text{Ca}_{10}\text{Na}(\text{PO}_4)_7$ ,  $\text{LaP}_5\text{O}_{14}$ ,  $\beta\text{-NaTiOPO}_4$ ,  $\text{Ca}_2\text{P}_2\text{O}_7 \cdot 4\text{H}_2\text{O}$ ,  $\text{Ca}(\text{PO}_3)_2$ ,  $\beta\text{-Ca}_3(\text{PO}_4)_2$ ,  $\text{Ca}_3(\text{PO}_4)_2$ ,  $\gamma\text{-Ca}(\text{PO}_3)_2$ ,  $\text{NaCa}(\text{PO}_3)_3$ . For  $\alpha\text{-Ca}_2\text{P}_2\text{O}_7$ ,  $\beta\text{-Ca}_3(\text{PO}_4)_2$  and  $\beta\text{-Ca}_2\text{P}_2\text{O}_7$ , the bioactivity, i.e., the ability to promote the formation of apatite (HAP) layer after reaction with the surrounding body fluid, has been reported. The obtained phase composition of the glass-ceramic indicated its possible application as a bioactive material for bone tissue engineering.

**Keywords:** polyphosphate glass, bioactive glass-ceramic,  $\alpha\text{-Ca}_2\text{P}_2\text{O}_7$ ,  $\beta\text{-Ca}_3(\text{PO}_4)_2$ ,  $\beta\text{-Ca}_2\text{P}_2\text{O}_7$

## SYNTHESIS AND CHARACTERIZATION OF COMPOSITES BASED ON EXPANDED VERMICULITE AND FERRITE SPINELS<sup>3</sup>

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Expanded vermiculite (EV) is clay, a typical 2:1 phyllosilicate which may be easily produced from the mineral vermiculite. After expansion EV withholds vermiculites physical structure but becomes far more porous than vermiculite which is accredited to the huge increase in the intercalation layer thickness. This feature in conjunction with the fact that the intercalation layer enriched by intercalation cations and water was the initial motive for this research, which opened a novel method for spinels synthesis. Intercalation layer of the EV is utilized as a reaction medium for the synthesis of  $\text{Fe}_3\text{O}_4$ ,  $\text{MnFe}_2\text{O}_4$ ,  $\text{CoFe}_2\text{O}_4$ , and  $\text{FeCrFeO}_4$ . While magnetite is synthesized using the co-precipitation method, others are synthesized using hydrothermal co-precipitation method. Composites are then characterized by XRD (X-ray diffraction), FTIR (Fourier transformed infrared spectroscopy), SEM/EDS (scanning electron microscopy with energy dispersing microscopy) that confirmed the presence of the ferrite spinels inside the intercalation layer. After performed synthesis of the spinels, further parameters were determined: the isoelectric point (IEP), the capacity of cation exchange (CEC) and specific surface area (SSA) is calculated using Brunauer–Emmett–Teller (BET) adsorption isotherm of each composite together with matrix material (EV). The composites could be used as potential adsorbents of heavy metals and/or organic pollutants, and this synthesis method could be used for producing numerous compounds that are mainly being produced by solvothermal method or co-precipitation method.

**Keywords:** expanded vermiculite, mineral composites, hydrothermal alteration, ferrites

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### <sup>3</sup> Acknowledgments

The authors wish to acknowledge the financial support from the Ministry of Education, Science and Technological Development of the Republic of Serbia through the projects TR34023.

## EFFECT OF $\text{In}_2\text{O}_3$ ADDITION ON STRUCTURE AND PROPERTIES OF HIGH-ENERGY MECHANICALLY MILLED $\text{Ag-SnO}_2$

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Indium and its oxide have been used as additives to  $\text{Ag-SnO}_2$  contact materials for a very long time, at first to assist the internal oxidation process and later in a powder metallurgy route to increase dispersion of  $\text{SnO}_2$  in silver matrix and to aid activation of the sintering process. The latter is related to the fact that  $\text{In}_2\text{O}_3$  also acts as a wetting aid to  $\text{SnO}_2$  particles. This property of  $\text{In}_2\text{O}_3$  is particularly favorable for a high-energy ball milling processing route, as one of the alternative processes for improvement of homogeneity and thus performance of  $\text{Ag-SnO}_2$  contacts, which produces composite powders that usually exhibit considerable inactivity during sintering. In the present study, (92:8)  $\text{Ag:SnO}_2$  and (87.8:9.3:2.9)  $\text{Ag:SnO}_2:\text{In}_2\text{O}_3$  powder mixtures were processed by high-energy mechanical milling using planetary micro mill. The samples were milled for 3h at 600 rpm with 20:1 ball to powder ratio using 5 mm WC balls. The obtained composite powders were afterwards consolidated to solid pieces by pressing into  $\text{Ø}16 \times 3$  mm disks in a steel die under pressure of 100 MPa, which was followed by sintering of the compacts for 2h at  $920^\circ\text{C}$  in an air atmosphere. The solid disks were then forged at  $800^\circ\text{C}$  with the low degree of reduction followed by annealing at  $750^\circ\text{C}$  for 30 min and quenching in water to improve their density. Characterization of the obtained electrical contact materials included microstructural evaluation by X-Ray diffraction analysis (XRD) and scanning electron microscopy (SEM); density measurements; Vickers hardness measurements and microindentation hardness testing as well as electrical conductivity measurements. The results of XRD analysis indicate significant refinement of the microstructure after milling with a reduction of crystallite size of about ten times. Microstructures of both  $\text{Ag-SnO}_2$  and  $\text{Ag-SnO}_2\text{In}_2\text{O}_3$  materials after consolidation and sintering exhibit high dispersion of the metal oxides in Ag matrix and a certain degree of porosity. The observed presence of pure Ag (oxide-free) zones within the microstructure of the obtained  $\text{Ag-SnO}_2$  material was ascribed to poor wettability of  $\text{SnO}_2$  particles by the silver melt and their high thermal stability that facilitate segregation of pure Ag on the surface of the composite particle during sintering. It was found that the formation of these zones within the microstructure of the obtained  $\text{Ag-SnO}_2\text{In}_2\text{O}_3$  material was noticeably suppressed by addition of  $\text{In}_2\text{O}_3$ , they are much less pronounced and appear in the form of thin lines. This finding is also corroborated by the measured more consistent microhardness values. The obtained values of the other studied physical properties were found to be in accordance with the observed microstructures and within the required range for this type of electrical contact materials.

**Keywords:**  $\text{Ag-SnO}_2$ , high-energy ball milling,  $\text{In}_2\text{O}_3$ , microstructure refinement

## SOFTWARE SIMULATION OF THE PROPOSED INTEGRAL TREATMENT OF ACIDIC WASTEWATERS AND OVERBURDEN OF THE CEROVO COPPER MINE<sup>4</sup>

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As the main industrial branch in Bor over the past hundred years, mining had a harmful influence on the city of Bor and its environment. Acid mine drainage (AMD) produced in RTB Bor facilities, and during oxidation of sulfide minerals from mine waste deposits, pollute Borska and Kriveljska River. Both rivers confluence to the Timok River, a right tributary of the Danube. Due to geographic position and hydrology of the area, pollution of Borska River became a problem of international importance.

This paper considers software simulation of integral treatment of industrial wastewaters and off-balance parts of deposits. The simulation aims to provide additional quantities of copper and to prevent further pollution caused by mining, which is the essence of sustainable development and circular economy.

The original idea was to neutralize acidic industrial wastewater and water from the acidic Lake Robule by mixing with overburden from copper mine Cerovo. The direct mixing of overburden and industrial wastewater and overburden and wastewater from the Lake Robule was tested by software simulation. Software simulation provided data on the quantities of Cerovo overburden needed for the neutralization of industrial wastewater or acidic water from the Lake Robule and copper quantities that could be leached from overburden during treatment. Implementation of this type of integral treatment gives a great contribution to a cleaner production not only at the location "Cerovo". Integral treatment would lead to the consumption of minerals, copper revalorization, prevention of the AMD formation, and the safe disposal of processed overburden.

**Keywords:** Copper revalorization, waste flow materials.

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#### <sup>4</sup> Acknowledgments

The authors wish to acknowledge the financial support from the Ministry of Education, Science and Technological Development of the Republic of Serbia through the projects TR34002 and TR34023.

## PROPOSAL FOR INTEGRAL TREATMENT OF THE ACIDIC WASTEWATERS AND OVERBURDEN OF THE CEROVO COPPER MINE<sup>5</sup>

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As a result of the chemical and biological oxidation of the sulfide minerals present in the „Oštrejski planir” overburden, located in the vicinity of the city of Bor, acid mine drainage (AMD) is generated. After a period of heavy rains AMD interflows and collects in a geologically suitable environment, making an artificial Lake Robule. The water of the lake Robule has extremely acidic characteristics with high concentrations of metal cations, mostly iron, manganese, copper, and zinc.

The lake is populated by acidophilic iron-oxidizing bacteria. Overburden of the Cerovo copper mine contains 70% of the copper oxide minerals and 30% of the copper sulfide minerals (mostly chalcocite and covellite). Warm and extremely acidic industrial wastewater from the copper smelter ( $t=50^{\circ}\text{C}$ ,  $\text{pH} < 0$ ) would be used in the first step for copper leaching from the oxide phase of the Cerovo overburden. During leaching, the pH of the acidic industrial wastewater will increase, and the temperature will decrease to a level that is suitable for the survival of acidophilic bacteria ( $\text{pH} \text{ }^12$ , temperature around  $30^{\circ}\text{C}$ ).

After that, the partially neutralized solution would be depressed in a reservoir in which acidophilic microorganisms collected from acidic Lake Robule will be grown. Since large amounts of pyrite are deposited as waste in the Bor area, microorganisms would use pyrite as a source of energy for growth. After that, the resulting solution, rich in bacteria and  $\text{Fe}^{3+}$  ions, would be used for bioleaching of copper from the sulfide minerals.

After bioleaching, copper can be recovered by solvent extraction and electrolysis, and the solid residue can be disposed of as "safe waste", free from any further oxidation and possibility to generate AMD. The goal of integral treatment of the industrial wastewater, off-balance parts of deposits and ores deposited at the Cerovo mine from RTB Bor is to prevent further pollution caused by the AMD and the revalorization of copper from waste streams. Also, the benefit of this treatment is its possibility to exploit the existing negative characteristics of the mining waste.

**Keywords:** neutralization, revalorization, safe disposal

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### <sup>5</sup> Acknowledgments

The authors wish to acknowledge the financial support from the Ministry of Education, Science and Technological Development of the Republic of Serbia through the projects TR34002 and TR34023.

## CHARACTERIZATION OF METAL-GLASS COMPOSITES MATERIAL

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Parts of industrial machines and structures are often exposed to the action of aggressive environments which can provoke the loss of their integrity. Commercial stainless steels have a significant application in making machine parts in the industry because of their outstanding properties. The high prices of this material can sometimes be a limiting factor. For this reason, the idea is to replace expensive stainless steel with a lower price composite material. The work aims to create a metal-glass composites material as well as to examine the influence of basalt content on the properties of austenitic stainless steel.

Spherical gas atomized powder of commercial austenitic stainless steel (SURFIT TM 316L) with a narrow size range of 45-63  $\mu\text{m}$  was used in the experiment. The source of glass was basalt rock. Basalt from the Republic of Serbia has a relatively low melting point and low viscosity. The composite material was manufactured by mechanical mixing steel powders with fresh crushed basalt rock in diameter below 45  $\mu\text{m}$ . Mixtures of basalt content of 5, 10, 20 and 40 wt.% were prepared.

Green compacts were obtained by single-side pressing at 150 MPa using a steel mold. Sintering was performed at 1250 °C for 1 hour in a High-Temperature Vacuum Furnace. Starting powder and sintered composites were characterized by X-ray diffraction method (XRD). Morphology of powders, as well as microstructural development, were followed by a scanning electron microscope (SEM). Mechanical properties were tested by Vickers hardness.

**Keywords:** composite material, metal-glass, stainless steel, 316L, basalt.



## CHARACTERIZATION OF Ti6Al4V ALLOY OBTAINED BY HOT FORGING PROCESS<sup>6</sup>

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Titanium alloyed with aluminum and vanadium gives lightweight alloys with high specific strength, low Young's modulus and high corrosion resistance, which are alloy's essential properties that provide versatile applications in automotive, military, aerospace and other fields of industry. The Ti6Al4V alloy, besides its high creep and corrosion resistance, is a biocompatible material used for surgical prostheses, orthopedic implants, and medical instruments.

The aim of this work was to examine the structural and mechanical changes that occurred during the hot forging process, a process to obtain a surgical hip implant, and the subsequent heat treatment. Microstructural characterization, morphological and semi-quantitative chemical analyses have been accomplished using an optical and scanning electron microscope. Chemical analyses of all surfaces obtained by the dotted method in all analyzed samples have shown the approximate content of aluminum, titanium, vanadium, iron, and silicon. The chemical analysis means values of the central and peripheral parts, slightly different for each sample, indicating that there was no significant change after the hot forging process and subsequent heat treatment. Further, this suggests that such heat treatment would not lead to diffusion and redistribution of the elements, i.e. the low-temperature heat treatment can be conducted to reduce the stress.

Comparing the microstructure figures, the significant change neither in structure nor in morphology could be observed for both series of samples, with and without subsequent heat treatment. The ratio between  $\alpha$ - and  $\beta$ -phases remains approximately equal. The mechanical properties of this alloy are highly dependent on the ratio of the two phases that have been formed during the heat treatment. Mechanical testing additionally proved that heat treatment can be conducted below the  $\beta$  transition temperature (BTT).

**Keywords:** Ti6Al4V alloy, hot forging, microstructure

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<sup>6</sup> Acknowledgment

The authors wish to acknowledge the financial support from the Ministry of Education, Science and Technological Development of the Republic of Serbia through the project TR 34002.

## HIGH TEMPERATURE TRANSFORMATION OF THE AUSFERRITE MICROSTRUCTURE

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Mechanical properties of metallic materials strongly depend on microstructure. Microstructure changes of metallic parts at elevated temperatures highly limits possibilities of their application. The microstructure of austempered ductile iron comprises constituents that are unstable at elevated temperatures. Therefore, the thermal stability of austempered ductile iron at high temperatures has been studied. The dilatometric curve shows a large exothermic transition of the ausferrite microstructure during heating up to 700°C. Amount of carbon enriched austenite in the microstructure after reheating were studied in this paper using scanning electron microscopy.

**Keywords:** ausferrite, microstructure stability, scanning electron microscopy.

## THE INFLUENCE OF PREFORMS QUALITY ON STEEL CARTRIDGE CASE PRODUCTION

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Steel cartridge cases are usually produced of low or medium carbon steel. Plate or bar materials as a preform are used. Low and medium carbon steels have moderate strength and hardness in the as-rolled or extruded condition and good formability in the annealed condition. They can be further hardened by cold work. The formability of these steels allows a high reduction of the wall thickness during the manufacture of a cartridge case. During the production of cartridge cases various kinds of defects can occur, related either to the material (macrostructure, microstructure, impurities) or technology parameters (inadequate load, temperature, cooling rate, lubricants, etc.). These defects can be deleterious to the manufacture of a quality cartridge case.

In this work, the properties of different preforms for the production of cartridge cases made of three different steels were evaluated. Preforms 1 were slugs made of a hot-rolled plate of AISI 1030 steel, preforms 2 were cups made of ČR2 steel, and preforms 3 were slugs made of bars of C10E steel. Visual and magnetic particles inspection, as well as metallurgical examination, including chemical composition, macrostructure, microstructure, and hardness measurement, were performed. It was assumed that metallurgical properties, presence of slag and coarse inclusions had a decisive influence on the preforms (slugs) quality and cartridge case failure during production.

**Keywords:** cartridge case, steel, preforms, defects.

## RESISTANCE OF EN AW-7075 ALLOY IN T6 AND T77 TEMPER TO THE EXFOLIATION AND INTERGRANULAR CORROSION

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Aluminum alloys Al–Zn–Mg–Cu (7xxx series) have been widely used in aircraft structures due to their high strength. These series of alloys provide high strength in the T6 temper but are prone to stress corrosion cracking (SCC) and exfoliation corrosion. The T77 over-aged temper can effectively improve the corrosion resistance of these alloys without or with small loss of its strength gained in the initial T6 state. With the intention to predict corrosion behavior of the EN AW-7075 alloy in T6 and T77 temper condition, two test methods were conducted, exfoliation corrosion susceptibility (EXCO test) and intergranular corrosion resistance. It has been found that resistance to exfoliating corrosion is greater in the T6 compared to the T77 temper. On the other hand, EN AW-7075 alloy in the T77 temper has better intergranular corrosion resistance than in the T6 temper. It was assumed that differences in corrosion resistance between these two temper conditions (T6 and T77) result from different dispersion of precipitated second-phase particles within the original phase matrix, where the precipitate particles act as obstacles to dislocation movement.

**Keywords:** EN AW-7075 alloy, T6 and T77 temper, exfoliation and intergranular corrosion.

## THE INFLUENCE OF SOLUTION ANNEALING ON MICROSTRUCTURE AND MECHANICAL PROPERTIES HEAT-RESISTANT CAST STEEL HK30 MODIFIED BY NIOBIUM

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In the production of engines for high-performance cars, heat resistant steels are used today for the production of parts exposed to elevated temperatures. Regardless of the long period of their application, heat resistant steels are still subject to intense research, as it has been established that certain modifications can improve their exploitation properties and thus achieve competitiveness concerning the use of much more expensive materials, especially superalloys.

The subject of research in this paper is the heat resistant austenitic steel HK 30 according to ASTM A 351 M standard modified by niobium (HK 30 Nb) in centrifugally cast state, which is used for making turbine engine parts for trucks. In this paper, the mechanical properties at room temperature as well as the microstructure for steel HK 30 Nb were tested in the initial (cast state) and solution annealed state. Research has shown that the tensile properties after the solution annealing have a slightly lower value than the cast state. Microstructure analysis of samples in solution annealed state showed dissolution of primary carbides that were presented in the initial cast state. The morphology, distribution, size, and composition of carbides were changed during a solution annealing. Analysis of microstructure was done by SEM (Scanning Electron Microscope) microscope.

**Keywords:** heat-resistant cast steel; solution annealing; mechanical properties; carbide.

## **SURFACE MODIFICATION OF A TITANIUM IMPLANT MATERIAL BY A PICOSECOND ND: YAG LASER IN AIR AND ARGON ATMOSPHERE**

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Interaction of an Nd: YAG laser, operating at a wavelength of 1064 nm, pulse duration of 150 ps, and repetition rate of 10 Hz, with the commercially pure (CP) titanium in air and argon atmosphere was investigated. Laser surface modification of CP-titanium was studied at a different laser pulse energy values (5 and 30 mJ) and for a different irradiation durations (5 and 100 s). During the interaction of laser irradiation with the implant material (target) surface, a part of the energy was reflected and the part was absorbed. The laser energy absorbed by the implant material surface layer caused various chemical, morphological and structural changes of the implant material. Scanning electron microscopy (SEM) was used in order to examine morphological changes in the target surface induced by the laser irradiation. Interaction of the laser irradiation with the CP-titanium surface resulted in the formation of the surface craters. Moreover, the presence of the microcracks and hydrodynamic effects in the form of droplets and/or wave-like structures was also identified. Energy dispersive spectroscopy (EDS) revealed the formation of the titanium dioxide film at the target surface, which was more pronounced during irradiation in air. Presence of the surface titanium dioxide film is highly desirable due to its positive influence on the implant material resistance to further oxidative degradation. Modifications of the implant material surface, on the macroscopic and microscopic scale, were directly influenced by the irradiation parameters. Observed research results showed that the surface damage, caused by the short-term interaction of laser irradiation with the investigated implant material, is almost superficial. With an increase of the laser irradiation duration and the laser pulse energy, more diverse changes in the target surface morphology were observed and craters with different characteristics (dimensions and shapes) were formed. Observed morphological changes were additionally characterized by profilometric analysis and differences in the target damage degree along the target depth were also detected. Namely, the increase of the irradiation duration and the laser pulse energy leads to the increase in the crater depth. The higher damage degree along with the target depth under the same investigated conditions occurred during the irradiation in the argon atmosphere. In conclusion, the results of the presented study showed that the irradiation parameters variation directly influence the target damage characteristics and in that way can significantly influence the implant material biocompatibility and osseointegration characteristics.

**Keywords:** CP-titanium, surface modification, picosecond Nd: YAG laser, laser-induced damage

## END-BLOW CARBON CONTROL IN SMALL CAPACITY CONVERTERS - CHARACTERISTICS AND POSSIBLE IMPROVEMENTS

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The difficulties that today's shops with small capacity converters are facing in end-blow carbon content control are mainly caused by two factors: a) at the time they were designed, there had been no vision for future developments in methods of the turn-down performances control, or for some other reasons designs could not be adjusted, and b) those shops were not later modernized in accordance with technology trends of the BOF process and modern ladle metallurgy. The consequences were the impossibility or limitations in applying the two most common methods for end-blow carbon content control: 1. Static-dynamic models supported by sub-lances or in-blow drop sensors (due to the need for large investments and because of the small diameter mouth of the converter), and 2. Chemical off-gas analysis (because small capacity converters are predominantly with full combustion with no CO at locations commonly used for the sampling of gases for chemical analysis, whereas an indirect estimate of the carbon content based on the CO<sub>2</sub> content in gases is unreliable). The subject of this paper is an overview of reasons for unsatisfactory end-point carbon content hit rate on the small capacity vessels (about 100t) and related metallurgical consequences. It was assumed that the carbon content estimate was based exclusively on the appearance of the flame on the mouth of the converters since the recommendations of the static models are not applicable. The additional consequences of unsatisfactory end-point carbon content hit rate for shops without bottom stirring, and slag detection during taping are included, with the focus on their impacts on the cleanliness of liquid steel. Several alternatives of the ISP (Intermediate Stop Practice) method have been listed, that, with few additional measures, can improve the success rate in achieving targeted carbon content. The need for exchanging (benchmarking) own production practices and values of key process indicators with similar BOF plants in the world is emphasized.

**Keywords:** process control, end-blown carbon, full combustion, yield, intermediate stop practice.

## SOFTWARE FOR THE REGULATION OF BURDEN DESCENDING SPEED THROUGH BLAST FURNACE

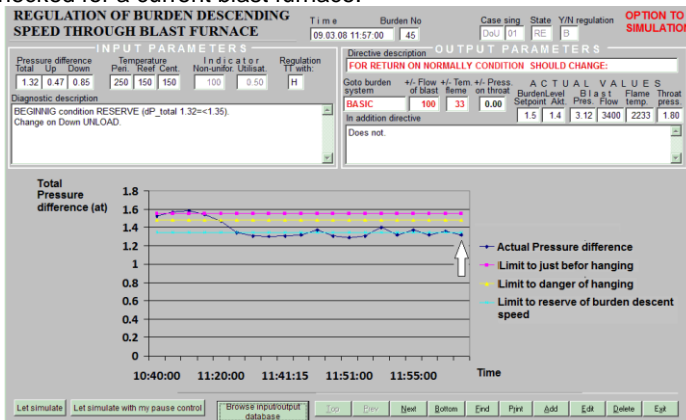
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Optimal descending speed (*DS*) of raw materials through blast furnace (*BF*) is one of the critical parameters to accomplish maximum productivity with minimum energy consumption. In most blast furnaces, operators "manually" control the descending speed – mainly by changing the airflow that enters in the BF. In the case of "manual" regulation, it is not possible to consider all the key factors affecting the *DS*, as well as the extent of their influence; the software, which is the subject of this paper, takes all the influencing parameters into account. The values of the software input parameters can be taken from the measuring instruments or the database of the acquisition system. With each change in the value of the descending speed influencing parameters, the software determines what the conditions for the *DS* are: if they are not in *NORMALLY* condition, the software calculates the correction values of the parameters for returning in *NORMALLY* condition. The correction is executed automatically via the blast furnace control system with operator verification or "manually". The software establishes the following general conditions for the descending speed: - *NORMALLY*; - *RESERVE* (can increase *DS*); - *JUST BEFORE HANGING*; - *DANGER OF HANGING*; (Note: hanging – as no burden descent). These conditions are determined by: Three limits and actual values of total pressure difference; Up and down pressure difference; Throat radial temperature (or  $\text{CO}_2$ ) on periphery, reef, and center. Three limits of total pressure difference and basic burden system are defined from previously collected data and periodically checked for a current blast furnace.



Software user interface for the regulation of burden descending speed through the BF

**Keywords:** descending speed, blast furnace, software, regulation, a burden.



## POSITION OF CRITICAL RAW MATERIALS WITHIN THE CONCEPT OF CIRCULAR ECONOMY

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As a contemporary way of living has critical raw materials (CRM) as essential addition for maintaining and improving quality of life, tackling the changes, and becoming an almost irreplaceable part of digital technologies, mobility, and low carbon energy technologies.

The sustainable supply of metals and minerals for high-tech applications became the issue of global concern by predictions for the future growth of demand for these materials. Having in mind CRM` high economic importance, and by EU reports on this subject, CRMs are at high risk of future supply disruption (some of them are of more concern than others, for instance, rare earth elements, niobium, and cobalt).

Dominant linear economy model, take-use-discharge, does not offer a solution for this growing problem, whereas the new circular economic concept gives some hope, as this model seeks opportunities to keep products/materials/resources in active use as long as possible. In the light of new relationships between producers and consumers, it is important to identify the factors significant for efficient use of all waste materials, particularly critical raw materials (CRM) and to identify gaps that limit the performance of the processing chains and hinder closing the loop for these materials.

Consuming CRM obtained from specific waste streams, as a result of a proper introduction of circular economy systems, could mean sustainable economic growth, sustainable industrialization and a contribution to achieving sustainable management of natural resources and their effective use. All this also includes discussing the potentials for substitution of particular critical raw materials, within the sustainable design concept, but this ought to be part of multidisciplinary, consistent research as well as wise legislative incentives.

The paper presents the prevailing flows of critical raw materials and the potentials for obtaining CRM by use of urban secondary sources in an efficient and effective way, within the framework of the circular economy.

**Keywords:** critical raw materials (CRM), circular economy, resources.

## QUARTZ SAND PROCESSING METHODS FOR THE APPLICATION IN WATER GLASS PRODUCTION<sup>7</sup>

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To use quartz sand for water glass production, processing of raw quartz sand is necessary. The objectives of processing are: achieving the required granulometric composition, increasing the content of SiO<sub>2</sub> and reducing the content of impurities that lower its quality. The most frequently used method is the attrition cleaning in combination with gravity and magnetic concentration. In order to eliminate quartz sand impurities to a greater extent, it is approached to different methods of preparing mineral raw materials, which depends of the content and type of impurities as well as on the way they appear (as individual grains, surface coatings on quartz grains or as a form of intergrowth with quartz). The size reduction processes are used when it comes to sandstone or large-grained sand, whereby the size decreases up to 0.6 mm. Besides that, washing, attrition scrubbing, grading and also gravity, flotation, and magnetic concentration are used. Desliming, washing, and grading procedures are used almost always because there is clay present in the quartz raw material as impurities. The scrubbing procedure in attrition machines is used when the quartz surface is kaolinized and limonitised. The flotation concentration is used when in raw material beside the quartz, there is mica and feldspar, with preceding washing and grading procedures to eliminate the clay component. That is so-called „reverse“ flotation consisting of flotating mica and feldspar is applied, and the pure quartz remains in the pulp. The magnetic separation process is used when in quartz raw material are also magnetic impurities type Fe<sub>2</sub>O<sub>3</sub> as carriers of total iron. Efficacy of the removal of iron is expressed as a rate of reduction of Fe<sub>2</sub>O<sub>3</sub>. In the company „Kesogradnja d.o.o.“ at Kozluk, near Zvornik (Republic of Srpska) from quartz sand deposit „Bijela Stijena Skočić“, quartz sand for the production of water glass was obtained by the procedures of washing, grinding, sizing and magnetic separation.

**Keywords:** quartz sand; separation; magnetic separation; water glass.

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<sup>7</sup> Acknowledgments

The authors wish to acknowledge the financial support from the Ministry of Education, Science and Technological Development of the Republic of Serbia through the project TR34013.

## GRAPE POMACE HYDROCHARS AS POTENTIAL ADSORBENTS OF Cd(II) AND Al(III) FROM AQUEOUS SOLUTIONS

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Recently, there is a growing interest in the utilization of waste biomass to curb its potential negative impact on the environment, which includes CO<sub>2</sub> emission and various types of soil pollution. Simultaneously, the development of thermochemical technologies for conversion of waste biomass into valuable materials becomes very attractive. Hydrothermal carbonization is proposed as a promising and highly effective technology in this field.

In this paper, grape pomace was hydrothermally carbonized at 220 and 240°C to explore the potential application of derived hydrochars (HC-220 and HC-240, respectively) for removal of Cd(II) and Al(III) from aqueous solutions. Obtained preliminary results showed that adsorption capacities achieved using the HC-220 were 65.25 mg/g for Cd(II) and 17.13 mg/g for Al(III). On the other hand, the HC-240 showed smaller capacities for both examined materials (24.25 mg/g for Cd(II) and 9.0 for Al(III)). Differences in the structural properties of hydrochars produced at different carbonization temperatures could be a reason for this observation. Literature data states that higher temperature causes the formation of energy-dense coal-like hydrochars with dominant aromatic structure. Therefore, material obtained at 240°C had less functional group on its surface and more aromatic structure compared to the HC-220, and thus less electron donating sites for metal ions adsorption.

Results from this paper suggest that the grape pomace could be a promising precursor for the production of low-cost hydrochars for adsorption of Cd(II) and Al(III) from wastewaters. Besides, results can be further used for the optimization of the HTC process parameters to find the most adequate reuse of the waste grape pomace.

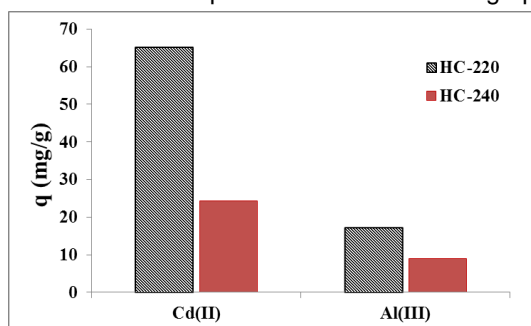


Figure: Cd(II) and Al(III) removal using HC-220 and HC-240

**Keywords:** Hydrothermal carbonization, Hydrochar, Adsorption, Cd(II), Al(III)

## ALKALI MODIFIED CORN COB HYDROCHAR AS BIOSORBENT OF $Mn^{2+}$ IONS FROM AQUEOUS SOLUTIONS

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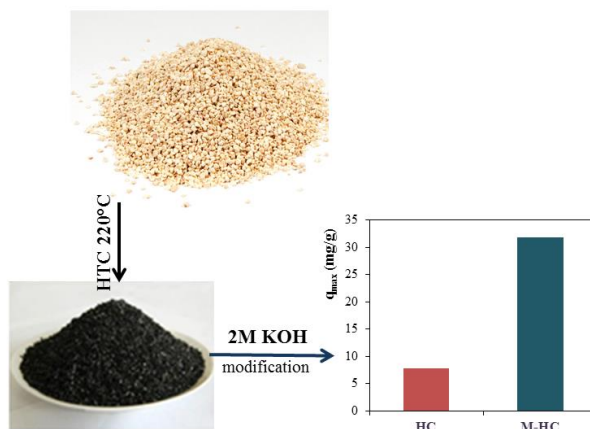
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In this study corn cob hydrochar was investigated as potential material for the biosorption process. The corn cob hydrochar obtained at different carbonization temperatures (180, 200 and 220°C) were examined as potential adsorbent of  $Mn^{2+}$  from aqueous solution.

The adsorption capacity of corn cob hydrochar obtained at different carbonization temperatures followed the decreasing order: 220°C > 200°C > 180°C

To enhance adsorption potential, hydrochar with best adsorption abilities obtained at 220°C (HC) was subjected to cold alkaline modification. The modified corn cob (M-HC) was prepared through a chemical treatment process using 2M KOH solution. Preliminary results showed that the M-HC exhibited excellent adsorption characteristics for  $Mn^{2+}$  removal from aqueous solution. The maximum adsorption capacity ( $q_{max}$ ) of M-HC and HC was 31.83 and 7.83 mg/g, respectively.

It can be concluded that modified corn cob hydrochar can be used as a low-cost, eco-friendly and promising adsorbent with a good adsorption capacity for Mn ions removal from aqueous solutions.



**Figure:** Adsorption capacity of HC and M-HC for  $Mn^{2+}$  removal from aqueous solutions

**Keywords:** corn cob, hydrothermal carbonization, alkali modification,  $Mn^{2+}$  biosorption.

## MICROSTRUCTURAL AND THERMAL CHARACTERIZATION OF 3.2C-5Si-1W NOVEL DUCTILE CAST IRON

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Ferritic ductile irons are preferred as high temperature materials such as exhaust manifold materials because of their sufficient oxidation resistance, good castability and low cost. However, the maximum service temperature of these cast irons remains low due to increased engine power requirements. In order to increase their maximum service temperature by increasing  $A_1$  temperature, elements like Al, Si, Cr, etc. must be added to the cast iron composition.

In this study, a novel cast iron composition consisting of 3.2 C, 5 Si, 1 W wt. % was developed and microstructural and thermal characterization was carried out. In the first stage of the study, thermodynamic calculations were performed by ThermoCalc software using TCFE6 database in order to determine the phase transformations and critical temperatures ( $T_L$ ,  $T_S$ ,  $A_1$ , etc.). In the second stage, thermal analyses were done to verify the calculated data. The calculations and experimental data revealed that the novel cast iron has ferrite, nodular graphite and W-rich carbides in the final matrix. The  $A_1$  temperature was determined to be higher than conventional cast irons, indicating that the studied alloy can be a candidate material for high temperature applications.

**Keywords:** Ductile cast iron, alloy design, ThermoCalc, high temperature material.

## STABILITY OF GOLD COMPLEX BASED ON MERCAPTOTRIAZOLE IN ALKALINE MEDIA

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Gold complex based on mercaptotriazole can be synthesized in a wide pH range (2-12). The stability of the gold complex in alkaline media was investigated in this research. Investigation was performed in the period of one year by visual monitoring, and electrochemical characterization of electrolytes. Electrochemical characterization was performed by open circuit potential measurement, cyclic voltammetry and polarization measurements with pH values of the electrolyte measurement before and after each electrochemical experiment at different pH values: 9 and 12 at optimal concentration of gold in the electrolyte of 2.5 g/dm<sup>3</sup>. For the electrolyte with pH=9, in the period of one year no visible signs of electrolyte decomposition were observed. After one month, for the electrolyte with pH=12 the appearance of precipitate in a form of brown flakes was observed at room temperature (25 °C), which was dissolved by heating the solution to the working temperature. After two months except brown flakes precipitate, a small amount of reduced elemental gold was observed. After three months of standing, the electrolyte was completely disintegrated.

**Keywords** stability, gold complex with mercaptotriazole, alkaline media, electrochemical characterization

## EFFECT OF ALUMINA COATINGS ON CORROSION BEHAVIOR OF X10CrAlSi7 STEEL IN SULFURIC ACID

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The results of experimental studies of the effect of the plasma sprayed alumina coating on the improved corrosion resistance of the heat-resistant ferritic stainless steel (W.Nr. 1.4713, EN X10CrAlSi7) in the sulfuric acid are presented in this research. In this investigation, the plasma spray process was employed on the substrate of ferritic stainless steel with deposition of Al<sub>2</sub>O<sub>3</sub> ceramic coatings with Ni-5%Al as a bond layer. Two different coating thicknesses of 0.3 mm and 0.5 mm were tested. The investigation was performed by electrochemical characterization and weight loss method. Electrochemical characterization of corrosion behavior of steel with and without ceramic coating was performed by: the open circuit potential measurement (OCP), the linear polarization resistance (LRP) method, and the Tafel extrapolation method. All electrochemical measurements were performed in the sulfuric acid with a concentration of 0.25 mol/dm<sup>3</sup> (pH ≈ 1.00). Weight loss method was performed with a duration of 240 h in the 38 wt.% sulfuric acid. Corrosion current densities obtained by electrochemical methods with the coatings were between 3 and 4 % of the values of the samples without coatings. Nearly the same results were obtained with the weight loss method. In both cases, these values are similar to the porosity of the coatings.

**Keywords:** corrosion, alumina coating, ferritic stainless steel, sulfuric acid.

## MICROSTRUCTURAL AND THERMAL ANALYSIS OF Cu–Al–Mn–Ag SHAPE MEMORY ALLOYS<sup>8</sup>

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Two quaternary Cu-Al-Mn-Ag shape memory alloys with nearly constant Al and Ag contents and variable content of Mn were prepared by arc melting of pure metals. Experimentally determined overall compositions of the investigated alloys were Cu-9.4%Al-1.1%Mn-3.7%Ag (alloy 1) and Cu-9.5%Al-5.6%Mn-3.9%Ag (alloy 2) (in wt.%). Microstructures of the prepared samples were investigated in the as-prepared condition and after heat treatment which included solution annealing at 850 °C followed by quenching into the water at room temperature. The effects of alloy composition and heat treatments on the microstructure and transformation temperatures of the investigated shape memory alloys were investigated by using scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS) and differential scanning calorimetry (DSC). It was determined that alloy with low Mn content (alloy 1) exhibits  $\alpha+\beta$  two-phase microstructure in the as-prepared state and martensite+ $\alpha$  two-phase microstructure in the as-quenched state. Ag was uniformly distributed between the coexisting phases. The microstructure of the alloy with a higher content of Mn (alloy 2) was fully martensitic in both investigated conditions. Phase transition temperatures, including martensite/austenite transition, order/disorder transformation of austenite phase, dissolution of  $\alpha$  precipitates and melting of the alloy were determined by using the DSC method.

**Keywords:** Shape memory alloy, Cu-Al-Mn-Ag alloy, Microstructure, Martensitic transformation

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<sup>8</sup> Acknowledgments

This work has been fully supported by Croatian Science Foundation under the project IP-2014-09-3405.



## THERMAL CONDUCTIVITY OF THE LOW-MELTING Bi-In EUTECTIC ALLOYS<sup>9</sup>

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Phase change materials (PCMs) are widely used in the field of thermal energy storage (TES). PCMs store the thermal energy of a phase change (typically fusion), called the “latent” heat of phase change, on heating, and then when cooled, they give off the stored heat by reversing the phase change. Low melting metals and eutectic alloys are a relatively new class of PCMs. The main advantages of the low-melting metallic materials usage as PCMs are their high volumetric latent heat and high thermal conductivity. Thermal conductivity represents an essential thermophysical property of PCMs because it controls the efficiency of the heat-transfer during the phase change. In this study thermal diffusivity of three Bi–In eutectic binary alloys (Bi–47.44In, Bi–66.33In, Bi–77.92In (all in at.)) was measured at room temperature using xenon flash method on a Discovery Xenon Flash DXF 500 device. Based on the experimentally determined values of thermal diffusivity and the specific heat capacity values obtained by thermodynamic calculation, the thermal conductivity of the investigated eutectic alloys at 25 °C was determined. It was determined that Bi–47.44at.%In alloy has the smallest thermal conductivity ( $8.131 \text{ Wm}^{-1}\text{K}^{-1}$ ), close to the thermal conductivity of pure Bi ( $8.2 \text{ Wm}^{-1}\text{K}^{-1}$ ). Obtained thermal conductivities for the Bi–66.33at.%In and Bi–77.92at.%In are  $12.182$  and  $19.880 \text{ Wm}^{-1}\text{K}^{-1}$ , respectively. Thermal conductivity of Bi-In alloys increases with an increase of In content although the values are considerably smaller than that for pure In ( $83,7 \text{ Wm}^{-1}\text{K}^{-1}$ ).

**Keywords:** low-melting alloy, Bi-In eutectic alloy, thermal conductivity, Xenon flash method.

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<sup>9</sup> Acknowledgments

This work has been fully supported by the Ministry of Education, Science and Technological Development of the Republic of Serbia, project No. OI172037.

## EVALUATION OF EN AW-5083 ALUMINUM ALLOY INGOTS HOMOGENEITY BY MEASURING HARDNESS

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The homogeneity of six aluminum alloy EN AW-5083 ingots cast with Direct Chill Process, in the as-cast state, was established in this work. In the experimental part, hardness examination was performed on specified places within the cross-sectioned 30 mm thick plate taken from the front and the rear of the as-cast ingots. The base for the homogeneity estimation was statistical examination plan ("Latin square"). Processing of hardness measurement results was performed by the software package StatSoft® STATISTICA. The differences in cross-sections of both front and rear cross-sectioned plates were determined by statistical analysis.

The homogeneity of the tested ingots was evaluated by comparing the grain number per unit area, and hardness across the front and rear cross-sectioned plates for all six as-cast ingots.

Quantification and statistical processing of obtained results lead to the following conclusions:

The effect of the *slice height (j)* ( $P = 0.001933$  for front and  $P = 0.009788$  for rear ingots) and *slice height (j)* ( $P = 0.000066$  for front and  $P = 0.000002$  for rear ingots) are very significant. The significance of differences between the charges was indicated for cast ingots' fronts.

Analysis of the influenced variable indicates the hardness changes within the particular charge and per slice height and width following the microstructure development, i.e., the linear correlation of number of grains per unit area in the as-cast state.

**Keywords:** aluminum alloy EN AW-5083, direct chill process, homogeneity, number of grains per unit area, hardness

## TUNDISH METALLURGY – ROUTE TO CLEAN STEEL MANUFACTURING

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The increasing demand for high-quality clean steels has entailed the continuous casting process to one of the most studied technologies of the steelmaking chain in recent years. To obtain quality steel from a continuous casting machine it is necessary to control many process parameters including ladle treatment, tundish treatment and finally continuous steel casting in a mould. The melting units like EAF and BOF are only of marginal influence on steel cleanliness. The main aims are to minimize the inclusion content in the liquid steel, to promote the separation of particles and to avoid reoxidation by ambient air, slags and refractory materials. Continuous casting tundishes normally have multiple outlets through which steel is continuously fed to the respective moulds at the constant rate. Because of this, tundish metallurgy has played a very important role in steel cleanliness, avoiding its reoxidation by air and ladle slag that retains inclusions, as well as aiding the removal of those same inclusions along with the continuous casting. Thus, beyond its traditional role as a buffer vessel, a steelmaking tundish is currently designed and operated to ensure maximum yield, superior cleanliness, negligible energy and materials losses and longer life.

On the other hand, the product such as submerged entry nozzles/shrouds (SEN/SES), exchangeable submerged nozzles (MT) and monoblock stoppers play an essential role in controlling liquid steel flow from the ladle down to the mold.

Finally, this review will consider the most influential factors in tundish metallurgy for clean steel production such as reoxidation, tundish size, tundish design, flow control, super-heat control, etc.

**Keywords:** tundish, clean steel, reoxidation, super-heat control.

## **Cu-Ni-Sn: INVESTIGATION OF THE EFFECT OF B AND Li ON MECHANICAL PROPERTIES**

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This paper presents the results of testing of some mechanical properties (tensile strength, yield strength, and elongation) and electrical conductivity of Cu-Ni-Sn alloy with B or Li or without these elements. Tests were performed on samples  $\varnothing 2\text{mm}$  that were cold-deformed as well as on samples after recrystallization annealing. The recrystallization annealing was performed at a temperature of 750 °C for a period of 60 minutes.

Mechanical characteristics were tested on a universal tensile testing machine. First, mechanical tests of cold-deformed samples were performed and then the cold-deformed samples were tested at the temperature range 20-500°C. The time of holding the sample at the test temperature was 15 minutes. Electroconductivity tests were performed on wire samples  $\varnothing 2\text{mm}$  in cold-deformed and annealing state by Thomson Bridge.

It was found that the value of  $R_m$  and  $R_{p0.2}$  decreases, while  $A$  increases with increasing test temperature. It has also been observed that Li has a somewhat greater influence on the mechanical properties of Cu-Ni-Sn alloy than B. In contrast, the presence of B in the alloy affects the increase in the value of electrical conductivity.

**Keywords:** CuNiSn alloy, tensile strength, yield strength, elongation.

## **Bi<sub>2</sub>O<sub>3</sub> INFLUENCE ON ELECTRONIC CERAMICS SINTERING PROCESS AND FINAL PROPERTIES<sup>10</sup>**

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The influence of Bi<sub>2</sub>O<sub>3</sub> as a functional additive on the process of obtaining cordierite, 2MgO-2Al<sub>2</sub>O<sub>3</sub>-5SiO<sub>2</sub> (MAS) was studied by sintering the MgO / Bi<sub>2</sub>O<sub>3</sub> system (sintered at 820 °C and 1100 °C), Al<sub>2</sub>O<sub>3</sub> / Bi<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> / Bi<sub>2</sub>O<sub>3</sub> (sintered at 1100 °C), the composition being 80% oxide and 20% Bi<sub>2</sub>O<sub>3</sub>. The effects of sintering, composition, and morphology were followed by X-ray diffraction, scanning electron microscopy and EDS analysis. It has been found that, in addition to the liquid phase, Bi<sub>2</sub>O<sub>3</sub> produces intermediate metastable compounds with MgO and Al<sub>2</sub>O<sub>3</sub>. The sintering of MAS ceramics with 10% Bi<sub>2</sub>O<sub>3</sub> at 1000 °C, 1100 °C and 1200 °C was also performed.

Binary systems MgO / Bi<sub>2</sub>O<sub>3</sub> at 820 °C and 1100 °C, Al<sub>2</sub>O<sub>3</sub> / Bi<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> / Bi<sub>2</sub>O<sub>3</sub> were sintered at 1100 °C, as well as MgO, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> / Bi<sub>2</sub>O<sub>3</sub> at 1000 °C, 1100 °C and 1200 °C to examine the reaction of the cordierite synthesis. The results show the formation of a liquid phase at 820 °C (melting temperature Bi<sub>2</sub>O<sub>3</sub>), as well as the formation of metastable compounds forming MgO and Al<sub>2</sub>O<sub>3</sub> with Bi<sub>2</sub>O<sub>3</sub> at 1100 °C and which diffuse through the liquid phase, thus enabling the mechanism of reaction in the multi-component system to be accelerated from two aspects. By the sintering of a three-component MAS system in the presence of Bi<sub>2</sub>O<sub>3</sub>, the existence of cordierite was established. The lowest temperature at which the tracer cordierite was observed (in hexagonal form - indialite) is 1100 °C, while in the sintered system at 1200 °C the most frequent phase is indialite. This study found that the presence of Bi<sub>2</sub>O<sub>3</sub> in basic cordierite mixture allows the sintering temperature to decrease by ~ 170 °C relative to the temperature of the formation of cordierite ceramics from the mixture without the presence of functional additives.

**Keywords:** indialite, sintering, mechanochemical activation

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<sup>10</sup> Acknowledgments

The authors wish to acknowledge the financial support from the Ministry of Education, Science and Technological Development of the Republic of Serbia through the projects OI172057 and TR34023.

## ACTIVATION AND RELAXATION TIME INFLUENCE ON CORDIERITE CERAMICS<sup>11</sup>

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Mechanochemical activation of  $2\text{MgO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$ , starting components of cordierite, was used in aim to decrease the sintering temperature. Cordierite is very attractive ceramic material due to its properties, excellent thermal shock resistance, low dielectric constants (~5) and low thermal expansions ( $20\cdot 10^{-7} \text{ }^\circ\text{C}^{-1}$ ). Specific surface area of the activated components was detected by the BET method. TG and DTA methods have followed behaviors of the three-component system. To see the influence of relaxation time on the activated components, FT IR has analyzed the starting components as well as activated mixture after 24h and 24 months.

TG analyses of starting cordierite mixture activated 5 to 240 minutes showed a mass decrease from 8 to 12%. DT analyses showed the influence of mechanochemical activation of the starting components as increasing energy of the starting cordierite mixture, which resulted in moving the endothermic and exothermic effects to lower temperatures. The increasing of the temperatures of these effects was about  $100^\circ\text{C}$ , depending on activation time.

IR analyses showed that relaxation time, that is laying-off time to the moment of sintering, do not influence changes of activated mixtures. Since the given results, it can be concluded that the activated sample has no changes to the moment of using for a certain purpose (sintering) for any time since activation.

**Keywords:** cordierite, sintering, activation

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<sup>11</sup> Acknowledgments

The authors wish to acknowledge the financial support from the Ministry of Education, Science and Technological Development of the Republic of Serbia through the projects OI172057 and TR34023.

## **FABRICATION AND PROPERTIES OF Cu-BASED COMPOSITE REINFORCED WITH ULTRAFINE WC AND NANO Al<sub>2</sub>O<sub>3</sub> PARTICLES BY POWDER METALLURGY PROCESS**

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The process of mechanical milling was used for synthesis of Cu-based composite powders reinforced with ultrafine WC and nano-sized Al<sub>2</sub>O<sub>3</sub> particles. Then, the mechanically synthesized WC/Al<sub>2</sub>O<sub>3</sub>/Cu composite powders, with 5-10wt% of WC, 2wt% of Al<sub>2</sub>O<sub>3</sub>, and a balance of Cu in composition, were consolidated to full density by hot extrusion processing. The synthesis of WC/Al<sub>2</sub>O<sub>3</sub>/Cu composite powders and the evolution of the powder morphology, the distribution of the WC and Al<sub>2</sub>O<sub>3</sub> phases, and the crystallite size of the Cu matrix, due to mechanical milling were characterized by X-Ray diffraction analysis (XRD), scanning electron microscopy (SEM), and Energy Dispersive X-Ray Spectroscopy (EDS). The mechanical and physical properties of the as-consolidated composite were characterized by tensile, hardness, electrical resistivity, and thermal expansion tests, respectively. The results show that, by milling for 80-100min with a rotation speed of 300rpm and a ball to powder mass ratio of 10:1, Cu-based composite powders with uniformly dispersed ultrafine WC and nano-sized Al<sub>2</sub>O<sub>3</sub> reinforcing particles and Cu crystallite grains of 0.3-0.4μm in average can be obtained. After consolidation by hot extrusion processing, the WC/Al<sub>2</sub>O<sub>3</sub>/Cu composite bulk material presents good mechanical strength, low thermal expansion coefficient, and relatively high electrical conductivity, with the yield strength, the hardness, the thermal expansion coefficient and the electrical conductivity falling in the range of 130-150MPa, 148-151 HB, 9.9-10.2×10<sup>-6</sup>K<sup>-1</sup> and 77-85%IACS, respectively.

**Keywords:** Cu-based composite; mechanical synthesis; extrusion consolidation; mechanical and physical properties

## SYNTHESIS AND CHARACTERIZATION OF NEW REFRACTORY COATINGS BASED ON BASALT

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Basalt is a hard volcanic rock. Good technical properties make basalt an important raw material for the production of glass and glass-ceramics for the application in various departments of industry. It is a cheap and widespread raw material for the synthesis of different products such as basalt wool, basalt fibers, basalt plastics, composite materials. The new application of basalt is to obtain refractory coatings and application in foundries and protection against fire, corrosion, and erosion. The basic components of the foundry coatings are refractory filler, binder, additives, and solvent. Preparation of basalt-based filler is done by grinding and mechanical activation at grain diameter 20 $\mu$ m. The composition of the coating was determined about the parameters of the casting process and ranged within limits: filler 92-95%; binder 2,2-2,5%; additives 0,5-1,5% and solvent to the density of coating: 2 g/cm<sup>3</sup>. Coating suspension with this density showed high sedimentation stability (below 4.5% of precipitated matters for 24h). These foundry coating can enable the development of new methods of casting alloys with casting temperature over 1200°C.

Composition of basalt-based protective coatings was determined by research and ranged within the limits: 80-85% refractory filler, 12-17% binder based on epoxy resin, with organic additives and solvent. To characterize the coating, an ultrasonic vibration method with a stationary sample was used in accordance with the ASTM G32 standard. The mass loss of samples in the function of the cavitation time was monitored. Sample surface degradation level was quantified by using the image analysis. During the test, changes in sample morphology were monitored by means of the scanning electron microscopy method. Cavitation rate was determined as an indicator of the resistance of the coating under the effect of cavitation. It has been shown that the cavitation rate in the case of coatings applied to metal surfaces is higher ( $v = 0.108$  mg/min) than in the case of coatings applied to non-metallic surfaces ( $v = 0.0435$  mg/min). In order to achieve greater resiliency of the coating under the effect of cavitation, good adhesion of the coating and a higher coating thickness are important. Test results can provide an estimate of the resistance of coat layers in similar operating conditions. These coatings are intended for the protection of metallic and non-metallic surfaces, parts of equipment in metallurgy and mining.

**Keywords:** basalt, refractory coating, cavitation resistance, mass loss, image analysis.



## REMOVAL OF COPPER BY PELLETIZED FLY ASH

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Recent investigations are focused on the possibility of waste utilization as potential adsorbents in wastewater treatments. Industrial by-products and wastes are almost zero-cost raw materials and at the same time, their utilization can contribute to the solution of management problem by improving the material efficiency within the several industrial activities. Fly ash as a potential hazardous solid waste produced like a by-product in power plants worldwide in a million tons has attracted researches interest for years. Fly ash has potential application in wastewater treatment because of its major chemical components (alumina, silica, ferric oxide, calcium oxide, magnesium oxide, and carbon), and its physical properties such as porosity and large surface area. However, the micron particle size distribution of the fly ash (0.5  $\mu\text{m}$  to 200  $\mu\text{m}$ ) that gives fly-ash poor hydraulic properties is the one that limits the use of this material in wastewater treatments. This obstacle can be effectively overcome through the agglomeration process such as pelletization. Fly ash can be efficiently pelletized using cement as a binder. For the production of the pellets with the satisfying mechanical properties, the required amount of the cement as a binder is 10 %. The dosage of plasticizer up to 3 % in relation to the amount of the cement, additionally improves the mechanical properties. Under the investigated pelletizing conditions, more than 80 % of the obtained pellets are suitable for the application in continuous systems for wastewater purification, from the particle size distribution point of view (1.0 – 5.0 mm). Obtained pelletized fly ash has been used as a sorbent for copper removal in a batch system. Copper adsorption by fly ash was examined using a range of initial Cu concentrations (25.0 to 600.0  $\text{mg dm}^{-3}$ ). Solid – liquid ratio was <sup>1</sup>100, and the investigated contact time was 120 min. After equilibration, all suspensions were centrifuged and the concentrations of the remaining Cu in supernatants were determined using AAS. The obtained results indicate that the rise of initial concentration leads to the higher adsorbed amount of the copper. The experimental data were analyzed using the Langmuir sorption model. According to this model, maximum adsorption capacity for copper removal by pelletized fly ash is 25.64  $\text{mg g}^{-1}$  and this is almost 3 times higher capacity compared to the one non-pelletized one (8.85  $\text{mg g}^{-1}$ ). The presence of cement leads to the higher pH value in the reaction solution (5.5 - 6.0) than in the one with micronized fly ash (4.6 – 5.1) and this higher pH value noticeably influence the removal efficiency.

**Keywords:** fly ash, pelletization, adsorption, Copper removal.

## ELECTROCOALESCENCE PROCESS BASED ON ELECTROHYDRODYNAMICS PRINCIPLES<sup>12</sup>

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**The problem:** In the solvent extraction (SX), operations and processes, the entrainment problems, important from, both, technical and economic reasons, are still challenging, and not well understood. Here presented is an example that will help to justify the idea of the coalescence process, and because this event occurs at small separations more appropriate term could be electrocoalescence process (EC). Hence, the EC process, as the subject of rheology or nano-rheology, may be situated on the border between the classical and quantum mechanics. The selected, representative, finely dispersed system (FDS)-double emulsion appeared, as an undesired consequence, in the designed, constructed, and exploited pilot plant; SX of uranium from wet H<sub>3</sub>PO<sub>4</sub>. The entrained double emulsion was droplet (H<sub>3</sub>PO<sub>4</sub>)-film [synergistic mixture 0.5 M D2EHPA-0.125 M TOPO in dearomatized kerosene (DTK)] structure submerged into the droplet homo-phase continuum.

**Electrocoalescence process:** A corresponding constitutive model of liquids was developed and applied to the selected double emulsion. Considering Marangoni instabilities of the first and second order and possible electrical analogues the EC process, in some extent, was elucidated, see Fig. 1 (a) the film rupture, and (b) its electrohydrodynamics. Hence, both, the qualitative and quantitative physical pictures of the EC process are presented and discussed.

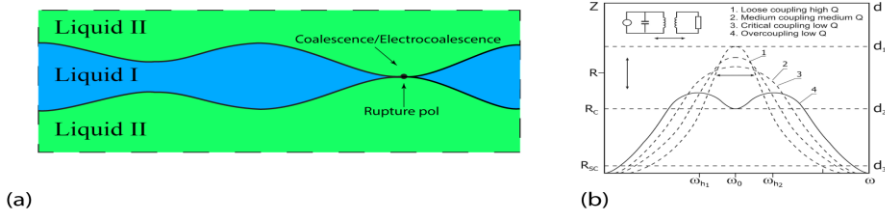


Fig. 1 (a) the double emulsion-rupture pole,  
(b) correlation impedance-frequency.

**Keywords:** nanorheology, solvent extraction, electrocoalescence, electroviscoelasticity.

<sup>12</sup> Acknowledgments

MESTD-Republic of Serbia, Grants No.: 34009 and 41010.

## EFFECT OF WATERBORNE ACRYLIC VARNISHES CONTAINING BARK EXTRACT ON THE WEATHERING PERFORMANCE OF IMPREGNATED WOOD

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Wood is an important material for construction and decoration purposes due to its unique properties such as easy workability, warm and healthy material. Transparent or semi-transparent coatings are designed to emphasize and preserve the natural beauty of the wood. The high performance and durability of coating systems are essential to maintain this appearance throughout the entire service lifetime of the end product. A drive for increased environmental sustainability of paint and coating systems has seen several innovations within the industry. Natural additives containing some starches, proteins and vegetable oils are integrated into coating systems. The attributes of natural additives consist of the polyhydroxyl patterns present of the flavonoid unit of condensed tannins. The chemical structures of these polyphenols also provide UV absorption and antioxidant protection roles in plants and trees. In this study, tree bark extracts are evaluated to be antioxidant and UV stabilizing functional additives in waterborne acrylic and styrene-acrylic coating formulations, considering the protective properties of polyphenols. The weathering resistance of 4 different test coating systems containing black pine, red pine, fir, and oak bark extracts were compared with the control coating containing commercial UV absorber. The UV absorbability of the test and control coating systems were determined by UV-VIS spectroscopy. However, the test and control systems were investigated on the impregnated scots pine wood surface. Because, in conditions where there is a risk of rotting, only the surface treatment does not provide effective protection. As a pre-treatment, the scots pine wood is impregnated with Tanalith E. The wood surface coated test and control systems were subjected to an artificial weathering test for 2016 hours. The color, roughness and macroscopic changes on the wood surfaces coated test and control systems were determined during the artificial weathering test. The changes on the dry film thickness of the coating systems were investigated by light microscope. Compared to the control coating, the color stabilization of the other test coating systems, except for the coating containing the black pine extract, was found to be high. The macroscopic and microscopic screenings have shown that the performance of the coating containing chestnut extract is high.

**Keywords:** artificial weathering, acrylic coating, bark extract, color change, impregnation, UV absorber.

## BIOLEACHING OF PYRITE BY MIXED CULTURES OF IRON AND/OR SULPHUR OXIDIZING BACTERIA ISOLATED IN ARMENIA

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Bioleaching of pyrite ( $\text{FeS}_2$ ) by pure and mixed cultures of new isolated iron- and sulfur-oxidizing *Acidithiobacillus* sp. 13Zn, *Leptospirillum ferriphilum* CC, *Acidithiobacillus albertensis* SO-2 and heterotrophic *Acidiphilium* spp. bacteria has been studied. According to data obtained the highest activity in pyrite oxidation among studied bacteria showed the pure culture of new isolated thermotolerant iron- and sulfur-oxidizing *Acidithiobacillus* sp. 13Zn. It was shown that the efficiency of *Acidithiobacillus* sp. 13Zn in the oxidation of pyrite was increased 1.8 times in association with *L.ferriphilum* CC. The association constructed on the base of *Acidithiobacillus* sp. 13Zn, *At.albertensis* SO-2 and *L.ferriphilum* CC allowed increasing twice the amount of total iron extracted from pyrite. Pyrite is acid insoluble and consequently according to sulfide minerals oxidation mechanisms can be dissolved only by ferric ion. Thus, the presence of *Leptospirillum* sp. bacteria in mixed cultures resulted in oxidation of ferrous ion ( $\text{Fe}^{2+}$ ) and regeneration of ferric iron ( $\text{Fe}^{3+}$ ), which in its turn accelerated pyrite oxidation. However, the highest activity in pyrite oxidation showed the association of *Acidithiobacillus* sp. 13Zn with heterotrophic *Acidiphilium* spp. bacteria. It is assumed that heterotrophic *Acidiphilium* spp. bacteria can utilize organic compounds contained in exudate or lysate of cells and thus reduce their toxic effect on autotrophic bacteria. Besides, heterotrophic bacteria excrete  $\text{CO}_2$  during respiration that can be assimilated by autotrophic bacteria in their constructive metabolism. Thus, synergetic interaction between different species of autotrophic chemolithotrophic bacteria and acidophilic heterotrophic bacteria in association leads to the enhancement of metal extraction from pyrite.

**Keywords:** bioleaching, pyrite, iron- and sulfur-oxidizing bacteria, heterotrophic bacteria, metal extraction.

## CONCEPTION OF TEXTURE IN METALLURGY, SCIENCE OF MATERIALS, GEOMORPHOLOGY, AND ARTS<sup>13</sup>

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Texture in the metallurgy or science of materials is strictly defined and determined, while in geomorphology is something different, especially in visual art. In the industry practice, it is common to study the texture, i.e., crystallographic orientations, of examined material with only one chemical composition, while in the art often is contrary, the texture is resulted from applying the different materials, whose role is to produce only the visual image - texture. Therefore, visual appearance is of great interest to the entire art. In the metallurgy and science of materials such a goal does not exist, but only the exact evidence of crystallographic orientations in products such as wire, sheet or coatings. Those principal textures will be summarized here.

In geology, as it also means in crystallography, the texture is clearly defined as favorable orientations of particular planes and their directions, while in geomorphology, the texture is used to determine the configuration of mountains and plains for mapping the landscapes, sometimes may be for military purposes.

The surface state of any artistic work may be perceived visually, by touch or both. Application of texture, among other elements of design, has an influence not only on our physical perception but also to evoke emotions. Making the emotions of texture present at the surface of artwork may be the goal of a designer or artist. Some examples will be shown here. However, in metallurgy or materials science when the presence of crystallographic texture analyzed by using Miller indexes and pole figures, there is no use in evoking emotion.

**Keywords:** texture, crystallography, geomorphology, textured surface in visual art

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<sup>13</sup> Acknowledgments

The authors wish to acknowledge the financial support from the Ministry of Education, Science and Technological Development of the Republic of Serbia through the project TR34002.

## INTRAGRANULAR NUCLEATION OF FERRITE IN TITANIUM-VANADIUM MICROALLOYED MEDIUM-CARBON STEEL DURING ISOTHERMAL TRANSFORMATION<sup>14</sup>

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Isothermal transformation behavior of Ti-V microalloyed medium-carbon steel was investigated using light microscopy and scanning electron microscopy. Mechanical properties were also examined by use of compression test. Samples were treated isothermally at 350, 400, 450, 500, 550 and 600 °C. To determine the start of intragranular ferrite formation, isothermal treatment was interrupted after different holding times and the samples were subsequently quenched. At temperatures above 500°C where diffusional transformations are taking place, idiomorphic intragranular ferrites were observed. With a decrease of isothermal treatment temperature, in the range where the shear transformation mechanism becomes prevalent, intragranularly nucleated acicular ferrite becomes predominant in the microstructure. Preferred sites for nucleation of acicular ferrite were found to be MnS inclusions treated by additions of Ca or complex MnS inclusions covered by V(C, N) precipitates. At intermediate temperatures, interlocked morphology of acicular ferrite was observed. At lower temperatures (350-400 °C) morphology of acicular ferrite changes from interlocked to sheaf type, which is rather similar to bainitic microstructure. Differences in the yield strength of the final structures after isothermal transformation were in accordance with the observed changes in the microstructures with temperature. Marked increase of the yield strength in the predominantly bainite or acicular ferrite structures obtained after isothermal transformation at temperatures below 350-450°C could be related to the incomplete reaction phenomenon.

**Keywords:** microalloyed steel, isothermal transformation, intragranular ferrite, acicular ferrite.

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<sup>14</sup> Acknowledgement

The authors are indebted to the Ministry of Education and Science of Serbia for financial support (Project OI174004).

## FINITE ELEMENT ANALYSIS OF THE CLEAVAGE FRACTURE IN MEDIUM CARBON V AND TiV MICROALLOYED FORGING STEELS

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Stress and strain distribution at the onset of cleavage fracture during four-point bending testing at liquid nitrogen temperature of two commercial medium-carbon V and TiV microalloyed forging steels, with predominantly acicular ferrite structure, was examined using finite element analysis. The finite element models were based on notched four-point bending Griffiths-Owens's type specimens, while the material mechanical properties data input was based on stress-strain curves obtained by tensile testing at liquid nitrogen temperature. Results of the modeling showed that there were no distinct differences in strain distribution along the distance from the notch tip between the two steel samples, aside from strain magnitude which stems from the differences in stress-strain curves. Based on the recorded load at fracture, the corresponding displacement calculated by finite element analysis was somewhat larger for the V steel. While the TiV steel breaks at cross-head displacement between 0.3 and 0.5 mm, the V steel breaks between 0.6 and 0.9 mm. Plastic strain at the cleavage initiation site for the TiV steel ranges from 0.0595 to 0.1612, while for the V steel these values range from 0.3694 to 0.6338. Observed differences in plastic deformations near the notch root, where cleavage initiation sites were detected, seem to reflect differences in deformation behavior at liquid nitrogen temperature.

Moreover, such difference could be ascribed to the observed differences in structure, primarily in the volume fraction of acicular ferrite. It was concluded that deformations are more uniformly distributed when acicular ferrite is predominant in the structure of the steel. It could be ascribed to the effect of "gradual yielding" related to the high dislocation density in acicular ferrite, somewhat lower yield stress and higher ductility of the TiV steel with predominantly acicular ferrite structure.

**Keywords:** microalloyed medium carbon steels, acicular ferrite, critical fracture stress, finite element model.

## AGE HARDENING BEHAVIOR OF Al-Mg-Si ALLOYS WITH DIFFERENT Mg AND Si CONTENT

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Age hardening behavior of industrially produced Al-Mg-Si alloys with different chemical composition was investigated in this study. The alloys were provided by ALPRO Alumil Group company as extruded rectangular profiles with 30 mm in thickness and 40 mm width. The extruded profiles were solution heat treated (SHT) for 1h at 510°C and 550°C, then rapidly quenched into water (WQ) and subsequently aged either at room temperature (NA) up to 70 days, or artificially aged (AA) at 180°C and 190°C for different periods of time.

Precipitation hardening potential of the tested Al-Mg-Si alloys was estimated through hardness measurements, while precipitation sequences during aging were followed by electrical resistivity measurements. Room temperature tensile testing and an instrumented Charpy impact testing were performed for all tested Al-Mg-Si alloys, in the peak hardness condition (T6 temper).

It was found that Al-Mg-Si alloys with a high amount of Mg and Si exhibited higher precipitation hardening ability under applied natural aging (NA) and artificial aging (AA) conditions. Solution heat treatment (SHT) at 550°C provided the higher potential for age hardening than solution treatment at 510°C, while hardness and strength levels were slightly improved after aging at 190°C compared to 180°C.

The results of the instrumented Charpy impact testing showed that alloys with low content of the main alloying elements give lower energy absorption than richer alloys with a high amount of Mg and Si. This indicated that a good combination of performance properties, including strength and impact toughness, was achieved in Al-Mg-Si alloys rich in Si and Mg<sub>2</sub>Si content.

**Keywords:** Al-Mg-Si alloys, aging, precipitation hardening, impact toughness.



## STUDY ON ROASTING PROCESS AND SILICON BEHAVIOR OF HIGH-SILICON ZINC CONCENTRATE

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Silicon in zinc concentrate is partially transformed into soluble silicon under roasting conditions, which is easy to be leached under acidic conditions. The formation of silica sol or even gel under specific conditions seriously affects the clarification effect and filtration performance. The metallurgy process of a high-silicon zinc concentrate ore was proposed. The process parameters on soluble silicon and silicon dioxide behavior in the roasting process were carried out, the results showed that: under the condition of temperature 850 ~ 950 °C range, with the increase of calcination temperature, the calcine soluble silica ratio increased, with the increase of zinc concentrate granularity fineness, calcine soluble silica ratio increased. After roasting, the vast majority of zinc sulfide was oxidized and desulfurized into zinc oxide. The proportion of zinc in zinc silicate changed from 0.04% of zinc concentrate to 10.08% of calcine, indicating that the majority of free silica changed into silicate by combining with zinc. For high-silicon zinc concentrate with a high content of free silica and coarse particles, the high roasting temperature can be adopted, so as to ensure high desulfurization rate and bed capacity. On the contrary, the lower roasting temperature should be adapted to inhibit a large amount of soluble silicon. The roasting parameters of high silicon and zinc concentrate should be determined according to the occurrence state of silicon and the comprehensive consideration of various technical and economic indexes. By controlling appropriate roasting process conditions, the production of soluble silicate was inhibited, which provided a basis for the determination of the roasting process of high-silicon zinc concentrate.

**Keywords:** high-silicon zinc concentrate, roasting, soluble silica, silicate.

## HYDROMETALLURGICAL TREATMENT OF EAF DUST BY ALKALINE LEACHING WITH THE AIM TO PRODUCE ZINC OXIDE WITH SPECIFIC CHARACTERISTICS

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The present paper describes individual steps of hydrometallurgical treatment of electric arc furnace dust (EAFD) with a more detailed focus on the alkaline leaching of this significant raw material in solution of ammonium carbonate ((NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>). Product of this process is zinc oxide (ZnO) with high purity and specific surface area that meets requirements of industry grade of ZnO known as "Active Grade". The contribution evaluates optimal conditions of designed process steps consisting of EAFD washing in distilled water, leaching of washed EAFD in (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>, cementation of impurities from the solution by means of powder zinc, crystallization and calcination of crystallized material with the aim to obtain ZnO. The obtained experimental results represent an important part of the research focused on optimization of individual steps of EAFD hydrometallurgical treatment at pilot plant, which is part of the Laboratory of Processing Industrial Waste established between Research and Development Center of Železiarne Podbrezová and the Institute of Recycling Technologies at the Faculty of Materials, Metallurgy and Recycling at the Technical University of Košice.

**Keywords** EAF Dust, Hydrometallurgy, Alkaline Leaching, Zinc Oxide

## STUDY OF A COLD DRAWING PROCESS BY SIMULATION AND EXPERIMENTAL TESTS

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The wire drawing is a manufacturing process used to produce wires, tubes and rods. It consists in a reduction in cross sectional area by plastic deformation where the raw material (wire rod) is pulled through a die. Some of the goals of the wire drawing process are the achievement of an excellent surface finish, good dimensional accuracies and increase in mechanical strength. However, this process generates inhomogeneous deformation due to different material flow into the drawing tool (die). The inhomogeneous deformation generates residual stresses, those present in a material free from the action of external loads and temperature gradients. Residual stresses are commonly related to distortions of shape and dimensional variations of the final products, appearing after heat treatments which are related to shape distortions, warping and dimensional variations, causing material and energy waste, in the case of waste generation and in attempts to correct nonconformities.

In this research, the influence of die geometry change on the surface residual stresses and the drawing forces of the bars manufactured by a cold-drawing process of SAE 1020 and SAE 1045 steel was examined. Computer models were carried out to simulate the process by using the Simufact.forming GP ® software in order to get the values of drawing forces, residual stresses, plastic deformations and the elastic return of the material. The models were validated by comparing results of simulation with experimental results and equations found in the literature. After the model validation, different die geometries were simulated and the results of the computational simulations were used to manufacture a scale die and a mechanical device to develop the physical process in a testing machine to compare with the industrial process. X-ray diffraction measurements were carried out to obtain surface residual stresses and the drawing force was taken directly from the testing machine to different die geometries, reductions, materials and lubricants of the process. The new die geometry allowed a reduction of the residual stresses level, but increased the drawing force when compared to the original process.

**Keywords:** Simulation, Cold Drawing Process, Residual Stresses, Drawing Forces, X-ray Diffraction

## OXIDATION ROASTING OF PENTLANDITE SAMPLES AT ELEVATED TEMPERATURES<sup>15</sup>

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It is known that virtually all of the nickel in nickel sulfide ores occurs in the mineral pentlandite [(Ni,Fe)<sub>9</sub>S<sub>8</sub>]. Therefore, pentlandite is the most common and abundant ore for extraction of nickel, accounting for over 60% of nickel world production [12].

The pyrometallurgical extraction of nickel from its sulfide ores is very similar to the copper extraction process from copper sulfide concentrates. In essence, iron and sulfur are eliminated by selective oxidation and fluxing. In fact, the oxidation of pentlandite is the most important chemical process in the pyrometallurgical route [3].

Because of all mentioned, the characterization of the structural properties of six pentlandite samples was carried out in this paper. The samples of pentlandite were roasted in air atmosphere for 30min, in five different temperatures: 400, 500, 600, 700 and 800 °C. Investigation of structural properties and obtained oxidation products was performed using X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS).

**Keywords:** Characterization, Nickel, Pentlandite, Oxidation

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<sup>15</sup> Acknowledgements

This paper was done in the frame project N<sup>o</sup>34023 by Ministry of Science and Technological Development of the Republic of Serbia.

## A NOVEL LEACHING PROCESS FOR LATERITE NICKEL ORE

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Reverse leaching process for laterite nickel ore is proposed. The process splits the conventional high acid pressure leaching process (HAPL) into two stages to realize leaching and iron removal. The first stage utilizes high acidity and atmospheric pressure to leach limonite so as to achieve the goal of high nickel and cobalt leaching rate; the second stage is under the condition of pressure to leach saprolite. In the second stage, iron precipitates due to the definite temperature and acidity and releases acid in the same time, the acid that released can leach saprolite, to realize the purpose of saprolite leaching and iron removal. The test results showed that: the laterite ore from the Philippines under the condition of first stage temperature 95 °C, retention time 5 h, initial pulp concentration 36%, 1ton ore sulfuric acid dosage, the atmospheric leaching rates of nickel and iron were 98% and 74% respectively. Atmospheric leaching pulp blended with saprolite pulp and under the condition of second stage temperature 150 °C, retention time 2 h, saprolite amount for 35% of the total amount of ore, nickel, and iron leaching rate were 93% and 1.5% respectively. Reverse leaching process breaks the traditional idea to leach limonite in atmospheric condition and leach saprolite in pressure leaching condition. Reverse leaching process can treat two typical kinds of laterite (limonite and saprolite) in one process, solve the problem that only one kind of laterite can be treated in the existed process. It is especially suitable for processing transitional laterite nickel ore that that stockpile in the mines and ports. The process greatly improves the resources comprehensive utilization.

**Keywords:** Laterite; Limonite; Saprolite; atmospheric leaching; pressure leaching.

## STUDY ON A NOVEL CHLORIDIZING VOLATILIZATION PROCESS FOR THE TREATMENT OF JAROSITE

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A novel chloridizing volatilization process for the treatment of jarosite stored in Serbia was developed. With the addition of chlorinating agent, a valuable metal such as lead, zinc, copper, silver, and indium are chlorinated and volatilized; iron is enriched in slag and used in iron making system after magnetic separation or other methods. Sulfur and calcium combine and form a stable calcium sulfate to avoid the production of sulfur dioxide that polluting the air. The effects of reducing agent, chlorinating agent, adding the amount of chlorinating agent and temperature on volatilization rate of zinc, lead, copper, silver, and indium were investigated. The results showed that: the addition of chlorinating agent, reducing agent and temperature all affected the metal volatilization rate, the addition of chlorination agent could improve the volatilization rate of lead, zinc, copper, silver, and indium, and the addition of reducing agent could further improve the metal recovery rate. Under the recommended conditions, the volatilization rate of each element is zinc 98.26%, lead 99.88%, copper 97.32%, indium 58.73%, and silver 95.22% respectively. The chloridizing volatilization process cannot only transform jarosite slag into general solid waste, but also comprehensively recover valuable metals such as lead, zinc, copper, silver, indium and iron, etc., and further comprehensively recover iron and carbon from jarosite slag, which completely solves the potential pollution of jarosite slag and has good economic benefits.

**Keywords:** Jarosite; chloridizing volatilization; comprehensive.

## LEACHING OF SOLIDIFIED/STABILIZED METALLURGICAL WASTE UNDER ENVIRONMENTAL CONDITIONS

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Waste generated during metallurgical processes is of great concern due to potentially high environmental impact. Solidification/stabilization (S/S) process of metallurgical waste is considered to be the best available technique for its treatment before disposal. Toxicity characteristic leaching procedure (TCLP) and EN 12457 leaching tests of solidified waste are standardized methods for evaluation of S/S efficiency. However, sometimes the results of these standard methods do not provide a realistic assessment of the potential leaching of pollutants from a treated waste.

To determine the actual leaching level of contaminants in a real environment, two types of metallurgical waste – secondary lead slag (SLS) and wastewater treatment sludge (WWTS) from the primary copper smelter, solidified/stabilized in cement matrices, have been exposed to atmospheric condition for one year. Experimental setup of leaching under environmental condition (LEC) test included L/S ratio of 1 according to the average annual amount of atmospheric precipitation for the scheduled sampling time, and sampling of drainage water two times during the year: after autumn/winter period (from October to April) and spring/summer period (from April to October).

Results of all three leaching tests (LEC, TCLP, and EN 12457) show that the concentrations of leached contaminants depend on the pH values of the applied leaching agent. The results of EN 12457 test are similar to the concentrations of contaminants obtained after the LEC test due to the use of distilled water as the leaching agent. This confirms the application of the EN 12457 test to determine the long-term effects of waste disposal. TCLP test results differ significantly. In the case of solidified WWTS, leaching with a glacial acid solution within the TCLP test overestimated the concentration of released pollutants under real environmental conditions. In the case of stabilized SLS, TCLP test results were notably lower than concentrations of contaminants obtained by LEC test.

**Keywords:** solidification/stabilization; TCLP; EN 12457; leaching.

## REMOVAL OF Mn(II) IONS FROM SYNTHETIC SOLUTION USING ADSORBENTS BASED ON APRICOT AND PEACH SHELLS<sup>16</sup>

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**Abstract:** Manganese occurs naturally in surface and groundwater due to mineral dissolution and leaching process, but also as a result of human activities such as manganese ore mining and processing, different alloys and salts production. It is also used as a gasoline additive, a component in ceramic/glass manufacturing, some agrochemicals (fungicides and fertilizers). Manganese is an essential element for the functioning of many enzymes and can serve as an activator of many others, but manganese is not biodegradable, and its bioaccumulation in living organisms can cause many diseases and disorders. In the present study, the biosorption efficiency for the manganese ions from synthetic solution by raw and modified apricot and peach shells has been investigated. These lignocellulosic materials were obtained from local juice factory, where they have been discharged as the waste. Removal of manganese ions was investigated using the following biosorbents: raw apricot shells (KK) (particle size <0.65 mm), modified KK with mixture of 2% alginate and bentonite (KKAIB), raw peach shells (particle size <100  $\mu\text{m}$ ) modified by: 2% alginate (KBAI), 2 mol/L  $\text{HNO}_3$  (KBM) and with the mixture of 10%  $\text{FeCl}_3 \times 6\text{H}_2\text{O}$  and 0.1 mol/L KOH (KBFe). Experimental biosorption parameters were: initial concentration of manganese ions: 35 mg/L; m/V ratio: 5 g/L; contact time: 24h and initial pH value of the solutions: 4.5. The results have shown that KK is the most suitable and cost-effective biosorbent for the removal of manganese ions from aqueous solution. As apricot shells are widely available in the Republic of Serbia as food industry waste, application of this biosorbent can help in minimizing waste disposal and in water treatment at the same time.

**Keywords:** biosorption, manganese ions, lignocellulosic biomass, apricot, peach.

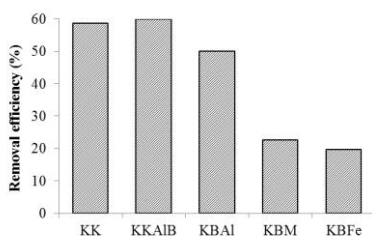


Figure 1. The removal efficiency of Mn(II) ions by different biosorbents

### <sup>16</sup> Acknowledgments

The authors are grateful to the Serbian Ministry of Education, Science and Technological Development of the Republic of Serbia for the financial support of this investigation included in the project TR 31003.





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CIP - Каталогизација у публикацији  
Народна библиотека Србије, Београд

**CIP**

669(048)  
66.017/.018(048)  
621.7/.9(048)

METALLURGICAL & Materials Engineering Congress of  
South-East Europe (2019; Beograd)

Book of Abstracts / Metallurgical & Materials Engineering  
Congress of South-East Europe (MME SEE 2019),  
June 5-7, 2019, Belgrade, Serbia ;[organized by]  
Associa-

tion of Matallurgical Engineers of Serbia [AMES] ...[et al.] ;  
editors Dragomir Glišić, Branislav Marković, Vaso Manojlović.  
Belgrade : Association of Metallurgical Engineers of Serbia  
(AMES), 2019 (Belgrade : Department of Printing Engineering  
Faculty of Technology and Matallurgy). - 84 str. : ilustr. ; 25 cm  
Tiraž 120. - Registar.  
ISBN 978-86-87183-30-8

1. Association of Metallurgical Engineers of Serbia (Beograd)

- a) Металургија - Апстракти
- b) Технички материјали - Апстракти
- c) Наука о материјалима - Апстракти
- d) Металоперађивачка индустрија - Апстракти

---

COBISS.SR-ID 276890124

*Publisher:*

Association of Metallurgical Engineers of Serbia  
Kneza Miloša 9/IV, Belgrade, Serbia  
[www.metalurgija.org.rs](http://www.metalurgija.org.rs)

