

10TH INTERNATIONAL CONFERENCE ON MATERIALS SCIENCE & ENGINEERING

Brașov – ROMANIA 8 – 11 March 2017

Coordinated by: Faculty of Materials Science and Engineering

BOOK OF ABSTRACTS





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TRANSILVANIA University of Brasov BRAMAT 2017 10TH INTERNATIONAL CONFERENCE ON MATERIALS SCIENCE & ENGINEERING 9 - 11 MARCH 2017, BRASOV, ROMANIA



Organized by: Faculty of Materials Science and Engineering – Transilvania University of Brasov Supporting Organizations: Academy of Technical Sciences of Romania – ASTR, Romanian Association of Heat treatment and Surface engineering – ATTIS, Romanian Foundry Technical Association – ATTR, Romanian Welding Society – ASR

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The Organizing Committee wishes to all the participants at BRAMAT 2017 "Welcome at TRANSILVANIA University of Brasov!"

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Program (Outline)

Wednesday, 08.03.2017

16.30: Registration (Hall H)

- 17.30: Presentations (Main Hall A)
- 18.10: Ceremony (Main Hall A)
- 18.30: Violin (*Eszter KLEINMAN-STANKOWSKY*) & piano concert (*Asaf KLEINMAN*) ISRAEL
- 19.45: Welcome cocktail (Hall H)

Thursday, 09.03.2017

8.30: Registration (Hall H)
9.00: Opening ceremony (Main Hall A)
9.20: Companies Introduction (Main Hall A)
9.30: Coffee break (Hall H)
10.00: Plenary lectures I (Main Hall A)
11.30: Coffee break (Hall H)
12.00: Plenary lectures II (Main Hall A)
13.30: Lunch (Hall H)
15.00: Keynote and oral presentations I (Sections I, III, IV, VIII) (Halls B, C, D, E)
16.45: Coffee break (Hall H)
17.00: Poster presentations I (Sections I, III, VI) (Hall G)
15.00: Companies Workshops (Hall F)
16.00: Workshop "Innovative valorization of metal waste" (Hall F)
9.30 - 19.00: Companies exhibitions (Hall G)
19.30: Sculptures and graphics exhibition (Hall R)

Friday, 10.03.2017

9.30: Plenary lectures III (Main Hall A)
11.00: Coffee break (Hall H)
11.30: Keynote and oral presentations II (Sections I, II, III, IV, V) (Halls B, C, D, E, F)
13.30: Lunch (Hall H)
15.00: Oral presentations III (Sections I, III, IV, V, VI, VII) (Halls B, C, D, E, F)
16.45: Coffee break (Hall H)
17.00: Poster presentations II (Sections II, IV, V, VII) (Hall G)
9.30 - 19.00: Companies exhibitions (Hall G)
20.00: Gala dinner (ARO night bar)

Saturday, 11.03.2017

9.30: Closing Ceremony, Poster Awards (Main Hall A) 9.00 - 13.00: Companies exhibitions (Hall G) 10.00: Sightseeing program meeting, 41A Iuliu Maniu Str., Braşov

- 11.00: Visit of Bran castle
- 13.30: Lunch in Bran



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PRELIMINARY PRESENTATIONS

Chairpersons:

Daniel MUNTEANU, Transilvania University of Braşov, ROMANIA **Andreea NECHIFOR**, Transilvania University of Braşov, ROMANIA



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THE NEED TO RE-ENGINEER ENGINEERING STUDIES

Nachum Finger

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Keywords: re-engineering process, engineering studies

Abstract: Our generation is witnessing drastic breakthroughs in technology, in information and in accessibility to existing knowledge. Some refer to these phenomena as a new "Industrial Revolution". To successfully cope with this revolution, engineering studies should undergo a re-engineering process.



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FROM THE BIBLE (AS BOOK) TO THE NANO-BIBLE (AS CHIP)

Strul Moisa

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Keywords: Bible, Nano-Bible, nanotechnology, FIB generator, gallium ions.

Abstract: The World's smallest Bible printed onto a single 5mm x 5mm surface, the Nano-Bible, "engraved" onto a Nano-Gold Stratum and a silicon substrate chip, smaller than a pinhead, is possible to read with an electron microscope. This outstanding achievement was possible by using one of the many advantages offered by nanotechnology: a focused ion beam (FIB) generator of gallium ions. The exposure will present the Bible's "adventure" - starting with manuscripts from Qumran / Dead Sea, through the Gutenberg Bible (the first major book printed using mass-produced movable metal type in Europe) and the mini-bibles as intermediary "stations" - to the actual "station": the Nano-Bible.



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PLENARY LECTURES

Chairpersons:

C. Barry CARTER, University of Connecticut, U.S.A. **Daniel MUNTEANU**, Transilvania University of Braşov, ROMANIA **Filipe VAZ**, Universidade do Minho, Braga, PORTUGAL

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P.01

IN-SITU AND OPERANDO TEM FOR MATERIALS ANALYSIS

C. Barry Carter

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Keywords: TEM, lithiation, nanomaterials

Abstract: Recent developments in transmission electron microscopy (TEM) [1] are well known and prize winning, while others are perhaps less obvious but are challenging 'conventional wisdom' all the same. We now have TEMs with the most important lenses corrected for spherical aberration (after being told for decades that it could not be done). For many researchers other improvements such as better stage stability, availability of FIB for specimen preparation and direct electrondetection cameras, are also having a great impact. In fact many in-situ experiments that are now producing so much excitement were possible 40 or more years ago but the results simply could not be recorded [2]! One challenge for the experimentalist is making the right the link to computer modeling—which is not, of course, a substitute for experimental observation. TEM is widely used to study nanoparticles. One challenge in the study of nanoparticles is that it is now so easy to produce 'pictures' that can be misinterpreted, but the use of 'old-fashioned' image simulation is often assumed to be unnecessary. So I will examine why it is still important to understand what the microscope is really telling us. I will illustrate these recent advances, and the challenges they bring. As an example, I will discuss defect processes occurring during lithiation as an example of using operando and video techniques in the TEM, and consider the future of 4D studies of these and related defect processes [3]. The lithiation process poses several challenges for the microscopists: Li has atomic number 3 so it is particularly light and does not scatter strongly, but it is also small especially when present as the ion Li+, and moves easily through many hosts which is, of course, why it is used in the lithium ion battery (LIB). TEM is the essential tool for characterizing, and thus understanding, nanomaterials. However, there are still many challenges that are inherent in studying nanomaterials, and many of these are glossed over when such studies are carried out. The challenges include the need for 3D information and for statistical analyses of the particles [e.g., 4].

Selective references:

1. Williams DB and Carter CB (2009) Transmission Electron Microscopy: A Textbook for Materials Science (Springer, NY) 2nd Edition updated with color and ~800 questions. The first Edition was published in 1996. (> 6,000 cites on Google Scholar incl. Chapters)

2. Carter CB and Williams DB Eds (2016) Transmission Electron Microscopy: Diffraction, Imaging, and Spectrometry Springer, Heidelberg. The Companion volume.

3. Janish, M.T. and Carter, C.B., (2015) Scripta Mater 107, 22–25 'In-Situ TEM Observations of the Lithiation of Molybdenum Disulfide'.

4. Roller, J., Yu, H., Vukmirovic, M.B., Bliznakov, S., Kotula, P.G., Carter, C.B., Adzic, R.R. and Maric, R. (2014) Electrochimica Acta 138, 341-352. 'Flame-Based Synthesis of Core-Shell Structures Using Pd-Ru and Pd Cores'.

Acknowledgements: The author thanks his collaborators, especially Dr. Matthew T. Janish, Dr. Paul G. Kotula and Dr. Katie Jungjohann, for many discussions and acknowledges the use of the TEMs at CINT, the Center for Integrated Nanotechnologies. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Department of Energy under contract DEAC04-94AL85000.

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P.02

NANOTECHNOLOGY AND ULTRASOUND FOR TARGETED DELIVERY OF DRUGS AND NUCLEOTIDES

J. Kost

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Keywords: nanotechnology, drug delivery, gene therapy

Abstract: The basic approach that drug concentration-effect relationships are significantly invariant, as a function of time in man has led to the development of constant rate drug delivery systems. Nevertheless, there are a number of clinical situations where such an approach may not be sufficient. Thus, in recent years several research groups have been developing targeted and stimuli responsive systems that could more closely resemble the normal physiological process. Advances in protein engineering and materials science have contributed to novel nanoscale targeting approaches that may bring new hope to patients. Several nanocarriers have been approved for clinical use. However, to date, there are only a few clinically approved nanocarriers with drugs to selectively bind and target cancer cells. Nanoparticles used as drug delivery carriers consist of different biodegradable materials such as natural or synthetic polymers, lipids, or metals. Nanoparticles are taken up by cells more efficiently than larger micromolecules and therefore, could be used as effective delivery systems. For therapeutic applications, drugs can either be integrated in the matrix of the particle or attached to the particle surface. A drug targeting system should be able to control the fate of a drug entering the biological environment. An effective approach for achieving efficient drug delivery would be to develop nanosystems based on the understanding of their interactions with the biological environment, cellular and intracellular barriers, target cell population and cell-surface receptors, mechanism and site of drug action, drug retention, molecular mechanisms, and pathobiology of the disease under consideration. Reduced efficacy could be due to instability of therapeutic agent, cellular and intracellular transport barriers, toxicity of the carrier, changes in signaling pathways with the progression of disease, or drug degradation. Better understanding of the mechanism of uptake, intracellular trafficking, retention, and protection from degradation inside a cell are required for enhancing the efficacy of the therapeutic agent. Physical approaches to increase efficacy and targeting to specific tissues have been also studies. In the presentation the drug delivery aspects of nanomedicine, the molecular mechanisms underlying the interactions of nanoparticles with cell-surface receptors, biological responses and ultrasound as a triggering and targeting tool and its effect on cellular transport would be discussed.

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P.03

OPTIMIZATION OF Au/TiO₂ THIN FILMS TOWARDS OPTICAL (BIO)SENSING

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Keywords: Thin Films; Au nanoparticles; Localized Surface Plasmon Resonance; Plasma Activation, LSPR-Biosensing

Abstract: Nanomaterials employing Localized Surface Plasmon Resonance (LSPR) [1] are revealing to



Fig. 1. LSPR optimization

be an interesting opportunity for optical (bio)sensing applications. The main reasons are mostly related to their higher sensitivity with label-free detection and to the simplified optical systems that can be implemented, since the incident light couples directly with metallic nanoparticles (NPs) without the use of prisms or gratings. In order to optimize the LSPR absorption band (fig.1), nanocomposite Au/TiO_2 thin films with different gold (Au) amounts, structures and morphologies were prepared. The films were deposited by reactive DC magnetron sputtering and the main parameters changed were the deposition time, Au concentration and the current applied to the titanium target (with Au pellets at its surface). The Au NPs formation was induced by several post-deposition annealing treatments at

different temperatures and investigated via scanning electron microscopy. It was observed that the Au/TiO₂ thin films with 13 at.% of Au, thickness of ABOUT 100 nm and annealed at temperatures above 600 °C had the most well-defined LSPR absorption band. The NPs formation studies revealed an incomplete aggregation at 500 °C and well-defined spheroidal NPs at 700 °C. Plasma activation with Ar led to a gradual blue shift of the LSPR absorption band, which demonstrates the sensitivity of the films to changes in the dielectric function of the environment surrounding the NPs.

Selective references:

1 J. Borges, et al., Broadband Optical Absorption Caused by the Plasmonic Response of Coalesced Au Nanoparticles Embedded in a TiO₂ Matrix, The Journal of Physical Chemistry C, 120 (30), 2016, 16931

Acknowledgements: This work was supported by the Portuguese Foundation for Science and Technology (FCT) in the framework of the Strategic Funding UID/FIS/04650/2013 and FCT Project PTDC/FIS-NAN/1154/2014.

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P.04

IMPROVEMENT OF FATIGUE LIFE AND SURFACE PROPERTIES OF METALLIC MATERIALS OF BIOMEDICAL INTEREST BY LASER SHOCK PROCESSING

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Keywords: Laser Shock Processing, Metal Alloys, Biomedical Interest, Mechanical Properties, Fatigue Life, Surface Properties

Abstract: Laser shock processing (LSP) is increasingly applied as an effective technology for the improvement of metallic materials' mechanical and surface properties in different types of components, mostly as a means of enhancement of their fatigue life behavior. As reported in previous contributions by the authors [1,2], a main effect resulting from the application of the LSP technique consists in the generation of relatively deep compression residual stresses fields into metallic components allowing an improved mechanical behaviour. On their side, bio-mechanical components (i.e. spinal, knee and hip prostheses) are key elements definitely improving the quality of life of human beings traditionally subject to mechanical and functional designs based primarily on



surface residual induced in a hip replacement by LSP [3] intuitive medical approaches, not always optimized from an engineering point of view. Laser Shock Processing (LSP) uses the high peak power of short pulse lasers to generate an intense shock wave into the material finally leading to the generation of a compressive residual stresses field definitely protecting the component against crack initiation and propagation, thus improving its mechanical response and in-service fatigue life. In the present paper, developments in the field of Laser Shock Processing application to several metallic materials of biomedical interest are presented along with results showing the induced surface and mechanical properties modifications interesting in view their high reliability performance.

References:

1. J.L. Ocaña et al.: Induction of Engineered Residual Stresses Fields and Associate Surface Properties Modification by Short Pulse Laser Shock Processing. Materials Science Forum, 638-642 (2010), 2446-2451. 2. J.L. Ocaña et al.: An emerging technique for the enhancement of surface properties and fatigue life of highstrength metal alloys. International Journal of Microstructure and Materials Properties, 8 (2013), 38-52. 3. C. Correa, et al.: Eigenstrain simulation of residual stresses induced by laser shock processing in a Ti6Al4V hip replacement. Materials and Design, 79 (2015), 106–114.

Acknowledgements: Work supported by Spanish MINECO (Grants MAT2012-37782, MAT2015-63974-C4-2-R and MAT2015-63974-C4-1-R).

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P.05

PROPERTIES OPTIMISATION OF TI-BASED BIOMATERIALS BY STRUCTURAL DESIGN

Mariana Calin¹*, Matthias Bönisch¹, Stefan Pilz¹, Supriya Bera¹, Annett Gebert¹, Jürgen Eckert²

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Keywords: titanium alloys, beta-type structure, mechanical properties

Abstract: Currently, titanium (Ti) and its alloys constitute the most favored implant metallic materials in the field of trauma and orthopedic surgery. However, despite the high rates of success of Ti-based implant materials, there still are serious problems of safety and long-term durability in the human body, resulting in repeat of surgical operations. Therefore, much research effort is dedicated to the development of new metastable Ti-based alloys with improved mechanical performance and biological compatibility.

Ti-based alloys offer a wide range of adaptable structures for use in different medical applications, especially in orthopedics. In the focus of our studies are the Ti-Nb-based alloys with various metastable structures (β -type, martensitic, nanostructured, glassy), which are processed by controlled casting and/or severe plastic deformation as bulk material or by powder metallurgy as



porous compacts [1, 2]. In the present paper we will show that tailoring specific by nanostructures (for example with bimodal grain-size distribution or ultrafine/nano-scaled grains with grain non-equilibrium boundaries) can produce unique combinations of properties, such as high strength and good ductility combined with high corrosion and wear resistance

Stress strain curves and microstructures of b Ti-Nb alloys.

Selective references:

M. Boenisch, A. Panigrahi, M. Calin, T. Waitz, M. Zehetbauer, W. Skrotzki, J. Eckert: *Thermal stability and latent heat of Nb-rich martensitic Ti-Nb alloys*, Journal of Alloys and Compounds 697 (2017), 300-309
 M. Calin, A. Helth, J.J. Gutierrez-Moreno, M. Bönisch, V. Brackmann, L. Giebeler, T. Gemming, C.E. Lekka, A. Gebert, R. Schnettler, J. Eckert: *Elastic softening β-type Ti-40Nb alloys by indium (In) additions*. J Mechan Behav Biomed Mater 39, (2014), 162-174.

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P.06

PHOTOACTIVE POLYMERS AND COMPOSITES

M. W. Tausch

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Keywords: photoluminescence, electroluminescence, functional dyes, organic photovoltaic cell

Abstract: Photoactive polymers and composites belong to the favorite materials for designing and assembling "intelligent" devices such as electrochromic and photochromic windows, organic solar cells, organic light emitting diodes and molecular machines. Photoprocesses occurring in these



Fig. 1. Electrochromic polymers [3]

materials are par excellence suitable for communicating basic concepts of chemistry, physics and related material sciences in close combination with convincing applications from science and technology in the 21st century. In order to get a high degree of motivation for students and to make the elementary steps of processes occurring on the molecular level, meaningful experiments and scientifically consistent teaching concepts are needed. The lecture will give an overview on photoactive polymers and composites emphasizing experimental approaches for

teaching purposes. Starting from the fundamental idea, that the electronically excited states of molecules are the "heart" of all photoprocesses, photo-, solvato- and electrochromism as well as photo- and electroluminescence will be compared with to each other. Examples of experiments will be given for demonstrating similarities and differences between these phenomena. During the course of the lecture further materials and experiments for an organic photovoltaic cell using a semiconducting polymer and a fullerene derivative as well as an "intelligent" foil with a molecular switch embedded in a polymer matrix will be shown and used for explaining the functional principles and the challenges for materials in order to improve the performance of such devices.

Selective references:

1. M. W. Tausch, A. Banerji, U. Scherf: "Classroom Experiments and Teaching Materials on OLEDs with Semiconducting Polymers", Education Quimica, 24 (1), p.17 (2013)

2. M. W. Tausch, C. Bohrmann-Linde, J. Ibanez, D.Zavala-Araiza, B. Sotomayor-Martinez Barranco, J. Torres-Perez, C. Camacho-Zuniga: "A Demonstration of Simultanous Electrochemo-luminescence", Journal Of Chemical Education, 90 (4), p.470 (2013)

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P.07

ECONOMIC AND ECO-FRIENDLY SOLAR CELLS

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Keywords : solar cells, TiO₂, Graetzel, CIS, thin layers, nanostructures,

Abstract : With the inevitable crisis of fossil fuels in the next few years (exhaustion of hydrocarbon reserves), everyone must be mobilized to find sustainable solutions to the problem of energy. Green energy (solar, wind) is a credible alternative. The energy produced by the sun is unlimited and gigantic. However, it is unevenly distributed over time and geographically. For these reasons, it is important to develop the processes for both capturing and storing this energy.

The photovoltaic effect, known for almost two centuries, that makes it possible to generate electricity from solar energy, has been greatly studied and developed in recent years. Different materials are used, mainly monocrystalline or polycrystalline silicon. Other technologies are promising and under development, like the perovskites multilayers. In the case of thin-film photovoltaic (CIS, CZTS or Graetzel-type organic dye) it is possible to manufacture eco-friendly and inexpensive solar cells using abundant and cheap materials, through a simple and clean process (unlike the silicon process). It remains to solve the problem of the efficiency of the photovoltaic conversion of these cells for the moment weak (<10%). Many research teams work to improve this technology. This will contribute to generalize the access to energy for million of people and so, to their development.

Our research studies on these thin-film and Graetzel solar cells aim to simplify production processes and to increase efficiency by controlling the synthesis steps, optimizing the steps of deposition of thin layers (semiconductors, active and transparent electrodes) [1], improving the adhesion of layers and the morphology of interfaces, designing nanostructured electrodes [2], improving all the electronic transfers in the photovoltaic cell and finally by creating the model of the photovoltaic system. The realization and development of prototypes (for future industrial production) is one of our objectives and will require an expansion of our collaborations.

The storage of electrical energy is also one of our concerns. We use our skills and experience in the field of TiO₂ nanostructures for the design of anode electrodes for ion-Li batteries [3].

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P.08

TRIBOMECHANICAL RESPONSE OF BARE AND HYDROGENATED AMORPHOUS CARBON COATED METALLIC BIOMATERIALS

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Keywords: metallic biomaterials, hydrogenated amorphous carbon coatings, nanomechanics, nanotribology

Abstract: Wear and corrosion have been identified as two of the main forms of metallic implant failure mechanisms [1-3]. The objective of this work was to improve the protective and tribomechanical characteristics of metallic surfaces used for medical implants. Towards this end, hydrogenated amorphous carbon (a-C:H) nanofilms have been deposited on Stainless Steel (SS), Titanium (Ti) and Niobium (Nb) metal plates using Plasma Enhanced Chemical Vapor Deposition (PE-CVD). The nanomechanical and nanotribological characteristics of the bare and a-C:H coated metallic surfaces have been quantified through nanoindentation and nanoscratch testing, respectively. The density and thickness of the a-C:H films have been probed using X-ray reflectivity (XRR) whereas the morphological characteristics of the metallic surfaces before and after a-C:H deposition have been studied using atomic force microscopy (AFM). Furthermore, the residual imprints through nanoscratch testing have been investigated using scanning electron microscopy (SEM). In summary, it is observed that the a-C:H films improve the tribomechanical properties of the metallic surfaces by reducing their friction coefficient and improving their resistance to plastic deformation and wear.

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P.09

REINFORCEMENT EFFECTIVENESS ON MECHANICAL PERFORMANCES OF COMPOSITES OBTAINED BY POWDER BED FUSION

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Keywords: additive manufacturing, powder bed fusion

Abstract: New material formulations to be used in Additive Manufacturing machines are one of the major interests in this fast growing field. The possibility to tune functional and mechanical properties, by the addition of reinforcements to a polymeric matrix, is hindered by the low provisional capability of the additive manufactured composite. The inherent anisotropy of layer manufacturing combines with mechanisms of filler dispersion and of filler/matrix adhesion in a complex scenario. The paper entails a critical evaluation of mechanical properties measured for several polymeric composites produced by Powder Bed Fusion, in the perspective of provisional models commonly accepted for composite materials. The models are reviewed versus experimental and literature data. The provisional effectiveness is generally good, except for the case of nanometric or surface treated fillers, or of specific anisotropic microstructures obtained by layer manufacturing.



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SECTION I Metallic Materials

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I.K.1

GROWTH KINETIC OF PYRAMIDAL FACES OF ADP AT VERY SMALL SUPERSATURATIONS

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Keywords: growth kinetic, pyramidal faces of ADP

Abstract:

Macroscopic growth kinetic at smaller supersaturations than σ <1% was confronted with the nanometer scale measurements performed by laser interferometry, Vekilov et al, J. Cryst. Growth **119**(1992)248).

A 3 liter crystallization chamber, thermostated in a 10 liter oven was used for growth and kinetic measurements of 3 crystals of $2 \times 2 \times 10$ cm, grown in kinetic regime by reversible rotation.

Apparently, pyramidal faces of ADP show incoherent growth rates dependence at very small supersaturations (σ <1%). In view of the theoretical parabolic dependence of the growth rate at such supersaturation, kinetic data were linearly represented as $\sqrt{R} = f(\sigma)$ and our data were scaled versus the Mullin's data (1967) considered with the growth efficiency of $\varepsilon = 4$. Other linear dependences with efficiencies $\varepsilon = 1$, 2, 4, 8 were drawn on the same graphic. Growth rate of the three crystals, grown in the same conditions are close to each other.

As known the number of dislocations emerging on pyramidal faces of tetragonal type crystals like ADP, substantially decreases with the increasing distance to the seed. During the seed regeneration period, many dislocations are generated by the solution inclusions. Then, the dislocations which have predicted directions in the lattice are slowly deviated towards the prismatic faces during growth.

Than, we have represented both parameters growth efficiency ϵ and the supersaturation σ , versus the order number of successive measured set of data.

A "switching" effect of efficiency ε vs. σ was surprisingly noticed. Every time the supersaturation crosses the line $\sigma \approx 5 \cdot 10^{-3}$, the efficiency "switches" to some other extreme value. In the same time, there is an overall decrease of the growth efficiency along the Z-axis of the crystal, due to the decreasing number of active dislocations remained on the pyramidal faces, due to the deviation towards the prismatic faces. Than, we have estimated the reverse of the growth efficiency like $1/\varepsilon = 1/m + (2/19) \cdot (L/ma) \cdot (\gamma/kT)^{-1} \sigma$. This allowed us to estimate a mean value of the ratio $L/ma \approx 160 - 200$, and $(\gamma/kT) \approx 0.85$ for the edge free energy ratio.

The extreme values of efficiency oscillations $\varepsilon = 8 \leftrightarrow 4$ were realized for $\sigma = (0.34 \leftrightarrow 0.70) \cdot 10^{-2}$, i.e. for the critical nucleus radius $\rho_c / a = 250 \leftrightarrow 120$. This surprising data at the macroscopic scale, are close to the data we have estimated from the literature, measured at nanometric scale, by laser interferometry or by AFM kinetic measurements.







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I.K.2

DESCRIPTION AND ROLE OF γ - γ ' INTERFACES DURING HIGH TEMPERATURE CREEP IN NICKEL BASE SUPERALLOY

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Keywords: creep deformation, high temperature

Abstract:

In operating aeroengines, an important part of the damage experienced by rotating turbine blades is due to creep deformation. At high temperature a microstructural evolution occurs corresponding to a directional coarsening of the strengthening γ' precipitates normally to the <001> tensile direction. This phenomenon, due to stress-aided diffusion, leads to the formation of the so-called γ/γ' rafted microstructure. Due to the relaxation of misfit stresses, a network of interfacial dislocations between γ and γ' rafts is created.

In this work, the creep behaviour at 1050°C of <001> oriented MC2 single crystals is investigated by means of SEM and TEM observations. The γ - γ' rafting process occurs rapidly and appears to be correlated with the early establishment of a pseudo stationary creep stage. In the rafted structure, the γ - γ' interfaces are not perfectly flat. The waviness of the γ - γ' interfaces in the rafted microstructure has been carefully analysed after different creep periods. They undulate around (001) plane. In correlation with this local orientation, the dislocation arrangement and the network stability vary. Regular and stable networks are seen in the (001) interfaces. The role of these networks during high temperature creep is completely analysed.



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I.K.3

METAL BASED CONFORMAL PROFILE FOR PRINTED IMPLANTED ANTENNAS

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Keywords: magnesium alloys, welding, microstructure, tensile properties, fatigue.

Abstract: Due to their excellent mechanical properties, appropriate corrosion resistance and good biocompatibility some metallic alloys are widely employed for biomedical purposes. The high strength and resistance to fracture they can exhibit when appropriately developed, offer reliable performance primarily in the fields of orthopedics and dentistry. Since the late 1960's, Titanium and its alloys have more and more been used for the development of orthopedic implants, especially for fracture fixation and joint replacement. Furthermore, they exhibit a strong osseointegration tendency that is important for such applications [1, 2].

In this paper, a new complex medical device is proposed using Ti-Nb based metallic alloy, acting also as a ground plane for a low profile printed antenna sited on a Polydimethylsiloxane (PDMS) substrate. The first step of the research is oriented on the experimental study of the properties of Ti-Nb based alloy and on the development of the orthopedic device. The second step is focalized on the electromagnetic characterization of the implanted printed antennas. The smart orthopedic device incorporating the antenna is placed inside the body environment and can be used for communication with external base stations or between different nodes of a network to monitor some human vital functions, as the numerically characterized scenario demonstrates it.

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1.0.1

EFFECT OF MECHANICAL ALLOYING ON THE TIB₂ PARTICULATE REINFORCED AI-7 wt.% Si MATRIX COMPOSITES

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Keywords: Mechanical alloying, Aluminum-silicon matrix composites, Titanium diboride.

Abstract: Al-Si based metal matrix composites (MMCs) have been used in many applications of the automotive, aerospace and electronics industries due to their superior mechanical, thermal and physical properties [1,2]. Although they have been generally produced using casting methods, powder metallurgy and mechanical alloying (MA) have been used as alternative fabrication techniques for producing more homogeneous and segregation-free products [3,4]. In the present study, 2 wt.% TiB₂ particles were used as reinforcement materials for the AI-7 wt.% Si alloys to produce MMCs. Also, stearic acid were added into the powder blends as a process control agent (PCA) to inhibit agglomeration. Before MA, powder blends were sealed under Argon (Ar) atmosphere in a glove-box. Then, MA experiments were carried out for 4 h in a Spex 8000 D Mixer/Mill using hardened steel vial/balls with a ball-to-powder weight ratio of 7/1. As-blended and mechanically alloyed (MA'd) powders were compacted under a pressure of 450 MPa by an uniaxial hydraulic press and then they were sintered at 570°C under Ar atmosphere during 2 h. Particle size measurements and phase, microstructural and thermal characterizations of the as-blended and MA'd powders were performed using laser particle size analyzer, X-ray diffractometer (XRD), scanning electron microscope (SEM) and differential scanning calorimeter (DSC). MA changed the morphologies of the powders from irregular to equiaxed shaped particles and provided particle size reduction. Density, Vickers microhardness and wear volume loss measurements of the sintered samples were conducted to show the physical and mechanical properties of the reinforced MMCs. The results showed that the 4 h of MA'd and TiB₂ reinforced Al-7 wt.% Si based composites have 91.76 % relative density, 175.21 ± 25.45 HV microhardness value.

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1.0.2

ABNORMAL BEHAVIOR OF LATTICE SPACING OF THE ORTHORHOMBIC MARTENSITE IN VT23 TITANIUM ALLOY

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Keywords: titanium alloys, orthorhombic martensite, coefficient of thermal expansion

Abstract: The material under investigation was VT23 titanium alloy. Samples were quenched from different temperatures between critical and β -transus and investigated during heating using *in situ* XRD. The characteristics of the anisotropy of coefficient of thermal expansion along different directions of orthorhombic martensite were estimated. Abnormal behavior of «b» spacing was found. It showed a negative value of the coefficient of thermal expansion.

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1.0.3

EFFECT OF POWDER PACKING ON THE PRODUCTION OF Fe BASED SHAPE MEMORY ALLOYS VIA POWDER METALLURGY APPROACH

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Keywords : Powder Metallurgy, Fe based SMAs, Fe-Mn-Si

Abstract : As an innovative solution for many different technical applications which require functional materials, engineers have been increasingly taking advantage of shape memory alloys (SMAs). Fe-based shape memory alloys are one of the classes showing shape memory effect (SME) for the specific compositions. Powder metallurgy approach has been applied to many SMA compositions including some Fe based systems for the improvement of microstructural features and has a potential to explore new compositions. In this study compressibility of Fe based powders was studied targeting different SMA compositions in Fe-Mn-Si system. For this purpose compressibility of individual Fe, Mn and Si powders and their binary and ternary mixtures were studied against compaction pressure. Effect of powder particle size and amount of binder were simultaneously studied to optimize densities at green state.

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1.0.4

PRODUCTION OF OPEN CELL ALUMINUM FOAM BY VACUUM CASTING METHOD

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Keywords: Al foams, Vacuum casting, Space holder technique, Density-strength relation

Abstract: The properties like low density, hardness and impact resistance have been found attractive by the researchers and the studies in this field have been started. There are various methods for production of metal foams were presented and discussed [1]. The casting of metals and alloys



Fig. 1. Aluminum foam

around space holder material is very economical way to obtain metallic foams or cellular parts [2]. In this study, vacuum casting setup was designed. NaCl as a space holder material and dissolution technique was used for foaming of the A360 aluminum metal. By changing the NaCl particle size change in the foam properties was investigated. Densitystrength relations were determined depending on the NaCl particle size. Compression test was used to evaluate the foam behavior under static loading. Optimum vacuum conditions for the successful foam production by infiltrating

the liquid melt between the NaCl particles were determined. It was concluded that vacuum casting can be used to produce open cell aluminum foam by integrating of NaCl dissolution process.

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1.0.5

MICROSTRUCTURE AND PROPERTIES OF AlCrFeNiMn/Gr HIGH ENTROPY COMPOSITE PROCESSED BY MECHANICAL ALLOYING AND SPARK PLASMA SINTERING

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Keywords: material high entropy composite, mechanical alloying, spark plasma sintering, microstructure, properties.

Abstract: Most of the high entropy alloys consist only metallic elements [1,2] and few are related with introducing of nonmetallic elements [3]. In the present paper will be described a new composite material obtained by mechanical alloying (MA) and spark plasma sintering (SPS) processes. Thus, AlCrFeNiMn/Gr composite was successfully synthesized in stainless steel vials. Alloying behavior, microstructure, mechanical properties and detailed phases of the composite were investigated systematically. The phase constituents and morphology of the alloying powders were characterized by X-ray diffractometer and scanning electron microscopy. During MA, the formation of a supersaturated solid solution with both body-centered cubic (BCC) and face-center cubic (FCC) structure occurred. The as-milled composite was consolidated by SPS at 900°C. Same phases were put in evidence together with carbides, and no grain growth occurred and no intermetallic phases presence in the AlCrFeNiMn/Gr composite. The compressive strength and Vickers microhardness of AlCrFeNiMn/Gr composite were around 2200 MPa and respectively 700 ± 25 HV.

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1.0.6

HOT WORKING EFFECTS ON THE DAMPING BEHAVIOR OF SHAPE MEMORY ALLOYS

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Keywords : Cu-Zn-Al shape memory alloys; dynamic mechanical analysis; martensite; morphology

Abstract : Cu-Zn-Al shape memory alloys (SMAs) were analysed in two different processing states: (i) hot-forged and (ii) hot-rolled. Both hot-forged and hot-rolled specimens were cut into lamellar configuration, before being homogenized (1073 K/ 18 ks/ water) and tempered (373, 473, 573, 673 K/ 300 s/ water). From each of the five differently treated lamellas, in hot-forged and hot-rolled states, rectangular specimens were cut for dynamic mechanical analysis (DMA). The remaining segments were sectioned into metallographic specimens. The metallographic specimens were embedded into could mounting resin, ground, polished and etched for scanning electron microscopy (SEM) -observations. DMA results revealed the influence of plastic deformation procedure and heat treatment temperature on the reversible martensitic transformation.

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1.0.7

INFLUENCE OF THE ELEMENT ADDITIONS TO THE MICROSTRUCTURAL AND MECHANICAL CHARACTERISTICS OF SELECTED HIGH ENTROPY ALLOYS

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Keywords : materials science, high entropy alloys, characterization, alloy development

Abstract : High entropy alloys are composed of five or more elements in equal or near equal proportion and due to their superior mechanical and corrosion properties are considered serious candidates for the replacement of conventional high grade steels and superalloys [1]. Present paper discusses the effect of elemental additions (Al, Mn, Cu, Ti and Si) on the microstructure and mechanical properties of high entropy alloys. Therefore, Al, Ti and Si were added to tailor the BCC structure content and improve oxidation resistance, Co was replaced with the less critical and less expensive Mn and Cu was added to induce toughness and oxidation resistance. The resulted alloys are based on Al-Cr-Cu-Fe-Mn-Ni, Al-Cr-Co-Fe-Ni-Ti, Al-Cr-Fe-Mn-Ni and Al-Cr-Cu-FeMn-Ni-Si systems. Ingots of high entropy alloys were prepared by induction melting and were casted in a cold copper mold. As-cast samples were characterized by optical microscopy, scanning electron microscopy, X-ray diffraction and mechanical analyses. Ti and Si additions determined a significant hardness increase due to the higher intermetallic compound content. Cu had a deleterious effect on mechanical properties due to significant intergranular segregations. Mn had successfully replaced Co, improving structural stability. Al had a remarkable effect on increasing alloy mechanical properties. Al-Cr-Fe-Mn-Ni type alloys showed best overall characteristics.

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1.0.8

OPTIMAL CHOICE OF MULTI-PASS TURNING PARAMETERS IN METAL CUTTING

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Keywords: metal cutting, multi-pass turning, dynamic programming, Particle swarm optimization

Abstract: Metal cutting represents a large portion in the manufacturing industries. A considerable cost can also be achieved with optimized cutting conditions taking into account the limits of performance of the machine, of the cutting tool and the metal machined. Effective optimization of



Fig. 1. Principle of dynamic programming

the machining process parameters affects the cost and time of production of machined parts, and the quality of the final products. An analysis of multi-pass machining operations has been made using a variety of optimization criteria [1, 2, 3 and 4]. Agapiou [4] has developed a multi-pass optimization methodology for turning, based on the technique of dynamic programming and cutting conditions are optimized with the simplex method of Nelder-Mead.

This paper describes the development and use of an optimization system which determines the optimum machining

parameters for turning operations. The objective considered is the minimization of the production time, take into account various constraints. The number of passes and the depth of cut for each pass are determined using dynamic programming process and the optimum cutting speed and feed rate for each pass are determined using a particle swarm algorithm. The results show that the strategy led to a decrease in the cost of production of about 6% compared to that of Agapiou [4] which proves their efficiency.

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1.0.9

EXPERIMENTAL INVESTIGATIONS TO EVALUATE THE EFFECTS OF CUTTING PARAMETERS ON CUTTING TEMPERATURE AND RESIDUAL STRESSES DURING MILLING PROCESS OF THE AISI 1045

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Keywords: Milling, Cutting parameters, Cutting temperature, Residual stresses, Surface response

Abstract: Today major metal cutting companies in industrial countries, looking to gain time and reduce manufacturing costs while respecting the environment. There are many phenomena which affect the quality and production costs of the product, including cutting efforts, cutting temperature,



residual stresses, etc. A better understanding of these phenomena will reduce the product failure and maximize their lifetime.

The main goal of this study is to examine the effect of machining conditions (cutting speed, feed rate and depth of cut) on cutting temperature and residual stresses, during the milling operations using the response surface method. A good agreement between the predicted and measured cutting temperature was found, the cutting temperature and the residual stresses increase with increases in cutting speed and depth of cut.

However, little influence has been registered in the case of an increase of the feed rate. The percentage of error is 4.81%, indicating that the numerical approach can accurately predict the cutting temperature of the AISI 1045.

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I.O.10

THE INFLUENCE OF INTERCRITICAL QUENCHING IN ULTRASONIC FIELD ON THE STRUCTURE OF SOME DUAL-PHASE STEELS WITH LOW CONTENT OF CARBON AND MANGANESE

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Keywords: dual-phase steel, intercritical quenching, ultrasonic field

Abstract: In the past few years researches has been done to obtain dual-phase steels with a low content of manganese (less than 1%) [1, 2] and for that reason such alloys were chosen for analysis; two types of commercial steels were used ("Steel A" and "Steel B"), materials produced and used primarily for welding electrodes. These alloys contain 0.087% C - 0.511% Mn ("Steel A") and 0.101% C - 0.529% Mn ("Steel B") and have the following critical points in solid-state phase transformation: Ac1 = 724.90 °C, Ac₃ = 901.10 °C ("Steel A") and Ac₁ = 725.70 °C, Ac₃ = 900.00 °C ("Steel B"). The chemical compositions and the critical points were determined with a FOUNDRY-MASTER Xpert Spectrophotometer and a DIL 402 Expedis-SUPREME Dilatometer. The ferrite-martensite structures, typical of the dual-phase steels, has been obtained by intercritical quenching that consisted of heating at temperatures ranging between 760 and 820 °C and cooling in water with a temperature of 20 °C (a Ultrasound Bath LBS 2). For determining the influence of ultrasound on the structure of dualphase steels, the cooling was made in three variants: water (without mechanical agitation), water in an ultrasonic field with f = 40 kHz and water in an ultrasonic field with f = 59 kHz. After carrying out intercritical quenching, the samples have been subjected to metallographic analysis (with LEXT OLS4100 Olympus Laser Microscope) through which the volume fraction of martensite, the morphology and distribution of this phase, have been determined; in addition, with a DuraScan 70 Micro Hardness Tester was determined the microhardness of the ferrite. The usage of ultrasound at cooling and the increase their frequency has led to an increase in the volume fraction of martensite in the dual-phase structures and to increase of the microhardness of the ferrite.

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I.O.11

EFFECT OF STRAIN HARDENING ON PRECIPITATION KINETICS IN ATI 718PLUS

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Keywords: n phase, precipitation kinetics, strain hardening, ATI 718Plus

Abstract: ATI 718Plus components are manufactured by forging a wrought billet in stages to obtain the desired geometry and microstructure. Parts are then heat treated to optimised proportions of γ'

and n phases. n phase is a plate-like phase that precipitates on the grain boundaries of ATI 718Plus, similar to δ phase in Inconel 718. This work is motivated by the knowledge that precipitation of δ phase on the grain boundary in Inconel 718 can, in small amounts, reduce high temperature notch sensitivity [1], resist intergranular crack propagation and improve grain size control during hot working operations [2]. Although excessive amounts of δ phase are detrimental to the alloys' mechanical properties [3]. However, the complete kinetic behaviour of η phase precipitation and dissolution during forging and heat treatment is still not fully understood. This paper investigates the effects of temperature and strain hardening on n phase precipitation kinetics and size in ATI 718Plus. This is achieved through the use of isothermal hot



Figure 1: Microstructure of ATI 718Plus with n precipitates

compression tests and heat treatment at various temperatures. Strain hardening was found to affect the η precipitation kinetics considerably. The results reported are a contribution to a fuller understanding of this important process.

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I.O.12

EFFECT OF PRE-STRAINING AND BAKING TEMPERATURE ON BAKE HARDENING BEHAVIOUR OF HOT-DIP GALVANIZED STEELS

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Keywords: Bake hardening, hot-dip galvanizing, pre-straining, mechanical properties.

Abstract: Cold rolled low carbon steels are used especially in the automotive industries such as car bodies, car panels, fenders and door panels [1]. These steels belong to a class of forming steels and provide good formability, but low strength. As known, good formability and high strength are generally contrary to each other. This obstacle can be overcome by using bake hardening (BH) technique [2,3]. On the other hand, corrosion is a major problem of the steels used in the car industry. Corrosion resistance of these steels can be improved by hot-dip galvanizing process [4]. In this study, bake hardening (BH) process was applied to hot-dip galvanized DX51D, DX52D and DX54D steels. After BH treatment, bake hardened samples were investigated regarding effect of prestraining and baking temperature on mechanical properties. The samples were pre-strained to 2, 5 and 8 % followed by baking at 170 °C and 210 °C for 20 min. The best bake hardenability of 90 MPa, and final yield strength (FYS) of 170 MPa were obtained in the DX52D specimen pre-strained 8 % and then baked at 170 °C for 20 min.

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I.O.13

ESTIMATING VOLUME FRACTIONS OF MICROSTRUCTURAL CONSTITUENTS IN AUSTEMPERED HIGH SILICON ALLOYED GJS 600-10 DUCTILE IRON THROUGH MAGNETIC MEASUREMENTS (VSM)

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Keywords: Austempering, high silicon ductile iron, retained austenite, X-ray diffraction, magnetization.

Abstract: Austempered ductile irons (ADI) are used in many industries including mining & construction, agriculture, automotive, and heavy truck due to their economic advantages and high reliability [1]. High strength and high toughness can be achieved through unique ausferritic microstructure obtained after austempering. Ausferritic microstructure consists of acicular ferrite (α) and high carbon retained austenite (γ_{hc}), and retained austenite (RA) content plays an important role in determining mechanical properties such as fracture toughness and fatigue resistance [2,3]. Therefore, it is crucial to estimate the amount of retained austenite. In this work, austempering heat treatment was applied to high silicon alloyed GJS 600-10 grade ductile iron. All samples were initially austenitized at 975°C for 2 h in an electrical furnace followed by austempering in a salt bath at 270, 330, and 390°C for 60 min. Volume fractions of the phases were estimated by using XRD and VSM method. VSM measurements indicated that the typical hysteresis loops of samples varied depending on the volume fractions of ferromagnetic (ferrite) and paramagnetic (austenite) phases. The measured volume fraction of ferrite (X_{α}) via XRD measurements against the magnetization saturation (m_s) was plotted for the austempered samples, and the data points were adjusted by a linear fitting to calculate the intrinsic saturation magnetization of ferrite. The intrinsic magnetization saturation value obtained from the plot was compared with the magnetization value of as-cast (100 % ferritic) sample, it was seen that the values were very compatible with each other.

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I.O.14

OXIDE NANOMATERIALS: SYNTHESIS, MECHANISTIC STUDIES AND TECHNOLOGICAL INNOVATION IN WASTEWATER TREATMENT BY FLOTATION

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Keywords: Oxide nanomaterials, wastewater treatment, flotation

Abstract: Nanoparticles may act as surfactant molecules during wastewater treatment by flotation, being incorporated into surfactant-stabililised foams for several years [1,2]. In the scientific literature there are some studies which presented the ability of nanoparticles to act as foams/emulsion stabilisers [3,4]. The formation and the stability of foams being dependent of the particles size, surfactant type and concentration [5,6]

In order to investigate the application of nanomaterials as froth stabiliser in flotation, our experimental study was based on testing the oxide nanomaterials for the removal of animal fats from food industrie wastewater.

 Fe_2O_3 and Al_2O_3 nanomaterials were prepared by coprecipitation and, respectively, sol-gel method. Their structural and morphological characterisation was done by XRD, SEM and TEM analyses. In order to evaluate their potential of application in fats removal from wastewater by flotation were evaluated various parameters such as: repartition coefficient, purification factor and separation efficiency. Moreover, the fotation efficiency was investigated by measuring the maximum froth height at equilibrium.

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I.O.15

FORMATION OF AI-TI BASED PHASES FOR GRAIN REFINEMENT OF AI ALLOYS PRODUCED BY HIGH ENERGY BALL MILLING

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Keywords: Aluminium, Grain refinement, Al-Ti-B, Scanning electron microscopy, X-ray diffraction

Abstract: In this experimental study, Al-Ti-B and Al-TiB₂ powders were ground in planetary ball mill to produce $Al_xTi_yB_z$ where x, y and z stands for the relative molar ratios of elements for the compilation of grain refiner compound [1]. Powder size distribution, phase formation (XRD) and particle morphology was investigated by means of SEM and XRD. The fabrication of AlTiB phases and the grain refinement of Al alloys by adding this phases were aimed. For this purpose, the powders were fed to planetary ball mill to be milled at a speed of 600 rpm. The powders were also milled at different milling times as 30 min and 150 min either in metallic form or compound form of Al, Ti and B powders. The powders were dried after each milling to be characterized by SEM and XRD. The phases and morphology-elemental analysis were also conducted by XRD and SEM, respectively. Moreover, the powders were added to Al alloy castings avoiding the breaking through alloy series which are mainly used in aluminum industry [2, 3]. The Al alloy series were examined for grain refinement by Brinell hardness and optical microscopy for mechanical properties and grain formation as well as by SEM (EDS) for grain formation, morphology and elemental distribution analysis.

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I.O.16

IDENTIFICATION OF THE CREEP MODELS PARAMETERS OD THE METALIC MATERIALS USING EVOLUTIONARY ALGORITHMS

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Keywords: Metallic creep, Evolutionary Algorithms, Small Punch

Abstract: Creep deformation and rupture are important in the determination of limiting design factors such as strain histories, damage field evolution and lifetimes.[1,2]. Therefore, creep modeling has gained considerable importance in recent years in view of the growing needs to develop high temperature metallic materials for modern super critical and ultra super critical power plants.

Every constitutive equation has its own method for parameter identification. In conventional approaches, the model of interest is first approximated and its parameters are identified sequentially through the curve fitting approach. However, the determination of its process is problem dependent, and thus may not be easy if the model is highly nonlinear. Then a systematic and objective computer based procedure for parameter identification is necessary. Therefore, In this paper we, propose to use the evolutionary algorithms such as ant colony algorithm for identifying the parameter set to the different creep models. The advantage of the proposed approach is that parameter can be identified without any divergence in every case. This algorithm has clearly demonstrated its capability to yield good approximate solution even in the case of complicated multimodal, discontinuous, non-differentiable, and even noisy or moving response surface optimization problems, and has been successfully implemented in areas of structural design, control, etc.

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I.O.17

ANALYTICAL DESCRIPTION OF THE BAINITE TRANSFORMATION KINETICS IN STEELS 300M AND D6AC

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Keywords: bainite, kinetics, Kolmogorov-Johnson-Mehl-Avrami equation, 300M, D6AC

Abstract: The most widely used equation for analytical description of the transformation kinetics of the metastable solid solutions (the steel austenite in particular) is Kolmogorov-Johnson-Mehl-Avrami (KJMA) equation [1]. However the practical analysis of the experimental isothermal bainite transformation kinetics often gives significant deviation from the conventional theory [2]. This problem can be solved by the derivation of an analytical function which would provide the best fit of the experimental results.

Two analytical equations describing the kinetics of bainite transformation in steels 300M and D6AC are proposed. The first one is based on an approximation of the experimental ln (-ln (1-P)) vs. In τ dependence by a second order polynomial function. The second function is based on the solution of the differential equation y(x) = ay'(x)+b, where x= ln τ , y(x) = ln(-ln(1-p)).

A comparison between the proposed equations and Kolmogorov - Johnson - Mehl – Avrami is conducted. The adequacy of the two analytical models is estimated using Fisher test.

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I.O.18

CONCEPTS REGARDING THE HEAT TRANSFER AND REDUCTION OF THE ENERGY CONSUMPTIONS AT THE METALLURGICAL HEATING FURNACES

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Keywords: heat transfer, technology, coefficients, productivity, energy consumptions.

Abstract: As a major consumer of energy the steel producers are always mentioned as interesting field of investigations. The article is focused on the study of the heating technology principles that occurs in the plastic deformation of the steels, in order to correlate them with some new global tendencies. The most frequent, but also one of the most important concerns is the reducing of the energy consumptions, on purpose to decrease the expenses and increase the productivity. Thereby, it is necessary to know the thermal parameters and the present heating technology, to have a better understanding of the present situation, by calculating an energetic balance. These parameters are very helpful in creating a new mathematical model, which can avoid the risk of the thermal stresses advent. There can be observed the authors contribution regarding the values of the parameters of the heat transfer.

Having in view the results of the experiment, there can be concluded that it can be an important solution in the context of the energy consumptions, by increasing the heating efficiency of the entire process. The energy saving represents an advantageous solution for the entire process, by reducing costs of the final product.

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I.PO.1

DETERMINATION OF THE HEAT DENSITY AT THE INTERFACE PIECE TOOL FOR TURNING OPERATIONS

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Keywords: Cutting Temperature, Modeling, measurement, thermocouple, turning.

Abstract: In machining operation, the quality of surface finish is an important requirement for many turned work pieces. Cutting temperature is one of the most important parameters in determining the cutting performance and tool life. The objective of this work is to estimate the cutting



Fig. 1. Insert mesh and temperature distributions at cutting time equal 60

temperature in 3D model on tool-chip interface and the interface temperature during turning process, using the digital simulation software COMSOL Multiphysics. The tool-chip interface temperature results obtained from experimental results by using, AISI 1060 steel work piece with natural contact tools, without the application of cooling and lubricating agents and a K type thermocouple technique was used for estimating cutting temperatures in a turning operation. This procedure facilitates the determination of the temperature at tool-chip interface in dry

turning process, which is still a challenge for existing experimental and numerical methods. The numerical model percentages of error are 3.57%, indicating that the numerical approach can accurately predict the cutting temperature of the AISI 1060 with an coated carbide inserts.

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I.PO.2

THERMAL PROCESSING OF A TITANIUM ALLOY WITH AERONAUTICAL APPLICATIONS

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Keywords: titanium, treatments, properties, biphasic structure

Abstract: The titanium alloys are used in aeronautical applications (up to 75% of titanium alloys), reinforcements, biomedical applications, army industry, because of a high traction resistance, low weight, excellent corrosion resistance and capacity to resist at extreme temperatures. However, some titanium applications are restricted by the small hardness, high friction coefficient and low usage resistance.

The paper shows the experimental researches made on Ti-6Al-4V alloy, subjected to quenching and tempering heat treatments. The hardening heat treatment of titanium alloys with α + β structure consists in stabilization phenomenon of β solid solution dissolved in α' and α'' metastable phase. After quenching and tempering heat treatments, are obtained good mechanical properties in biphasic alloys, maintaining the plasticity characteristics in reasonable limits.

The efficiency evaluation of heat treatments was realized through micro-hardness measurement and structure analyses on electronic microscope.

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I.PO.3

TRIBOLOGICAL BEHAVIOR OF DISTALOY DC COMPONENTS

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Keywords: Distaloy DC, SiC, sintered steel, nitriding, wear

Abstract: Sintered material base on pre-alloyed powders (Fe-Ni-Mo) are expansively applied in the automotive industry and Distaloy is today the most widely used raw material worldwide for the



Fig.1. Results of wear resistance for the Distaloy DC

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production of complex, precise, high strength PM machine parts [1-3]. In this study a diffusion bonded powder type Distaloy DC (Fe-2 wt. % Ni-1.5 wt. % Mo, Höganäs-Sweden) was used. It was alloyed by 0.5 and 1 wt. % silicon carbide with the addition of 0.6 wt. % lubricant in the form of introlube. All the powder mixtures were compacted at 600 MPa, and then sintered for 30 min. in argon atmosphere at 1120° C. After the completed sintering process, the sintered alloy samples were plasma nitrided at 520°C for 2h in the 20N₂-80H₂ atmosphere. The effect of nitriding on the wear and friction behavior of Distaloy DC with different compositions was investigated.



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I.PO.4

FRACTOGRAPHIC ANALYSIS OF A SUPER DUPLEX STAINLESS STEEL AFTER HOT DEFORMATION

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Keywords: super duplex stainless steel, hot deformation, fractography

Abstract: With a good combination of extreme high corrosion resistance and strength, super duplex stainless steels (SDSS) are successfully used in the oil and gas industry. Due to the high ratio of property to cost, these steels are a good alternative to other higher performance materials such as super austenitic stainless steels and Ni-based alloys. However, sometimes the duplex microstructure of SDSS may cause some embrittlement in certain inadequate conditions of thermo-mechanical treatment, which can induce premature failure. In the present paper, the fractographic analysis of a F55 super duplex stainless steel was performed, in an attempt to determine the causes of cracking during industrial hot deformation by forging. The morphology of fracture surfaces was studied by SEM analysis, in order to determine the crystallographic character of fracture under different conditions of hot deformation. The experimental results have allowed to draw some conclusions regarding the fracture mechanism of the F55 super duplex stainless steel.

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I.PO.5

INFLUENCE OF DYNAMIC THREE POINT BENDING ON THE WORK HARDENING CAPACITY OF MANGANESE STEELS

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Keywords: manganese steel, DMA, internal friction, storage modulus, work hardening

Abstract Hadfield steel, with approx. 1.2 C and 12 Mn (mass. %) was invented by Sir Robert Hadfield in 1882, being destined to the manufacturing of tramway wheels but, owing to his exceptional hardness, its destination shortly switched to railway passages and digging equipment. Later, during World War I, it became the main component of armors and shields for warfare protection [1]. This unique austenitic steel, which combines high tensile stress and ductility with elevated work hardening capacity and excellent abrasive wear resistance [2] has an equilibrium structure comprising manganese-alloyed austenite and alloyed cementite, (FeMn)₃C. When subjected to repeated compression stresses, localized on contact surface, it work hardens due to the formation of ε-hexagonal close packed martensite, containing mosaic blocks, which contributes to the augmentation of both hardness and abrasive wear resistance [3]. The present paper compares two states of T105Mn120 manganese steels specimens: untreated (as cast) and solution treated to 1100°C. Paralellipipedic specimens were wire-cut by spark erosion and subjected to Dynamic Mechanical Analyser (DMA) tests performed with a three-point-bending specimen holder. DMA tests were done by two variants: (i) temperature scans, between -150 and 400° C and (ii) isothermal strain sweeps, up to 0.15 % strain amplitudes. The formed emphasized the critical temperatures of thermally induced reversible martensitic transformation while the latter enabled to monitor storage modulus increase due to the work hardening caused by dynamic bending. By optical and scanning electron microscopy, the structural changes induced by dynamic bending were discussed.

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I.PO.6

OXIDATION OF FeNiCrMnAl HIGH ENTROPY ALLOY - WEAR RESISTANT ALLOY. CHEMICAL AND STRUCTURAL CHARACTERISATION.

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Keywords: oxidation, FeNiCrMnAl high entropy alloy, characterisation, microstructure;

Abstract: With the increase of temperature and with with degradation of metallic materials the reducing of mechanical properties occurs. The oxidation process leads to the formation of oxides, which may be harmful if the oxide is formed at high speed.

If the layer is made adherent thin and has slow development the formed oxide have a protective role on the metal or alloy base. This paper presents the FeNiCrMnAl high entropy alloy oxidized at 900 °C for 3 hours respectively 6 hours. It also presents the microstructures and mechanical properties of FeNiCrMnAl high entropy alloy. Investigation on high entropy alloys have show that on their surface is formed a dense and protecting oxide film. Oxides formed improve the mechanical properties of the FeNiCrMnAl high entropy alloy investigated.



Figure 1. Oxidation of FeNiCrMnAl high entropy alloy at 900°C temeperature, for 3 hours.

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I.PO.7

SEQUENTIAL ELECTRODEPOSITION OF MULTILAYERED Sb-Te/Zn-Sn THIN FILMS FOR THERMOELECTRIC APPLICATIONS

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Keywords: thermoelectric materials, thin films, electrodeposition, sequential layers, annealing

Abstract: Thermoelectric materials and devices which can directly convert heat into electricity have numerous applications for solid-state power generation and cooling.

The sequential electrodeposition of Sb-Te / Zn-Sn / Sb-Te / multilayered thin films and the annealing of the obtained multilayers were investigated. The number of layers was varied from 2 to 5. Electrodeposition of each individual layer was carried out in aqueous acidic baths (H_2SO_4), with the addition of metallic salts. The influences of the deposition potential, electrolyte concentration and temperature on film stoichiometry were studied. The compositions, morphologies, structures, Seebeck coefficients and resistivities of the deposited thin films were characterized and compared by LA-ICP, SEM-EDX, XRD, Seebeck coefficient measurement system and four-probe resistivity measuring device respectively. The chemical composition as well as the morphology of individual layers were strongly dependent on the deposition conditions: 30-50 wt.% Sb, 50-70% Te and respectively 50-80% Zn, 20-50%Sn. Annealing of the layers at 200-250⁰C in a protective Ar atmosphere for 3 hours revealed the formation of thermoelectric compounds such as Sb₂Te₃ and Zn4Sb3.

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I.PO.8

INFLUENCE OF THE HEAT TREATMENTS WITH CONCENTRATED ENERGY SOURCES ON THE ALLOY STEELS MECHANICAL PROPERTIES

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Keywords: alloy steels, concentrated energy sources, mechanical properties

Abstract: The results presented in this paper are part of a larger project that includes the study of the heat treatment influence with concentrated sources on the microstructure and mechanical properties, when this are applied to the alloyed steels X210Cr10 and HS 18-0-1. The numerous tests





made, show that indifferent of the studied properties: microstructure, hardness and wear resistance, the heat treatments application with concentrated sources, lead to higher values of the studied properties in comparison with those obtained with the conventional treatments. For some properties, the values can increase up to 245% when are the heat treatment with applied concentrated sources, versus the classical heat treatment. The difference between the properties values obtained by heat treatment with laser and cathode beam not are very high, especially that for some properties the laser heat treatment provides better values but for other

properties, those with cathode beam (see figure 1). The study shows that depending on the material, tools destination and of the property whose value is desired to be maximized, will be applied the adequate heat treatment with concentrated sources, more exactly with laser or cathode beam.

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I.PO.9

NEW FeMnSi+Al ALLOY PROPOSED FOR HIGH DAMPING CAPACITY

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Keywords: damping, SMA, SEM, EDS

Abstract: High damping capacity materials present an increased interest in many applications were vibration and noise reduction is absolutely necessary. Metallic materials with a high internal friction (IF) are becoming valuable because of them usual mechanical properties that fulfill the damping capacity in applications. Some of the shape memory alloys [2] present a huge damping capacity during the solid state transformation ($M \leftrightarrow A$) based on the re-orientation and accommodation of the material structure.

Iron based shape memory alloy present the best advantages for industrial application as dumpers in different areas. Beside civil construction domain these materials can cover also applications in automotive industry as shock impact absorbers for low velocities as protection for engine parts and also for noise reduction. By these means in this article we analyze FeMnSi+Al alloy with a new chemical composition obtained through classical melting method in Ar controlled atmosphere. The experimental samples surface was analyzed using SEM (scanning electron microscopy with VegaTescan LMH II, SE detector) equipment and EDAX detector (X-ray energy dispersive spectroscopy Bruker type) for structural and chemical analyses. XRD determinations on the surface were done in order to establish the main alloy compounds.

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I.PO.10

COLD END CORROSION AVOIDING BY USING A NEW TYPE OF AIR COMBUSTION PRE-HEATER

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Keywords: cold end corrosion, pre-heaters, flue gases, combustion air.

Abstract: Cold end corrosion appears due to catalytic oxidation of the sulfur dioxide to sulfur trioxide and then due to the sulfuric acid condensation at dew point. Calculating dew points of various acid gases and options for reducing cold end corrosion of heat recovery exchangers are presented [1,2]. For avoiding the cold end corrosion we design a new type of air combustion pre-heater for boilers and furnaces. In order to determine the different construction model of air combustion pre-heater, we use a mathematical model, transcribed in C₊₊, for boilers and furnaces heat exchanger calculation. Also, the tube skin temperature of the first row of pipes of the actual air pre-heater was simulated with this computer program, in order to determine whether this temperature is lower than acid dew point of flue gas. The values of the combustion air temperature and of the flue gas temperature, at the entrance and at the exit of the actual air pre-heater [3,4,5]. With the simulation for this configuration of the actual combustion air pre-heater, the skin temperature for the first row (for the combustion air flow) of tubes from the upper bundle was TS = 134 ^oC.

A way to reduce the cold end corrosion in the combustion air pre-heaters is raising the temperature of the combustion air at the air pre-heater entrance. This solution involves taking a quantity of preheated air, recirculation and then reintroducing it in the air pre-heater [5]. The combustion air temperature at entrance in pre-heater, after mixing cold air (temperature 45° C) with hot air (temperature 240° C), is 105° C.

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I.PO.11

TITANIUM INFLUENCE ON THE MICROSTRUCTURE OF FeCrAI ALLOYS

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Keywords: FeCrAl alloys, titanium, chemical composition, microhardness, microstructure

Abstract: 4R generation nuclear power plants should work with metallic material of the highest quality, capable of withstanding in maximum safe conditions for 15-20 years. The FeCrAl alloys are capable of such performance, because of the resistance to: oxidation at high temperatures, corrosion, erosion and penetrating radiations in liquid metal environments. In addition, such materials are capable of forming on their surface some oxide layers, textured and self-renewable, with high adhesion to metal base. These properties may be improved by microalloying with metal elements such as Ti, Zr, Y, Hf in an amount of 1 to 3%. These metals with high affinity to oxygen are capable of stabilizing the formed structure of alumina and increase adhesion to the metallic substrate. The FeCrAl alloys microalloyed with titanium were obtained in a VAR (Vacuum Arc Remelting) equipment under argon atmosphere (99.99% purity). Were obtained three microalloyed alloys with 0.5%, 1% and 1.5% titanium, while maintaining the same Fe-14Cr-5Al metal matrix. EDAX analysis has been performed to determine the chemical composition of the oxide layer and of the bulk of sample. SEM analysis has been done to determine the microstructural features. The results have shown the capacity of FeCrAl alloy to form oxide layers, with different texture and rich in elements such as AI and Ti. Compositional analysis performed on FeCrAI samples microalloyed with 0.5, 1.0 and 1.5% Ti in the central area shows a relatively similar composition compared with the technological calculations made. Ti distribution reflects the homogeneity of the alloy. Analysis of the $HV_{0.2}$ hardness on the samples attests values in the domain of 163-183, values that fall within the normal range of these materials, which is exclusively due to the influence of alloying elements in the metal alloy composition and also due to the homogeneous arrangement of constituents in the metal matrix.

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I.PO.12

VIRTUAL TESTING OF THE COMPOSITE STRUCTURES MADE FROM HIGH ENTROPY ALLOYS AND STEEL

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Keywords: High entropy alloys, composite structures, dynamic loads, simulation

Abstract. High entropy alloys (HEA) are metallic materials obtained by a mixture of at least five atomicscale chemical elements. They are characterized by high mechanical strength, good thermal stability and hardenability. AlCrFeCoNi alloys presents high compressive strength, high values of yield and tensile strength of 2004 MPa, respectively 1250 MPa and elongation of about 32.7%. These materials can be used to create composite structures HEA-steel type which resists at dynamic deformation during high speeds impact (projectiles, explosion). The paper presents four different composite structures made from a plates combination of HEA and carbon steel, using different joining processes such as: explosion welding, brazing, contour welding and soldering. Numerical simulation of impact behavior of the composite structures was performed by virtual methods, taking into account the mechanical properties of both materials. For analyzing of each constructive variants three virtual shootings were designed, using an incendiary perforating bullet (caliber of 7.62x39 mm) and different impact speeds. The best ballistic behaviour was provided by the composite structures realized by welding and brazing, that have a good continuity and rigidity. The other composite structures, that haven't a good surfaces adhesion (unbonded or obtained by soldering using organic adhesives) presents high fragmentation risk, because the rear plate can fragment on the axis of shooting due to the combination of the shock waves with the reflected ones. The placing order of the materials into composite structure has a very important role for decreasing the impact energy. The tougher plate, even less tenacious, is placed first into the composite structure, serving as receptor of fragments resulted by dynamic breaking of the hard material.

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I.PO.13

THIXOFORMING AND POWDER METALLURGY COMPARATIVE STUDY AND PRACTICAL CASE FOR OBTAINING PISTON HEADS

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Keywords: piston, powder metallurgy, thixoforming, metallic matrix composite

Abstract: The engine can be considered as the heart of any means of transport. One of the key parts is the pistons, which transforms chemical energy of the burned fuel into a mechanical energy [1]. For this reasons, the pistons are submitted to a complex combination of thermal stresses and high temperature mechanical cycles [2]. In this study both powder metallurgy (PM) and thixoforming are used to process a metallic matrix composite (MMC) as a promising material for pistons. Aluminum as matrix and copper powder, to enhance thermal conductivity, and glass fiber, which increases Young's modulus and a lower thermal expansion coefficient, as reinforcement, are obtained for this aim. The following images in Fig. 1 are showed the excellent distribution of the glass fiber in the matrix. These results can be the basis of new research to develop to obtain materials for piston functions.



Fig. 1. Images for optical microscope at 100x magnification. a) thixoforming, b) powder metallurgy

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I.PO.14

PROPERTY IMPROVEMENT of SUBZERO/CRYOGENIC HEAT TREATED CAMSHAFTS MADE OF 8620H, 16MnCr5 and 100Cr6 STEELS

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Keywords: camshaft, subzero/cryogenic heat treatment, retained austenite, wear, hardness, 8620h, 16mncr5, 100cr6

Abstract: Several types of Camshafts that made of 8620H, 16MnCr5 and 100Cr6 steel were subzero and cryogenic heat treated and the effect of these heat treatment parameters such holding duration and temperature on the microstructure, retained austenite volume ratio, hardness and wear resistance were investigated. Camshafts for all kind of steels were grinded and different heat treatment cycles have been applied. 8620H, 16MnCr5 grade camshafts were carburized at 925-940°C 'in endogas (%20 CO, %40N2, %40H2) atmosphere between 660-1440 minutes. At the end of carburing, the diffusion temperature was decreased to 830-850°C (holded 90-150 min.) which is the austenizing temperature and the samples were hardened in oil at 60-80 °C .Samples were subzero treated at -80°C and -120°C 'for 120-150 minutes and were tempered at 160-200 °C for 120 minutes. The microstructure was revealed that the subzero/cryogenic heat treatment increased the hardness up to 62 HRc and increased the wear resistance of camshafts surface. The decrease in the retained austenite ratio was observed from %25 to %5-10 after cryogenic heat treatment. All of steels the hardness values were increased with the transformation of retained austenite into martensite. This increase is the result of the transformation of martensite from retained austenite and the carbide precipitation mechanism. It can be concluded that subzero/cryogenic treatment results with a increase in the wear resistance and the mechanical properties for all type of camshaft steels were being controlled with different heat treatment and physical metallurgy mechanisms, due to the formation of fine and homogeneous carbide particles during cryogenic treatment and coherency of interface between fine carbide particle and steel matrix[1-4].

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I.PO.15

REDUCTION OF CuO IN H₂ BY USING CONCENTRATED SOLAR ENERGY

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Keywords: copper oxide reduction, copper, hydrogen, concentrated solar energy

Abstract:

The reduction of metal oxide to obtain metal is a high temperature process which means very high energy consumption. The use of concentrated solar energy (CSE) to supply the energy required is envisage as a realistic alternative to implement "green" process in the pyrometallurgical sector [1]. In this sense, our work shows the preliminary results of the reduction of CuO in H_2 atmosphere, using a



Fig. 1. XRD pattern and optical microscope of Cu obtained by CSE

Vertical Axe Medium Size Solar Furnace of the PROMESS-CNRS solar installation with 1.5 KW of thermal power at the focus (15 mm) and power density up to 16 MW/m². Compacted CuO powder was subjected to the concentrated solar radiation under a 5% H₂/95% N₂ gas flow. The sample reached a temperature of 1000 °C in 3 min and it was maintained for 7 min. Fig. 1 shows the XRD pattern of the sample obtained. Peaks correspond to hkl indexes (111, 200, 220) of Cu. A typical Cu morphology with dendritic microstructure was observed by optical microscopy of the polished surface. The reduction of copper oxide follows the sequence CuO \rightarrow Cu₂O \rightarrow Cu [2]. These promising

results can be the base to prepare new alloys which are obtained by a complex conventional process.

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I.PO.16

DETERMINATION OF DISCRETE MICROSTRUCTURAL MODIFICATIONS IN GREY CAST IRONS

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Keywords: cast irons, elasticity modulus, internal stress, microstructure, XRD spectroscopy

Abstract: This study highlights an original approach in characterizing the internal stress relaxation in the structure of different types of grey cast irons by analyzing changes of the longitudinal elasticity modulus and the logarithmic decrement over a period of 38 years in which the test pieces have not



Fig. 1. Experimental schematic setup for determining the resonance curves in cast irons

undergone mechanical stresses.

The evaluation of the elastic properties was carried out by analyzing the resonance curves of the test pieces subjected to forced bending oscillation (Fig.1) [1]. It has been concluded that microstructural discrete changes are produced in cast irons which could be identified only by dynamic tests. In all the analyzed specimens, the elasticity modulus increased within the limits 1.04 -5.94%. The logarithmic decrement, respectively the internal friction decreased within the limits 1.98 - 11.4%. These results lead to the conclusion that

in the structure of cast irons which have not undergone heat treatment for stress relief, several processes occur as a part of internal energy dissipation in the material, correlated with complex diffusional processes that are difficult to evaluate by traditional methods of analysis. The results obtained are in good agreement with other microstructural analysis methods, such as SEM and XRD.

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I.PO.17

CORROSION PROPERTIES OF FEMTOSECOND LASER TEXTURED TI6AL4V-ELI IN SIMULATED BODY FLUIDS

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Keywords: Laser surface modification; micro-structuring; femtosecond laser; Ti6Al4V-ELI; corrosion

Abstract: The performance of the materials used in implants depends both on their bulk properties and on the surface characteristics; in this sense, implant surface topography can improve cell responses at the interface with the surrounding tissue and promote osseointegration [1]. Laser micro-machining is a successful process for surface modification that allows to create highly controlled textures [2]. Nerveless, essential properties such as corrosion resistance must be conserved during surface modification [3]. In the present work, a femtosecond laser system was implemented to obtain ordered patterns, such as arrays of grooves and dimples, on a Ti6Al4V-ELI alloy surface. A study on electrochemical behavior was performed in simulated body fluid in order to analyze, in a comparative way, whether the corrosion properties are modified after laser texturing. In addition, scanning electron microscopy was used to characterize the topography of the patterned surface and an optical surface profiler was applied to measure the size of resulted microstructures. The results highlight the ability of a femtosecond laser to obtain the desired texture in the biomaterial surface as well as its influence in biocompatibility and electrochemical properties.

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I.PO.18

PASSIVE FILM ON TITANIUM-TANTALUM ALLOYS. 1) EIS STUDY

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Keywords: titanium, tantalum, EIS, medical devices

Abstract: The limited mechanical properties and the toxicity of certain alloying elements require the development of new titanium alloys [1-3]. Recently, titanium-tantalum binary alloys have been developed and are expected to become promising candidates for medical applications due firstly, to



Fig. 1. Nyquist spectra for Ti-30Ta

alloying with tantalum, which is a nontoxic element, and secondly, due to their compatibility with bone tissue. The studied titanium tantalum alloys were Ti-5Ta, Ti-15, Ti-25Ta and Ti-30Ta. All measurements were carried out in Ringer solution at different applied potentials in 3 domains: anodic transition, passive and near transpassive transition (see Fig. 1).

The obtained impedance spectra exhibited one time constant suggesting the formation, in these conditions, of a compact layer on the surface of the alloys. The response is determined by the bulk oxide conductivity and its

geometrical capacity. The influence of the alloying element, tantalum, is reflected, mainly, in an increasing oxide resistance and growth rate. Among Ti-Ta alloys, the Ti-25Ta exhibits superior properties of the passive film and corrosion behavior, therefore it appears to be a promising candidate for metallic biomaterials.

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I.PO.19

VALIDATION OF 42CrMo4 STEEL FOR A "MAHLE" PISTON HEAD TAKING INTO ACCOUNT THE THERMO-MECHANICAL REQUIREMENTS

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Keywords: alloy stell, materials property, piston, diesel engine

Abstract: Increasing the performances of supercharged diesel engines with direct injection, has imposed the use of steel pistons, which can better resist at thermomechanical requests in comparison with aluminum alloy pistons.

Due to the cooling channels used by aluminum pistons, they can just be done by gravity casting technology using salt cores. For this reason, for a given engine an aluminum piston is heavier and has a head taller bigger than a piston made from steel, instead for reamings with dimensions of 90 mm upwards.

Due to the configuration of the cooling zone of the head of the piston, it is significantly reduced the transfer of heat from the head of the piston towards the cylinder and the segment, compared with aluminum pistons.

From the technological point of view, this solution offers the possibility of obtaining temperature around 250° C values in the bottom of the first channel for fire segment .

Simulation results are presented in this paper using the following characteristics of material: tensile strength, yield strength, elongation at fracture, fatigue strength, Young's Modulus, thermal conductivity factor, average linear thermal expansion, density.

Validation of the material was done by determining of temperature fields in the piston, thermomechanical stresses and internal stresses fields for the demands of the piston.

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I.PO.20

TENSILE STRESS-INDUCED STRUCTURAL CHANGES ASSOCIATED WITH MARTENSITE TRANSFORMATIONS IN Fe-Mn-Si BASED SHAPE MEMORY ALLOYS

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Keywords: shape memory alloys, martensite, tensile tests, X-ray diffraction, microstructure.

Abstract: Specimens belonging to two Fe-Mn-Si shape memory alloy (SMA) systems were obtained with chemical composition Fe-28Mn-6Si-5Cr and Fe-14Mn-6Si-9Cr-5Ni (mass. %). The former was produced by ingot metallurgy (IM) [1] while the latter was produced by both IM [2] and powder metallurgy (PM) [3]. PM specimens were sintered and pressed from as-blended powders as well as from mixtures of as-blended and mechanically alloyed (MA) powders, under volume fractions as high as 50 % [4]. Both IM and PM specimens were hot rolled and solution treated (1100^oC/ 5 min/ water), then tensile specimens were wire-cut by spark erosion and pre-strained by static tensile loading-unloading tests, to permanent strains between 3 and 4 %. This procedure aimed to stress-induce martensite, which was further analyzed, on the gauges of pre-strained specimens, by optical and scanning electron microscopy (SEM) as well as X-ray diffraction (XRD). Thermally induced reversion to austenite, of stress-induced martensite, was emphasized, during heating, by differential scanning calorimetry (DSC) as well as cinematographic analysis, monitoring consecutive shape changes due to free recovery shape memory effect.

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I.PO.21

RECOVERY OF PRECIOUS METALS FROM ANODIC DISSOLUTION SLIME BY EXTRACTION IN IONIC LIQUIDS

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Keywords: WEEE, anodic dissolution, anodic slime, ionic liquids, electrodeposition, silver, gold,

Abstract: The recycling of metals from electrical and electronic equipment waste (WEEE) is of great concern today. The work described in the article focuses on the application of ionic liquids (ILs) to selectively recover precious metals (Ag and Au) from the anodic slime obtained at the anodic dissolution of cast WEEE. The ingots obtained from molten and cast anodic slime is selectively dissolved in ionic liquids. The chemical composition of the ingots is: 12-15 wt.% Cu, 20-22% Sn, 3-5% Pb, 38-40% Ag, 18-20% Au and 1.5-3% other metals. The ionic liquids used (DES type) are mixtures of choline chloride with ethylene glycol in a 1:2 molar ratio. As catalytic agent, there was used pure iodine in a concentration of 0.1-0.3 mol·dm⁻¹. The operating temperature was maintained at 25-30 degrees Celsius. Cyclic voltammetry was employed for the determination of the electrochemical windows of ILs as well as of the dissolution and electrodeposition potentials of principal metals present in the ingot (anode). For Ag and Au, the deposition potentials determined are 0.07-0.08 V and respectively 0.4-0.5 V. The SEM and EDAX analyses revealed that the content of precious metals in the cathodic depositions is 98-99 wt% for Ag and respectively >99% for Au. The work demonstrates that ILs could be a solution to selective recovery of precious metals from WEEE.

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I.PO.22

ISOTHERMAL DIAGRAMS OF INTERMETALLIC PHASES FORMATION IN HEAT-RESISTANT TITANIUM ALLOYS

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Keywords: Titanium alloys; silicides; α2-phase; precipitation.

Abstract: Heat-resistant titanium alloys are known to combine high specific strength, high ductility and satisfactory creep resistance at operating temperatures. The combination of these features provides their high reliability in operation of products in the aviation and space engineering. The structural investigation of titanium alloys is usually focused on the morphology and behavior of the main phases — α and β . However, the alloying by silicon and aluminum leads to the formation of Ti₃Al and different silicide particles [1, 2]. The purpose of the present study was to investigate the microstructure evolution of heat-resistant Ti-Al-Sn-Zr-Mo-Si alloys containing various amount of aluminum, zirconium and tin using transmission and scanning electron microscopy. Moreover, the processes of silicide and aluminide particles precipitation at different conditions of heat treatment have been analyzed. The temperature ranges of intermetallic phases formation have been examined and the possible mechanisms of the different types particles transformation have been analyzed. Based on obtained results the schematic isothermal diagram of intermetallic phases formation has been suggested. It shows that the decay of the metastable β -phase (β met) begins with the precipitation of supersaturated α^* and β^* phases. Aging at 600...650°C leads to precipitation of (TiZr)₅Si₃ (S₁) silicide particles at α/β - boundaries. At the same time α_2 -phase is formed in supersaturated α^* -phase in the temperature range of 400...750°C. During long exposure at temperatures above 700 °C S_1 silicides are enriched by zirconium. Therefore, it can be formed to silicides $(TiZr)_6Si_3$ (S₂) or $(ZrTi)_2Si$ (S₃) according to the ratio of Zr/Si in alloys. In general, the precipitation of silicide particles has a negative impact on service properties of alloys. The precipitation of S_1 silicides on the interphase α / β -boundaries decreases the technological plasticity of the alloys, and the formation of S_2 particles in the body of α -phase decreases the characteristics of heat resistance due to the depletion of solid solution by silicon and aluminum and the difficulty of the formation of α_2 - phase particles.

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I.PO.23

INVAR TYPE NANOCRYSTALLINE COMPACTS OBTAINED BY SPARK PLASMA SINTERING FROM MECHANICALLY ACTIVATED POWDERS

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Keywords: Invar type alloy, spark plasma sintering, mechanical alloying

Abstract: In 1987 Guillaume discovered the Invar effect to Fe - Ni alloys with face-centered cubic (fcc) structure with Ni concentration around of 35 %. This effect consists in an extraordinarily low thermal expansion coefficient (near zero) over a wide range of temperature [1]. The Invar's coefficient of



Fig. 1. ΔI versus Temperature for Invar type nanocrystalline compacts.

expansion at thermal ambient temperature is $2x10^{-6}/K$ and is much lower as compared to the coefficient of thermal expansion of most metals - (10 ÷ 20)x10⁻⁶/K [2]. It is known that an important role in the behavior of type INVAR effect plays the relationship between magnetization and crystal parameters [3]. This paper is focused on compacts obtaining by spark plasma sintering (SPS) from Invar and Invar alloyed with Cu powders. The Invar/Invar with Cu powders was synthesized by mechanical alloying route. For Invar type compacts obtained from Fe and Ni₃Fe powders the coefficient of

thermal expansion value is 0.6×10^{-6} /°C in the temperature range of $20 \div 220$ °C. In the case of compacts obtained from Fe₉₀Cu₁₀ and Ni₃Fe prealloyed powders the value of coefficient of thermal expansion is almost 10 times higher – 5.6×10^{-6} /°C between 20 and 270 °C. The evolution of Δ I versus Temperature for both Invar type compacts is shows in Fig. 1.

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I.PO.24

STUDY OF THE INFLUENCE OF GRAIN SIZE ON THE TOTAL MAGNETIC LOSSES IN SILICON STEEL

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Keywords: non oriented electrical steel, roll, magnetic loss, grain size, cold rolled strip

Abstract:

The purpose of this work was to study microstructural changes of the bands investigated during processing occurring siliceous strips with non-oriented grains , and the study the influence of grain size on the total magnetic losses at 1.0T and 1.5 T.

There have been studies 10 rolls intended to be processed into quality electrical steel M400-50A (according to EN 100027-1) rolls who underwent conventional lamination technology. For the 10 rolls were made measurements of magnetic characteristics at the induction J = 1 500 mT and at a frequency f = 50 Hz and after that, we made correlations between the specific losses, and grain size.

Materials were analyzed by metallographic microscope Neophet 32 and the magnetic characteristics was made with Epstein frame according IEC 6040/, with an exiting current frequency of 50Hz at 1.5T and 1.0T induction after aging treatment of 2250C for 24 hours. Sample for light microscopy observation were prepared by polishing and etching in 5% Nital.

Subsequently, the microscopic analysis was performed on a band in which the non-oriented grain silicon steel which have 1.224%Si , wich was decarburized and annealed, so obtaining the different values of microstructures with different grains size.

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I.PO.25

MECHANICAL PROPERTIES AND MICROSTRUCTURE ASPECTS OF AlSi10Mg CASTING IN VIBRATING FIELD

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Keywords: AlSi10Mg alloy, casting, vibration, silicon phase

Abstract: Aluminum alloys are one of the most extended groups of functional materials by reason of combination between good mechanical properties and low mass. In [1] Sun et al. analyzed the nucleation and growth of eutectic cell in hypoeutectic Al–Si alloy. The tested material was Al–10%Si alloy modified by 0.025% Sr. Authors suggested that eutectic nucleation mode cannot be determined based on the



crystallographic orientation between eutectic Al phases and the neighboring primary dendrite Al phases. The changing of the silicon morphology with increasing of vibrations' amplitude was demonstrated by Abu-Dheir et al [2]. The decreasing of the silicon lamellas thickness during AI solidification with vibrations up to 24 Hz frequency was demonstrated by Chirita et al [3]. The aim of the present paper is to test the influence of the vibrations on the AlSi10Mg (EN 1706-2010) solidification. For comparison, six lots were casted in cold and

preheating molds, with or without vibrations within 50 Hz and 1050 Hz. For each casting frequency was measured the silicon phase dimension, hardness, tensile stress and impact strength. Increasing the vibration frequency the structure will be refined and also the size of the silicon phase is decreasing (fig. 1). The mechanical properties increase with the vibration frequency.

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I.PO.26

STUDY OF HYDROABRASIVE WEAR OF HARD THIN FILMS DEPOSITED ON SPECIAL STAINLESS STEEL USED AT FRANCIS TURBINE BLADES

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Keywords: thermal spraying, thin films, hydroabrasive wear, SEM

Abstract: The paper presents a comparative study of hydroabrasive wear of thin films deposited on special stainless steels with alloys based on W (88W2C) and Ni (NiCrBSi 74-14). The stainless steel used as basis material for deposition is GX3CrNi13-4 (1.6982), which has good properties of corrosion resistance but with low properties of hydroabrasive wear. The method of deposition is plasma jet thermal spraying. The analysis of deposited thin films has been carried out with Scanning Electron Microscope (SEM). The deposited pieces were subdued to hydroabrasive tests, the wear degree being quantitative determined. Alloys based on iron, nickel, chrome, titanium are used in the construction of hydraulic turbine blades. In this paper, the special stainless steels class has been approached. X2CrNiMoCuWN25-7-4, X3CrNiMo13-4, GX5CrNi19-10, GX3CrNi13-4 make part of this class, because they have closed mechanical, physical and chemical properties and during the research one of these variant will be chose. The properties of metallic materials can be improved by thermal treatments, thermo-chemical treatments and by thin films deposition [1,2]. Along with conventional technologies for obtaining deposited layers, the development, improvement and expansion of modern techniques for depositing them through physicochemical methods which ensure purity and high adhesion through a wide variety of processes for thin films are remarked. These modern methods of deposition with special materials can be used to increase the resistance at hydroabrasive wear and corrosion [3].

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I.PO.27

DENDRITIC SEGREGATION OF Zn-AI EUTECTOID ALLOYS

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Keywords: Zn-Al alloys, eutectoid alloy, segregation

Abstract: Good exploitation (antifriction capability, corrosion resistance) and good technological (excellent castability, cutting machinability) properties make Zn-Al alloys promising material for industrial applications, where, currently more and more compositions have been employed (from 3-5% to 22-27%). Introducing Zn-Al alloys with increased Al content, the difficulties related to the single phase Al-Zn alloys appear, mainly due to the dendritic segregation [1]. In Al-Zn system, the diffusion of the alloying elements takes place, at low velocity, determining significant structural modifications compared to the equilibrium structure, like the presence of the eutectic transformation in the alloy with 40% (wt.) of Al [1].

In this paper, qualitative and quantitative investigations of the Zn-Al eutectoid alloy (22% Al) structures in equilibrium and non-equilibrium state have been considered and studied. The performed investigations pointed out that also in this composition the dendritic segregation determines the eutectic transformation. Solidification in non-equilibrium condition controls both the eutectic transformation as well as the significant variation of the chemical composition with in the dendritic unit and in other constituents). The most important effect of the dendritic segregation is on the ratio of different phases (of the different constituents) and on their geometry and distribution within the metallic matrix, i.e., the topology. The phenomena involved in such conditions are governed by the structural transformations, which take place during solidification and cooling in solid state [2]. The research have been carried out through microstructural analysis by optical and scanning electron microscopy and through thermoanalytical measurements by dilatometry and differential scanning calorimetry.

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I.PO.28

THERMAL STABILITY AND MAGNETIC PROPERTIES OF NICOFECRGA HIGH ENTROPY ALLOY

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Keywords: High entropy alloys, Structural Transition

Abstract: The aim of this work is to study the magnetic and mechanic consequences of FCC to BCC structural transition due to applied heat treatments in NiCoFeCrGa (FCC+BCC) high entropy alloy. As the volume fraction of FCC/BCC phases plays key role in the physical properties, the evolution of the phases was studied as a function of annealing time and temperature. By scanning electron microscopy (SEM) and electron backscattered diffraction (EBSD) investigations (see Fig. 1) we

observed a new needle like BCC phase evolved inside the FCC grains. This small amount of this new BCC phase increased the hardness from 200 to 400 Vickers. Moreover the Ga (as an sp element) addition [2-3] and heat treatment change drastically the magnetic landscape [1] of the sample, investigated by MFM.



Figure 1: Phase composition (left) and Image Quality map of annealed NiCoFeCrGa sample.

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I.PO.29

EFFECT OF PHASE COMPOSITION OF EK77 ALLOY ON THE MELT VISCOSITY AND THE FEATURES OF THE INGOT STRUCTURE FORMATION

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Keywords: austenitic alloys, thermo-time processing of melts, σ-phase, aging

Abstract: The stability of the supersaturated solid solution of non-ferrous aging alloys is a fundamental characteristic which determines the stability of the material properties during operation The degradation of properties during the operation of different constructions at elevated temperatures is a common incident. It is obvious that the tendency of the supersaturated solid solution to the decay will be determined by the chemical composition of the alloy and the density of crystal lattice defects. However, it was recently shown [1] that a solid solution stability may greatly depend on the melting mode and the initial state of charge materials. The effect of the charge phase composition and the melting mode on the stability of the solid solution after the remelting and homogenization was shown using the corrosion-resistant austenitic alloy EK77 susceptible to 6-phase precipitation in the temperature range of 600...1000 ° C. By means of high-temperature viscometry, optical and electron microscopy, X-ray structural analysis, STA and durometry it was found that the presence of a second phase in the initial structure of the charge changes the viscosity of the alloy in the liquid state and reduces the solid solution resistance to decay after the remelting and homogenization. The fundamental basis of modern ideas about the nature of metallic melts is their micro inhomogeneity allowing the presence in the melt of atoms groups (clusters) ordered in a certain way. Kraposhin et al. [2] have considered that the melting is a polymorphic transition with the formation of the phase with noncrystallographic long-range order. Moreover, they are often isomorphic to various topology close packed (TCP) phases. The scheme of melt structural state changing under high temperature impact has been proposed based on these positions. It consists of the changing of clusters pattern: grinding, and then restructuring in the structure elements of FCC lattice. The relationship between the original structure of the EK77 alloy before remelting and structure forming during aging of pre-homogenized alloy after the remelting can be schematically shown based on all the studies. In this scheme it is clearly seen that the presence of 6-phase in the EK77 alloy before melting adversely affects the stability of the austenite during aging after remelting.

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I.PO.30

RELATIONSHIP BETWEEN DEFORMATION AND RECRYSTALLIZATION TEXTURE IN COPPER WIRE

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Keywords: drawing, deformation, recrystallization, copper wire, texture, strain-stress state

Abstract: By means of EBSD in case of electrolytic tough pitch copper wire, drawing texture of FCC metals has been shown to consist of several preferred orientations, similar to the rolling texture. Texture similar to the shear one was formed in periphery region of the wire, although it was rotated to texture of central region at an angle slightly smaller than 90°. Distribution of the main preferred orientations along the cross section of deformed wire has been explained with stress-state occurring upon drawing in the central and periphery regions. Relationships between deformation and recrystallization textures in regions with different stress-strain state were investigated. Regularities of formation of recrystallization texture depending on the deformation texture has been discussed from the point of mobility of CSL boundaries between <111> and <100> orientations – $\Sigma 25b$.

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I.PO.31

NITI SHAPE MEMORY ALLOY USED FOR MULTIPLE-RESETTING ACTUATOR FOR FIRE PROTECTION

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Keywords: NiTi shape memory alloy, martensitic transformation, thermal expansion, fire protection, DSC.

Abstract The paper introduces the possibility to replace the "wet alloy", used for sprinkler-triggering within automatic fire protection systems, with a shape memory alloy (SMA) type. The idea of the present application is based on the thermoelastic reversible martensitic transformation, governing SMA functioning, which has completely reversible character, and enables the occurrence of two-way shape memory effect (TWSME) after the application of a thermomechanical treatment called



"training" [1]. For this purpose a commercial NiTi rod, which was martensitic at room temperature, was subjected to thermal analysis tests, performed by differential scanning calorimetry (DSC) and dilatometry. Martensite (M) reversion to parent phase (A), during heating, was emphasized by an endothermic peak on the DSC thermogram, in Fig.1(a) and by a length shrinkage, on the dilatogram, in Fig.1(b) which shows, as well, the capacity to develop TWSME, by the change in displacement-temperature variation, with increasing the number of training cycles. This stabilized fully reversible behavior recommends NiTi rods as executive elements of a new concept of resettable sprinkler for fire protection.

Fig.1 Thermal analysis experiments emphasizing martensite (M) reversion to parent phase (A) during heating of a NiTi rod: (a) DSC; (b) dilatometry

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Acknowledgements: This research work was supported by UEFISCDI through project codes PN-II-PT-PCCA-2011-3.1-0174, contract no. 144/ 2.07.2012 and PN-II-ID-PCE-2012-4-0033, contract no. 13/ 2.09.2013.


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SECTION II Biomaterials

Chairpersons:

 Iulian ANTONIAC, University Politehnica of Bucharest, Romania
 Mariana CĂLIN, Leibniz Institute for Solid State and Materials Research Dresden (IFW Dresden), Institute for Complex Materials, Dresden, GERMANY
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II.K.1

EVALUATION OF DIFFERENT MAGNESIUM BASED ALLOYS WITHOUT ALUMINIUM FOR POTENTIAL ORTHOPEDIC IMPLANTS

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Keywords: magnesium alloys, orthopedic implants, microscopy, corrosion, surface.

Abstract: Various studies on magnesium alloys have shown encouraging potential in their development as biodegradable implant materials used in orthopedic applications [1]. The objective of this study was to characterize and test different biodegradable magnesium alloys from the system



Fig. 1. Microstructural aspects for an experimental MgCa0.8 magnesium alloy: light microscopy (left) and scanning electron microscopy (right).

Mg-Ca and Mg-Zn-Zr, in order to identify an optimal magnesium alloy for orthopedic applications. Microstructural characterization was performed them, using MO, SEM-EDS, in view of establishing correlations between microstructure and their properties. Experimental magnesium alloys was tested by immersion in different simulated mediums [2]. We follow the hydrogen release rate, pH variation, weight loss, and the modification of the surface morphology. Also, based on the

experimental results obtained after immersion test we calculate the corrosion rate. As conclusion, we observe that the testing medium have a strong effect on the corrosion rate. Especially the medium who contain proteins have a less pronounced effect and looks to be more appropriate to the real degradation in living tissues. Also, chemical and microstructural composition of magnesium alloys had a strong influence on the rate of degradation and surface morphology. It was found that these alloys are too accelerated degradation rate compared to the needs imposed biodegradable orthopedic implants. Based on our experimental results, we consider that it is necessary to modulate the rate of degradation for these magnesium alloys in order to be more closed to the clinical need imposed to the orthopedic implants in term of mechanical properties and surface stability..

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II.K.2

SYNTHESIS AND CHARACTERIZATION OF SOME CERAMIC COATINGS ON Mg-Ca-Zr METALLIC SUBSTRATE FOR MEDICAL APPLICATIONS

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Keywords: zirconia coatings, Mg-Zr-Ca alloys, electrochemical tests, biodegradation, MTT test



Fig. 1. ZrO_2 - Y_2O_3 – ceramic

Abstract: Coatings are considered an alternative to the improving process of mechanical, corrosion and cell viability properties of magnesium based metallic alloys. The most known coating techniques for these kinds of materials are micro-arc oxidation and sol-gel method. The paper proposes an extensive study focused on obtaining and characterization of two types of ceramic coatings using plasma atmospheric deposition: ZrO_2 - Y_2O_3 and ZrO_2 -CaO on a Mg-Ca-Zr substrate. It has been observed the morphology, specific phases and constituents using SEM and X-ray diffraction. Elastic modulus and apparent coefficient of friction were also calculated. Electrochemical analyses for corrosion rate determination were performed in Ringer solution at different periods of time: one hour, one day and one week. Biocompatibility was evaluated with indirect toxicity tests using a cellular line with

osteoblastic phenotype (human osteosarcoma line). The results of XRD presented the predominant cubic type compound - ZrO_2 . Values of elastic modulus of the layers are between 11.37-27.54 GPa for ZrO_2 -Y₂O₃ and 16.7-30.54 GPa for ZrO_2 -CaO. The corrosion rate for ZrO_2 -CaO has superior values than ZrO_2 -Y₂O₃. Cellular viability results studied by MTT colorimetric tests (3 - (4,5 - dimethylthiazol – 2 - yl) - 2,5 - diphenyltetrazolium bromide tetrazolium salt) haven't reveal significant differences of cytocompatibility between the coatings.

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II.O.1

INDUCED WETTABILITY AND SURFACE-VOLUME CORRELATION OF COMPOSITION FOR BOVINE BONE DERIVED HYDROXYAPATITE PARTICLES

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Keywords: surface composition, wetting properties, hydroxyapatite particles, XPS, EDS, FT-IR, XRF

Abstract: Hydroxyapatite powders represent the base material of multiple conventional and solid freeform fabrication methods for bone reconstruction products. In this stage of production, powder characteristics need to be determined both for quality control purposes and for a proper control of microstructural features of the final product. Besides the physical, chemical, and structural characteristics of the powder, the particles surface needs to be characterized mainly because those surfaces will become grain boundaries in the fabricated material, thus directly influencing its microstructural evolution [1-2].

This study focuses on the surface characterization of hydroxyapatite (HAP) particles prepared in the laboratory by thermal processing of bovine bone. Wetting properties of HAP particles were characterized by contact angle measurements and the surface composition was established by X-ray photoelectron spectroscopy (XPS). The XPS results were included in an extended analysis performed between complementary compositional techniques with different analysis depths (EDS, FT-IR, XRF). All compositional results were evaluated against powder particle size and process parameters used in bone thermal treatment, to establish adequate characterization protocols for ceramic particles based on the specific requirements of different conventional and solid freeform fabrication methods.

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II.O.2

EFFECT OF SINTERING TEMPERATURE ON STRUCTURE AND PROPERTIES OF SOME NEW HYDROXYAPATITE-BASED COMPOSITES FOR BONE TISSUE ENGINEERING

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Keywords: ceramic composites, hydroxyapatite, sintering, bone tissue engineering.

Abstract: The aim of this study is to evaluate the effect of sintering temperature on structure and properties of some new hydroxyapatite-based composites potentially used for bone tissue engineering. As raw materials for the experimental hydroxyapatite-based composites we use



Fig. 1. SEM image-surface of the experimental composite after microhardness test, 1000X.

hydroxyapatite extracted from bovine bone (BHA) as matrix and beta - tricalcium phosphate (β -TCP) with magnesium oxide (MgO), in different ratio, as reinforcement material.

The experimental composites were: BHA + 10 % β -TCP + 20 % MgO; BHA + 15 % β -TCP + 15 % MgO; BHA + 20 % β -TCP + 10 % MgO. For processing the mixed powders, we used cold isostatic pressing followed by sintering at three different temperatures: 1000°C, 1100°C and 1200°C, in order to develop the optimal

sintering temperature for better mechanical properties. The structural characterization of the experimental composites was made by XRD, FTIR and SEM. Microhardness and compression strength of the obtained composites was measured. In conclusion, we consider that the experimental hydroxyapatite-based composites sintered at 1100°C have a great potential to be used as bone substitutes from the mechanical properties point of view. Of course, if their biodegradation profile according clinical needs and citotoxicity will be confirmed by the further study.

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II.O.3

MORPHOLOGY AND BIOCOMPATIBILITY CHARACTERIZATION OF NANOSTRUCTURED TiO₂ SURFACES FOR BIOMEDICAL IMPLANTS DEVELOPED BY ELECTROCHEMICAL ANODIZATION

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Keywords: TiO₂ nanotubes, electrochemical anodization, biocompatibility, Ti6Al4V

Abstract: Titanium based implants are of great importance in craniofacial surgery and orthopedics, providing excellent outcomes in terms of functionality. The aim of present research was to develop ordered TiO_2 nanotubes on Ti6Al4V surfaces for biomedical implants, using electrochemical



procedure, anodization and to assess the biocompatibility between nanostructured layer and human osteoblasts. We have successfully developed ordered nanostructured TiO₂ layer on the surface of Ti6Al4V alloy for biomedical applications (orthopaedic and dental implants) by using electrochemical anodization in H₃PO₄/HF electrolytes. Our results on surface morphology show ordered nanostructures development in H_3PO_4/HF electrolytes, nanotubes have internal diameter of ~50-100 nm (see figure) and 200-250 nm thickness. As regards biocompatibility the response of osteoblasts behavior when in contact with

titanium based nanostructured surfaces shows a fast and strong adhesion osteoblast-titanium in the very first moments after the contact, whereas is progressively weakened in time.

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II.O.4

CORROSION BEHAVIOR OF BIODEGRADABLE FeMnSi-MgCa ALLOY

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Keywords: biodegradable, corrosion rate, Fe-base, "in vitro", "in vivo"

Abstract: Iron-based materials are a proper solution for biodegradable applications based on them mechanical and chemical properties. In order to control the corrosion rate of the material we propose to use two addition elements as Ca and Mg. The new material was obtain in Ar controlled atmosphere furnace, resulted fine times for chemical homogenization and microstructural and chemical analyzed using SEM and EDS techniques. Furthermore the material was "in vitro" and "in vivo" analyzed by corrosion resistance point of view. After experiments the surface was 2D and 3D analyzed using Vega Tescan software in order to determine the effects of the biological environment on the material surface. The "in vitro" experiments were realized by immersion (7, 14 and 30 days) in SBF solution at 37° C and constant pH and through electrochemical tests also in SBF electrolyte. The "in vivo" tests were realized by implantation on vivant rabbits the metallic materials for 30 days using standard protocol routine. Using EDAX detector(Bruker) we analyze the materials surface to establish the compounds form on the surface after experiments and the elements last on top of the implant (the first part expose to environment) from the main alloy elements (Fe, Mn, Si + Mg, Ca). All samples, for both tests, were "weighed" before and after experiments. A discussion on the degradation rates of the materials obtained from different tests was realized for comparison the results for "in vitro" (two experiments) and "in vivo". The results present a good chemical composition homogeneity after the re-melting stages with small percentages of Ca and Mg in the material but with a good spread in the alloy. After chemical experiments, at the surface were observed two main areas, one with corrosion pits and the other with new compounds on it. The stability of the compounds on the surface was analyzed trough ultrasound cleaning for 1 hour in isopropyl alcohol. After the cleaning stage the surface samples were analyzed by EDS.

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II.O.5

RESVERATROL COVALENT IMMOBILIZATION ONTO CELLULOSE ACETATE MEMBRANES FOR IMPROVED OSSEOINTEGRATION

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Keywords: Cellulose acetate membranes, osseointegration, resveratrol

Abstract: Covalent immobilization of resveratrol onto cellulose acetate polymeric membranes is presented for potential application in the improvement of osseointegration processes. For this purpose, cellulose acetate membrane is hydrolysed in the presence of potassium hydroxide, followed by covalent immobilization of aminopropyl triethoxy silane. Resveratrol was immobilized onto membranes using glutaraldehyde as linker. The functionalized membranes were thoroughly characterized by different characterization techniques such as infrared spectroscopy (FT-IR), Raman spectroscopy, Xray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA/DTG). Subsequently, in vitro cell tests were performed for evaluating the cytotoxicity of synthesized materials and also the osseointegration potential of obtained derivatized membrane material. Obtained results were compared with previously reported results regarding immobilization of sericin onto cellulose acetate membranes [1, 2].

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II.PO.1

CHARCOAL OBTAINED FROM THE WASTE VEGETABLE PRODUCTS, USED LIKE SUPPORT FOR PLANT EXTRACTS

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Keywords: plant material, pyrolysis of activated charcoal, mathematical models.

Abstract: The study presented in this paper is part of an extensive research on the development of enzyme dietary supplements with balancing role on digestive functions.

The main objective of this work was the use of wastes resulted from plant material and cakes remaining after obtaining extracts. This objective has led to pyrolysis the wasted plants, aimed at making charcoal as support for their own extracts. Another objective was to characterize the enzymes from plant extracts as fresh juices, which were subsequently adsorbed onto activated charcoal support. The plant material used in that studies was represented by: Seabuckthorn (*Hyppophae rhammnoides*), Black currant (*Ribes nigrum*), Aloe (*Aloe arborescens*), Parsley (*Petroselinum crispum*). By cold pressing, were obtained following extracts from: fruits (buckthorn, black currant), leaves (aloe, parsley). From this cold pressing process were resulted cakes representing wastes (peels and seeds). Also were resulted wastes material from harvested plants such as spines and twigs. The assumption of that plant residues were used for recovering, it were morphologically characterized and after pyrolised, and so making charcoal, characterized and used as support for their own extracts. The extracts obtained were analyzed in terms of interest of bioactive compounds (enzymes).

There were also investigated initial and final absorption concentrations on activated charcoal using adsorption kinetics. Adsorption kinetics were examined and evaluated by using Langmuir, Freundlich, Temkin models. Experimental results on the use of activated charcoal as adsorbent material for extracts indicate that the adsorption process showed good effectiveness, in correlation with the main objective.

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II.PO.2

OBTAINING AND MECHANICAL PROPERTIES OF TI-MO-ZR-TA ALLOYS

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Keywords: titanium alloys, biomaterials, mechanical properties.

Abstract: Ti-based alloys are successfully used in the area of orthopedic biomaterials for their enhanced biocompatibility, good corrosion and mechanical properties. The most suitable metals as an alloying element for orthopedic biomaterials are zirconium, molybdenum and tantalum because are non toxic and have good properties. The paper purpose development of three alloys of Ti-Mo-Zr-Ta (TMZT) prepared by arc-melting with several mechanical properties determined by microindentation. The mechanical properties analyzed was Vickers hardness and dynamic elasticity modulus. The investigated alloys presents a low Young's modulus, an important condition of biomaterials for preventing stress shielding phenomenon.

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II.PO.3

ZIRCONIA DENTAL IMPLANT MATERIALS

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Keywords: zirconia, dental, implants, biomaterials

Abstract: Ceramic materials are used for the fabrication of dental restorations respectively esthetic dentistry. The main ceramic materials are glass ceramics, spinel, alumina and zirconia. Zirconia was introduced into dentistry domain in the 1990s used like frameworks, implants, dowels, abutments and orthodontic brackets. Recently, zirconia materials are getting much attention for dental implants because of its toothlike color, mechanical properties, good corrosion and biocompatibility. This article presents an review of zirconia dental implants osseointegration and mechanical strength compared with other dental implants. Clinical studies published indicate that zirconia dental implants have the potential to become alternative of titanium dental implants used in medical applications.

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II.PO.4

BIOSYNTHESIS OF METALLIC NANOPARTICLES USING MICROORGANSMS

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Keywords: synthesis, silver nanoparticles, zinc nanoparticles, bacteria

Abstract: Formations smaller than 100 nm are called by specialists in the field, nanoparticles (the prefix is derived from the Greek $v\dot{\alpha}vo\varsigma$, meaning "dwarf"). Nanoparticles have captured the interest of researchers for several reasons and directions.

Firstly, we put great emphasis on their wide applications, the most important area being medicine. Different kind of metallic nanoparticles, like Ag NPs, are used for high anti-microbial activity, cancer treatment, drugs delivery, antibiotics [1]. Metal oxide nanoparticles, for example ZnO NPs, are in an advanced stage of research, for special photocatalytic and cosmetic properties.

Secondly, we are focusing to develop nanoparticles synthesis methods. Until recently when chemical and physical methods represented the most accessed and simple ways to obtain nanoparticles, nowadays, the biosynthesis using microorganisms is among the most promising techniques.

Biosynthesis is an eco-friendly and a low-cost technology, which provides great results, regarding the size of nanoparticles, which can reach between 1-100 nm, most of them having spherical shape. Using different microorgansims, like *Bacillus subtilis, Bacillus amyloliquefaciens, Rhodococcus erythropolis*, in this process allows the user to optimize the biosynthesis process, thus tailoring the shape and size of the nanoparticles.

The characterization of nanoparticles obtained using microorganisms, starts usually with ultra-violet visible spectrophotometry (UV-visible), which shows if the synthesis takes place intra- or extracellulary. After this, the characterization continues regarding the structural information with X-ray Diffraction (XRD), from a morphological point of view, Scanning Electron Microscopy (SEM) or Transmission Electron Microscopy (TEM) and very important in observation of surface topography, Atomic Force Microscopy (AFM)[2].

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II.PO.5

EFFECT OF SIC PARTICLE SIZE ON THE PHYSICAL AND MECHANICAL PROPERTIES OF NITI ALLOYS

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Keywords: NiTi shape memory alloy, SiC, powder metallurgy, wear.

Abstract: NiTi shape memory alloys (SMAs) have attracted significant interests due to their unique shape memory effect (SME), superelasticity and high damping performance [1,2]. In this work, the



Fig.1. SEM images of the Ti₅₀+Ni₄₉+1SiCp composite

effect of SiC particle size on both physical and mechanical properties of NiTi matrix composite were investigated. SiC of particle size 20 µm and 40 µm, and NiTi powder of particle size 45 μ m were used. Composites of NiTi with 1wt.% SiC were fabricated by powder metallurgy technique. The effects of SiCp additions, hardness, porosity and wear behaviour on the characteristic of NiTi composites have been investigated. The samples were examined by scanning electron microscope (SEM), for microstructural studies and phase identification. The results showed that the distribution of the reinforced particle was uniform. Moreover, as the SiC particle size decreases, hardness and wear resistance

increase. It was demonstrated that SiC particles size significantly enhanced the wear resistance of NiTi composite.

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II.PO.6

XRD ANALYSIS AND FTIR STUDY ON CALCINED BONES

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Keywords: calcination, hydroxyapatite, chemical and structural properties

Abstract: The present work focuses on the study of cortical bone samples of different origins (human and animal) subjected to different calcination temperatures (700, 800, 900, 1000 and 1100°C) with



Fig. 1. FTIR spectra of the calcined samples at 700°C

regard to their chemical and structural properties [1,2]. For this purpose, standard techniques such as Fourier transform infrared spectroscopy, X-ray diffraction and Scanning electron microscopy were used to investigate bone properties. These techniques provide the change in organic and inorganic content across the species and in different conditions. At 700 °C the organic component was removed and a carbonate apatite was obtained. At 900 °C, carbonate was no longer detected and traces of CaO were found at 1100 °C. Crystallinity degree and crystallite size progressively increased with the calcination temperature, contrary to

porosity that strongly decreased at

elevated temperatures. The results show that the calcination temperature highly conditions the properties of the human and animal bone samples. As expected, higher temperatures lead to more pure forms of hydroxyapatite, with higher crystallinity degrees and larger crystallite sizes and a less porous structure [3].

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II.PO.7

CHITOSAN/CASEIN MULTILAYER FILMS AS A NOVEL DRUG DELIVERY SYSTEM

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Keywords: polyelectrolyte multilayers, chitosan, casein, drug delivery, drug release

Abstract: The aim of the present study was to investigate the influence of the structure and physicochemical properties of chitosan/casein multilayer films on their potential as drug delivery systems. The multilayer films were prepared using the layer-by-layer self-assembly method of chitosan and casein on polylactic acid substrates, preliminary treated in corona discharge system [1]. The deposition process was studied by changes in the ATR FT-IR spectra, zeta potential and surface energy. ATR FT-IR spectra proved the formation of polyelectrolyte complexes between the chitosan and the casein []. The increasing content of chitosan and casein with increasing the number of bilayers was further confirmed by XPS analysis. Surface topography was investigated by AFM and the average roughness was evaluated. According to the drug release studies the multilayers showed a potential to be used as drug delivery systems.

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II.PO.8

ALLOYS WITH SUPERIOR MECHANICAL PROPERTIES USED IN MANUFACTURING OF IMPLANTS

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Keywords: biomaterials, cobalt alloys, EDAX, tensile strength, fluidity

Abstract:

This paper presents the characterization of a new alloy CoCrMoSi6, in terms of mechanical properties using tensile strength and the study of fluidity. The original version of the alloy was obtained by casting process in a vacuum arc furnace. Experimental results obtained from this study confirms that by increasing content of silicon, the mechanical properties are superior and the positive results obtained at fluidity studies favoring the formation of compounds, that lead to the reduction of alloying grade for α solid solution and the plasticity of the alloys.

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II.PO.9

INFLUENCE OF THE MODULATED TWO-STEP SYNTHESIS OF BIOGENIC HYDROXYAPATITE ON BIOMIMETIC PRODUCTS` SURFACE

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Keywords: naturally derived HAP, Ca/P ratio, SEM, contact angle, surface roughness, in vitro assay

Abstract: Processing of marble and seashells into biomimetic products is motivated by the widespread of these biogenic resources and by the necessity of such products on the market. This study targeted the optimization of Rathje synthesis method by chemical composition modulation of final ceramic powders (CaCO₃ dissociation through CO₂ emission at high temperature, followed by CaO transformation into Ca(OH)₂ by hydration and followed by its conversion to HAP using the addition of phosphoric acid at various concentrations) [1]. Synthesis stage (concluded with drying thermal treatment) was ensured by hydraulic compacting of powders (grain size of 10-50 μ m) with ultrapure deionized water. After morpho-compositional and structural characterization of powders (by SEM/EDS, FT-IR and XRD), pellets surface features were evaluated by roughness parameters and wettability influencing factors (surface tension, contact angle) assessment. *In vitro* biocompatibility assay was conducted by indirect cytotoxicity testing (cellular viability and proliferation) [2]. It was established that acid concentration is the key-factor for Ca/P molar ratio adjustment and thus the amount of synthesized HAP. The study revealed also a correlation between the chemical composition, the material structure, the pellets surface morphology and the *in vitro* behavior during cell culture experiments.

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II.PO.10

OVERVIEW OF MOST POPULAR ROMANIAN MEDICINAL AND AROMATIC PLANTS AS POTENTIAL SOURCES OF BIOMATERIALS

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Keywords: Medicinal and Aromatic Plants (MAPs); biomaterials; plants wastes

Abstract: Nowadays, human society search for a balance between multiple socio-economic forces including the consumption, overuse of resources, traditional resource depletion, climatic changes, the approach of renewable resources usage or environmental protection.

Exploiting of Medicinal and Aromatic Plants (MAPs) as raw material, by-products and waste derived therefrom, can take advantage of greater collaboration between research and industry, applying the processing systems necessary to treat large volumes of biomass characterized by specific economic values, requiring new processing technologies capable of reducing solvent consumption and increase overall environmental sustainability cycle.

The category of biomaterials as raw materials, products and waste from the processing of MAPs is a resource for obtaining: compounds (alkaloids, polyphenols, glycosides, terpenes), essential oils, substances for pharmaceutical use, dietary supplements, nutraceuticals, pigments and dyes, cosmetics and personal care products for plant protection/crop plants [1]. These can be used as a resource nonfood industries such as the production of biofuels, preparation of vegetable polyesters (polyhydroxyalkanoate - PHA) and biopolymers, rubber and textiles [2].

In conclusion, the use of biomaterials and MAPs subsumed them represent a direction that brings the balance to some extent sought or at least pave the way towards this balance.

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II.PO.11

STRUCTURAL AND COMPOSITIONAL ANALYSIS OF PHOSPHOCALCIC BIOACTIVE GLASSES

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Keywords: bioactive glasses, sol-gel process, apatite

Abstract: In this paper are present experimental results in case of two phosphocalcic glasses composition: 50% SiO₂ - 45% CaO - 5% P₂O₅ and 47% SiO₂ - 45% CaO - 5% P₂O₅ - 3% Ag₂O obtained by sol-gel process. In order to study the bioactivity of of these compositions, apatite formation and



Fig. 1. Sol-gel glass synthesis (gel samples)

other compounds resulting from the same process after 14 days of soaking in simulated body fluid (SBF) have been analyzed by X-ray diffraction analysis. Functional groups present in the structure of the two sol-gel glasses before and after soaking in SBF were highlighted infrared spectroscopy (FTIR). bv Thermogravimetric (TG), differential scanning calorimetry (DSC) and dynamic light scattering (DLS) analysis have been carried out in order to study thermal stability and size distribution of glass particle. TG analysis confirm stability of sol-gel glasses at high temperatures. The

results of X-ray diffraction analysis revealed the formation of apatite on the surface of glass powders soaked in SBF for 14 days, this phenomenon has been confirmed by FTIR analysis.

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II.PO.12

SURFACE MODIFICATIONS OF TITANIUM-BASED IMPLANTS: THE EFFECT OF SURFACE CHARACTERISTICS ON BIOCOMPATIBILITY AND OSSEOINTEGRATION

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Keywords: titanium, nanotubes, osteoblast adhesion

Abstract: Nowadays titanium is among the most preferable biocompatible metals for implants manufacturing [1]. Anodization of titanium is a feasible method to produce ordered arrays of nanotubes on titanium surface, which may improve the osseointegrative qualities of implants. Since implants are used for bone defect sites, osteoblast-like cell cultures serve as ideal in vitro model systems for biocompatibility and osseointegration tests [2]. The current study aimed to analyze osteoblast adhesion on anodized nanotubular titanium. After anodization in NH₄F the titanium surface was oxidized and possessed nanotubular structures, uniformly distributed over the whole substrate surface. The analysis of samples employing Carl Zeiss Auriga CrossBeam scanning electron microscopy (SEM), revealed that the inner diameter of nanotubular structures was equal to 35±4.29 nm. For *in vitro* biological assays, rat osteoblasts were used: a total of 1.1x10⁵ cells in 1 ml of culture medium were seeded on anodized and flat-surfaced titanium discs, placed into 6-well culture plates. Cell adhesion, determined after 1 day, was shown to be significantly higher on the nanotubular anodized titanium. Indeed, 57% more cells were counted on anodized substrates in comparison with flat-surfaced titanium. SEM of cells, seeded onto anodized titanium discs, was conducted at three different time points: 2 h, 1 day and 4 days. After 2 h, a round cell morphology was observed, which is a sign of the initial stage of cell adhesion. Osteoblasts, cultured for 1 and 4 days, exhibited mostly spindle-like and branched morphology, which points to a successful adhesion and proliferation. The latter was also confirmed by staining the cells with DAPI and TRITC-phalloidin. To summarize, our data, consistent with several other studies, indicates, that the further biomedical research, employing anodized titanium, is promising. In particular, the design of nanotubes, delivering drugs and/or the binding of biomimetics onto the arrays of nanotubes, deserve a special attention.

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II.PO.13

THE STUDY OF MATERIALS USED TO MANUFACTURE PROSTHETIC COMPONENTS FOR MEDICAL IMPLANTS

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Keywords: biomaterials, 316L stainless steel, Ti6Al4V titanium alloy, medical prostheses, corrosion behavior

Abstract: The materials used to manufacture prosthetic components must meet the conditions of biocompatibility imposed by the human body, to be mechanically resistant, to present a good resistance to corrosion, to be resistant to aging process and not to generate flows of attrition particles.



Fig. 1. An image by optical microscopy, of a AISI 316 L steel sample analyzed by OES technique

This paper aims the selection and testing of some biocompatible metallic materials (of the austenitic stainless steel and titanium alloy range) with optimal characteristics (high durability, low weight, good corrosion resistance) and mechanical properties used, in particular, with the aim to realize the metal elements of prosthesis for medical implants. For this reason, samples of 316L stainless steel and Ti6Al4V titanium alloy were analyzed [1,2]. The conformity requirements for biocompatible steels AISI 316L and Ti6Al4V are specified in the ISO 5832-1, ISO 13485 and ISO 9001 standard.

The main objective of conformity assessment is risk and malpractice diminishing in the medical practice. On the other hand, the practice of conformity assessment complies with ccurrent concepts of quality assurance and risk mitigation of products exploitation in the medical applications. The samples were morphologically and compositionally examined by using metallographic optical microscopy, optical emission spectrometry (OES), electronic microscopy SEM and corrosion tests.

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II.PO.14

BIOMATERIALS PERSPECTIVE ON THE COMPLICATIONS RELATED TO THE USE OF SURGICAL MESHES IN UROGYNECOLOGICAL SURGERY

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Keywords: polypropylene; surface, mesh; FTIR; SEM; adhesion.

Abstract: Stress urinary incontinence and pelvic organ prolapse represent important conditions affecting adult women's health. The essential property of good prosthetic mesh for gynecological surgery is its ability to be incorporated into endopelvic tissue. Materials investigated in our study are



Fig. 1. SEM image-interaction between polypropylene mesh and tissue, 50x.

polypropylene meshes used for surgical treatment of urinary incontinence. We analyze the structure and surface morphology of synthetic polypropylene meshes in comparation with some explanted fragments from the same mesh type after clinical use. Different clinical aspects related to the clinical use of the polypropylene mesh were taken in consideration. Structural characteristics were revealed by Fourier Transform Infrared Spectroscopy (FTIR) investigations and the

surface morphology by Scanning Electron Microscopy (SEM). Based on our results, we consider that the surface modification of polypropylene mesh after passing through the transobturator passage have a strong influence on the interaction biomaterials-tissue and the functionality of the polypropylene mesh. Also, analysis of the explanted meshes proves the encapsulation process of the mesh to be more similar to a foreign body reaction rather than to the adhesion and integration of the synthetic polypropylene mesh into the tissue.

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II.PO.15

IN VITRO COMPARATIVE ASSESSMENT OF THE CHEMISTRY, HANDLING AND MECHANICAL PROPERTIES OF CEMENTS FOR VERTEBROPLASTY

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Keywords: biomaterials, bone cements, spectrometry, microscopy, mechanical properties.

Abstract: The vertebral fractures represent significant societal burdens, especially for the elderly persons. One clinical method used for the treatment of the vertebral body compression fractures caused by different pathologies is Percutaneous Vertebroplasty (PVP), which supposes a



Fig. 1. Schematic representation of the use of bone cements for vertebroplasty

percutaneous injection of bone cement into a collapsed vertebral body in order to stabilize the vertebra. One important aspect is that the bone cements for vertebroplasty requires specific properties to support the spinal column, and induce specific requirements for the biomaterials used [1]. The material must be radiopaque to track filler material movement and detect or avoid material leak that may cause neurological or other tissue injury and present clear contract under fluoroscopy because the

surgical procedure is performed under fluoroscopy. From clinical point of view, they should be injectable and have adequate viscosity. The biomaterials handling characteristics must be: easy preparation, appropriate flow, and polymerization or crystallization characteristics. From the biomechanical point of view, they must have considerable mechanical strength and toughness. The purpose of this paper is to present a comparative analysis of two different bone cements for vertebroplasty, and other bone cements used for arthroplasty. Structural characteristics and morphological aspects were investigated using Fourier Transform Infrared Spectroscopy and Scanning Electron Microscopy, in different setting conditions. Also, it was evaluated the mechanical properties for experimental bone cements. Main issue revealed by our research was the strong influence of the chemical composition on the handling and mechanical properties. Also, based on our experimental results, we could confirm that the properties of bone cements for vertebroplasty.

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II.PO.16

FAILURE ANALYSIS OF A TITANIUM CLAVICULE PLATE

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Keywords: metallic biomaterials, failure analysis, microscopy, finite element analysis.

Abstract: This study presents a failure analysis of an orthopedic implant that was used for fixation of a clavicle fracture. The implant is a locking compression plate with 16 holes that was fixed to a broken clavicle using 7 screws. The study elucidates the causes of an in situ premature failure of the



Fig. 1. SEM image-detail of the plate failure area, 50X.

plate. Chemical analysis and microstructure analysis confirmed that the implant was manufactured from cold-worked unalloyed titanium [1]. The macro and micro fractographic analyses performed by stereomicroscopy and scanning electron microscopy revealed that the failure mechanism was fragile failure, probably in the most loaded area of the plate [2]. A finite element analysis (FEA) was accomplished in Ansys 12.0 environment in the various conditions, in order to identify the stresses acting on clavicle plate, when the assembly is subjected to certain loads. According to

this loading condition and to the material properties, the stress amplitude that initiated and propagated the crack is estimated to be in the range of 150 N. Also, FEA analysis of the assembly indicated that the crack sites are located in the region where the highest stresses are observed. This FEA numerical analysis confirmed that some patient activities induce the loading condition needed for this specific failure. Several considerations, from biomaterial, mechanical and clinical point of view, are discussed in order to explain the failure and to improve the system durability. From an engineering perspective, there are some possibilities to optimize the implant and clinical procedure in order to reduce the stress concentrations that developed near the crack origin.

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II.PO.17

ADDITIVELY MANUFACTURED FEMORAL STEM TOPOLOGY OPTIMIZATION: CASE STUDY

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Keywords: additive manufacturing, femoral stem prostheses, topology optimization.

Abstract: The main problem of femoral stem prostheses is their primary stability after implantation, that is assured by the so-called press-fit implantation, but immediately after that it is to install a secondary stability, due to the best osteo-integration of the prosthesis in the bone of the femur. Additive manufacturing (AM) allows the designer to create tridimensional models that can have a diffused porosity in the contact area with bone, which confers to the femoral stem significantly higher osteo-integration. Furthermore, through topology optimization, the shape of the prosthesis can be improved, in conjunction with significant mass reduction. This work presents our results concerning the topology optimization of a medium length femoral stem, intended to be manufactured by powder bed fusion. The CAD models have been analyzed through computer-aided simulations concerning their mechanical characteristics, according to ISO 7206, and were topologically optimized using commercially available software. The CAD models, both original and optimized, have been built by selective laser sintering and analyzed concerning their mechanical characteristics. An optimized femoral stem has been obtained, which answers to the ISO 7206, but with a 15% mass reduction.

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II.PO.18

COMPARATIVE STUDY OF MECHANICAL PROPERTIES FOR THE MAIN BRANDS OF DENTAL IMPLANTS

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Keywords: mechanical features, dental implants, Titanium alloy

Abstract: Main brands of dental implants demonstrates superior features confirmed for years now. Among them, specific mechanical properties of materials dental implants are made of, and mechanical properties of the parts itself, are together with surface treatments the most important traits behind their success.

The comparison among the main brands of dental implants was made by means of objective analysis (roughness measurement, SEM microscopy with identifying chemical composition, tear tests, etc). The study tried to penetrate into the depth of crystal structures and chemical formula behind good behavior demonstrated in clinical applications. The study demonstrates the diversity of titanium alloys used in the production of dental implants and opens opportunities of new formulas. New proportions between the percentages of aluminum or titanium and vanadium, and other elements use (zirconium, niobium, etc.) will make possible to obtain new implants with superior performance.

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Acknowledgements: We hereby acknowledge the project "Metoda inovativă pentru funcționalizarea suprafețelor implanturilor dentare cu scopul îmbunătățirii osteointegrării" cod MySMIS 104809, contract ANCSI 73/8.09.2016



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II.PO.19

RESEARCH OF POSSIBLE CAUSES BREAKING OF DENTAL IMPLANTS

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Keywords: dental implants, causes breaking, micro-fissure

Abstract: Dental implants become increasingly used among the population. This development led also to a variety of designs and equipment used in order to achieve increased surface characteristics for obtaining improved osseointegration. But due to the increased usage of them there comes an increased failure rate of implants. These shortcomings come because of complications which follow the treatment, because of its breakage from fatigue demands, defects of the material used for producing the implant or because of the execution of assembly errors. Although it is rare, these complications are very serious in dentistry. In order to introduce dental implants into clinical practice there is taken a lot of researches on biocompatibility. Problems regarding the material, its structure or properties are less studied in the specialized literature, those being studied by the producers.

Breaking dental implants during surgical maneuvers, prosthetic loading or during use (chewing, bruxism, accidents, etc.), is the second most common cause of loss of a dental implant after consecutive peri-implantitis rejection. Although the frequency of this type of failure for a dental implant is much smaller than those caused by peri-implantitis, detailed study of broken implants can explain possible causes.

Using scanning electron microscopy (SEM) in the study of areas of crack explain the mechanism of producing tears started from micro cracks that may be found inside the used alloy for producing dental implants.

This micro cracks in weak areas of implants (anti-rotational corners of polygons, etc.) generates a risk for tear on first increased demand.

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SECTION III Ceramics, Polymers and Composite Materials

Chairpersons:

Liana Sanda BALTES, Transilvania University of Brașov, ROMANIA Ionel CHICINAȘ, Technical University of Cluj-Napoca, ROMANIA Cătălin CROITORU, Transilvania University of Brașov, ROMANIA Santiago FERRANDIZ, Universitat Politècnica de València, SPAIN Rodica Mariana ION, ICECHIM, Bucharest; Valahia University of Targoviste, ROMANIA Dana LUCA MOTOC, Transilvania University of Brașov, ROMANIA Ionuț Claudiu ROATĂ, Transilvania University of Brașov, ROMANIA Michael TAUSCH, Bergischen Universität Wuppertal, GERMANY





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III.K.1

CRYOCONITE HOLES FROM SVALBARD GLACIER - NEW SOURCE OF MATERIALS

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Keywords: cryoconite material, quartz, muscovite, magnetite, illite, labradorite, chlorite serpentine.

In a world where the Arctic area is in a continuous dynamics, it is very important to study the behaviour of different materials which appear visible after the glaciers de-icing. Life on glaciers and ice sheets is mostly associated with airborne debris, so-called cryoconite material which binds to



Fig. 1. Svalbard glacier area (personal photo)

inorganic minerals, settles on to the ice [1]. In this paper the cryoconite materials from the Svalbard archipelago (in Arctic Ocean, between Norway and the North Pole) are analyzed and discussed. The mixed samples (water and soil) (Sample 1 Cryoconite Lonyearbreen from and Sample 2 from Cryoconite Larsbreen) were separated into soil samples (A,B) and water samples (A,B). The water and soil samples were analyzed by ICP-AES, energy-dispersive X-ray fluorescence (EDXRF), X-ray diffraction (XRD), SEM-EDS, OM. ICP-AES results for the water separated from the soil samples revealed

the presence of Dy and Ce (3,43 mg kg⁻¹ and 2.77 mg kg⁻¹, respectively) in A sample, but less than detection limit in B sample. By XRD some mineral phases have been detected, as: quartz, muscovite, magnetite, illite, labradorite, chlorite serpentine, in both samples. Also, the sulphur identified in the EDXRF spectra was quantified using a dedicated calibration curve, obtaining values of 575.46±14.85 mg kg⁻¹ for Soil A and, respectively, 666.88±11.73 mg kg⁻¹ for Soil B. The mineral dust on the ablation ice of glaciers is an aggregate of minerals, visible by scanning electron microscopy. Also, some varieties of sungite and taurite are detected and identified.

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III.K.2

SPARK PLASMA SINTERED COMPOSITE COMPACTS OBTAINED FROM Ni-Fe ALLOY@Ni_{1-x}Me_xFe₂O₄ PSEUDO CORE-SHELL POWDERS

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Keywords: nanostructured magnetic powders, pseudo core-shell powders, Rhometal interface

Abstract. High magnetic properties (magnetic flux density and magnetic permeability) and high electrical resistivity are required in many AC applications for magnetic cores, in order to reduce core losses. Usually, the powders Fe-based magnetic alloys are covered with a thin dielectric



Fig. 1. SEM/EDX image of microstructure of the Fe/NiFe₂O₄ composite (600 MPa, 700 $^{\circ}$ C)

organic/inorganic layer [1, 2]. To avoid the decrease of the magnetic properties of the material by dielectric layer, we use a magnetic dielectric layer which consist in a layer of nanosized soft magnetic ferrite particles, in order to electrically isolate metallic particles between them, figure 1 [3].

In the Fe-Ni alloys@Ni_{1-x}Me_xFe₂O₄ composite powders the core is a large metallic particle and shell is a pseudo continuously layer of nanosized soft magnetic ferrite particles (NiFe₂O₄, Ni_{0.5}Zn_{0.5}Fe₂O₄, Ni_{0.5}Cu_{0.5}Fe₂O₄). The composite particles with pseudo-core shell structure have

been prepared using acetone as surfactant by mixing metallic particles with very small ferrite particles and subsequent annealing in argon [3]. The as obtained pseudo core-shell powders have been compacted by spark plasma sintering (400 °C – 900 °C, 20 MPa, 0 – 10 minutes). The sintered compacts have been investigated by: X-ray diffraction, scanning electron microscopy, energy dispersive X-ray spectrometry, magnetic and electric measurements.

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III.O.1

THE INFLUENCE OF COMPOSITION AND CALCINATION TEMPERATURE ON THE PHASES IN (Ba_{1-x}Sr_xZr)O₃ PEROVSKITES

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Keywords: cubic perovskite, barium strontium zirconate, microstructure, X-ray crystallography

Abstract: Several ceramic materials with a perovskite structure often exhibit interesting electronic properties and, therefore, are often called "electroceramics" [1]. Electroceramics with high dielectric constants are used in capacitors and superconductors [2]. The effect on calcination temperatures



Fig. 1. XRD patterns of ceramic powders

(1000–1400°C) and the changing of ions in A and B sites on the phase formation and microstructure of barium strontium zirconate ($Ba_{1-x}Sr_xZr$)O₃, BSZ, x = 0.2 and 0.4 ceramics were investigated. The BSZ powders were prepared using the solid state reaction method. The phase purity, crystal structure and microstructure of samples were examined using Fourier transform infrared spectroscopy (*FTIR*), X-ray diffraction (XRD) and field emission scanning electron microscopy (FE-SEM). We found out that the percentage phase purity of perovskite increases with an increase in the calcination temperatures

as expected and it was found that BSZ powders adopts a perovskite structure with a cubic (Pm3m) unit cell as well [3]. The purity of perovskite powders was obtained above 1300°C and the purity phase of ceramics was detected in all samples. The SEM results indicated that the particle size of the samples increased with the increase of the calcination temperatures.

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III.O.2

DIELECTRIC PROPERTIES OF Sr-DOPED BaZrO₃ CERAMICS

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Keywords: oxides, electron microscopy, powder X-ray diffraction, dielectric properties material

Abstract: Perovskite compounds have received keen interest from academic research due to their



Fig. 1. FE-SEM micrographs of $Ba_{0.6}Sr_{0.4}ZrO_3$ ceramics sintered at 1450°C for 2h.

unique properties such as ferroelectric, pyroelectric, piezoelectric, and dielectric properties [1-3]. Nanosized oxides of strontium doped barium zirconate of general formula $Ba_{1-x}Sr_xZrO_3$ (x=0, 0.2, 0.4, 0.6) have been prepared by a solid-state procedure.

The surface morphology, microstructure, and phase analysis, of the sintered ceramics were characterized by field emission scanning electron microscopy (FE-SEM) and X-ray diffraction (XRD) respectively.

Dielectric studies were also carried out as a function of frequency to explore the electrical properties of Sr-doped BaZrO₃.

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III.O.3

EFFECTS OF HEAT TREATMENT ON PHASE COMPOSITION OF MECHANOCHEMICALLY SYNTHESIZED MOLYBDENUM SILICIDE POWDERS

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Keywords: tungsten silicide, heat treatment, mechanochemical synthesis, mechanical alloying

Abstract: Molybdenum silicide has a great interest due to its high-melting temperature, high-temperature oxidation stability, high-temperature strength, thermal and electrical properties [1, 2]. The binary phase diagram of Mo and Si consists of three silicide compounds, $MoSi_2$, Mo_5Si_3 and Mo_3Si [3]. Furthermore, in the $MoSi_2$ stoichiometry, α -MoSi₂ (tetragonal phase, C11b type) and β -MoSi₂ (hexagonal phase, C40 type) phases exist. In our previous study [4], molybdenum silicide powders were obtained from MoO_3 -SiO₂-Mg initial powders by a two-step process of mechanochemically synthesized and selective HCI leaching. In the result of that study, the most efficient selected composition among the synthesized powders was composed of α -MoSi₂ (~73 wt.%), β -MoSi₂ (~11 wt.%) and Mo_5Si_3 (~16 wt.%) phases. The aim of this study was to investigate the heat treatment effects on powder composition of mechanochemically synthesized and leached molybdenum silicide powders which in the selected composition by using various temperature and durations in order to eliminate β -MoSi₂ and Mo_5Si_3 phases. Phase and microstructural characterizations of the heat-treated powders were held using X-ray diffraction (XRD), particle size analysis (PSA), pycnometer density measurement and scanning electron microscope (SEM) techniques.

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III.O.4

ANALYSIS OF TEMPERATURE EFFECT ON MECHANICAL AND TRIBOLOGICAL PROPERTIES OF SU-8 PHOTORESIST MATERIAL

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Keywords: SU-8 polymer, nanoindentation, tribomechanical properties, temperature effect

Abstract: In this study, the mechanical and tribological properties of SU-8 polymer from MEMS application were investigated as a function of temperature in a range between 20°C and 100°C. The samples for tests are SU-8 polymer fabricated at different temperatures: 125°C, 165°C, 195°C and



215°C. As a function of deposited temperatures, properties different of samples are determined. The mechanical properties under investigation are the modulus of elasticity and hardness. The tribological properties include the adhesion and friction forces. The tests are performed using an AFM XE 70 and a thermal controlled stage. As the

temperature for testing



increases the modulus of elasticity and hardness decrease based on the thermal relaxation of material [1, 2]. Figure 1 presents the nanoindentation experimental AFM curves of SU-8 polymer deposited at 195°C and tested at 20°C (Fig.1a) and 100°C (Fig.1b). As it can be observed, the modulus of elasticity decreases with 15% and the hardness with 25% based on thermal relaxation of material.

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III.O.5

ECOEFFICIENT MELTING OF CERAMIC FRITS

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Keywords: ceramic frits, glass, concentrated solar energy

Abstract: Ceramic frits are glassy materials prepared by melting a mixture of raw materials at high temperature (1350-1550°C) [1,2]. Burning fossil fuels furnaces are used to provide the energy







required for frit manufacture, which is a highly energy intensive process. This work aims to study the feasibility of using a solar furnace for melting different types of frits and compare the characteristics of the frits obtained with

crystalline frit

Fig. 1. Different frits obtained by CSE

those shown by frits of same composition but melting in a conventional electric furnace, in order to assessment the quality of final products in relation to the standard commercial ones. Different types of ceramic frits were studied. Their compositions were adjusted according to the chemical composition criteria which include glass formers, fluxing, stabilizers, opacifyers and devitrifying agents. Melting tests were performed into ceramic crucible. The concentrated solar energy (CSE) was provided by a Vertical Axe Medium Size Solar Furnace of the PROMESS-CNRS solar Installation with 1.5 KW of thermal power at the focus (15 mm) and a power density up to 16 MW/m². Total incident sun radiation was controlled by a shutter positioned between a sun-following heliostat and a parabolic concentrator. Fig. 1 shows the aspect of the different frits obtained by melting the raw materials under concentrated solar radiation and quenching.

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III.O.6

SOFT MAGNETIC COMPOSITES BASED ON HYBRID COATED Fe-Si NANOCRYSTALLINE POWDERS

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Keywords: soft magnetic composite, Fe-Si powder, hybrid coating, AC magnetic characteristics

Abstract: Soft magnetic materials play a major role in numerous electrical and electronic devices. The market of soft magnetic materials is dominated by Fe-Si steels (laminated sheets), the largest part of electrical motors and transformers being actually realized by a stack of laminated electrical steels



Fig. 1. The applicable regions for soft magnetic materials used in AC fields (after Ref. [2]).

and soft magnetic ferrites (especially for AC applications). As compared to the above mentioned classes of soft magnetic materials, soft magnetic composites (SMC) is a relatively new and promising class of materials. SMC's cover the regions were electrical steels and soft ferrites cannot be used (figure 1). Typically, an SMC consists of electrically insulated ferromagnetic particles that are compacted into 3D finished parts. The SMC offers a series of advantages over electrical steels and ferrites such as magnetic and thermal isotropy, relatively low total core loss at low and medium

frequencies, complex designs through the use of traditional powder metallurgy techniques etc. [1]. The Fe-Si powder used in this study was prepared by milling of Fe-Si laminated sheets. The structural investigation revealed that after milling a nanocrystalline α –Fe(Si) solid solution is formed (the mean crystallites size of 15 nm). In order to prepare the SMC's, the particles were subjected to a hybrid coating. This consist in covering the particles with an inorganic layer (phosphate) and an organic layer (polymer). The two coatings are complementing each other. The influence of the parameters of the coating process as well as the interface particle-inorganic coating is investigated. The influence of processing parameters (compaction pressure, the amount of polymer) over the AC characteristics (core loss, initial permeability) of the compacts up to the frequency of 10 kHz is discussed.

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III.O.7

CHARACTERIZATION OF THERMOPLASTIC BEARINGS BY USING OF HORIZONTAL ROTATIONAL FORMING

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Keywords: bearing, FEA, horizontal rotational forming, thermoplastic material

Abstract: The paper presents an experimental research of main characteristics of thermoplastic sliding bearings for machine tools, obtained by the horizontal-rotational forming process. Heat of plastic material at a melt-temperature point is realized by High-Frequency Current Induction (HFCI) on a centrifugal molding installation. The experimental tests were showed that between the melt-



Fig. 1 The principal of rotational forming process

from additive PA.6, PVC or PVA obtained by horizontal-rotational forming, and main characteristics have been determined on the base of calculus, and then confirmed by experimental tests and FEA by COSMOSXpress Analysis.

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temperature points of thermoplastics, speed and molding a revolution synchronism is required, which depends of material density and whirl form of melting. The predictable revolution speed of metallic mold was achievement by technological attempts, in function of workpiece size and type of material. The charge weight is experimental determined in dependence of part's thickness walls. This process leads to obtain the tub parts at high quality level and constant thickness walls that allowing the replacement of bimetallic sliding bearings. The thermoplastic sliding bearings with different internal diameters



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III.O.8

INFLUENCE OF AQUEOUS MILLING DURATION ON THE SINTERING ABILITY OF SOME HARD METAL POWDERS

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Keywords: WC-Co composite, hardness, oxidation, aqueous processing

Abstract: Hard metals represent a very important class of composite sintered products, showing an interesting combination of hardness, toughness and wear resistance. The characteristics of the hard carbide phase, the metal binder, as well as their interactions affect the final properties of the sintered product[1, 2]. In order to achieve a desired carbide particle size and a homogeneous chemical distribution of the phases, the powders are processed in attrition mills [3]. The excessive heating and oxidation upon milling are generally prevented by milling under organic media (alcohol, acetone or alkanes) [2, 4]. Changing the organic milling media with an aqueous one would be more environmentally friendly, cost effective and with reduced fire risks. However, preventing or controlling the oxidation of the constituents is a great challenge. This paper presents the effects of aqueous processing of WC-Co powders upon their sinterability and mechanical properties. WC-Co powders have been milled in aqueous media (distilled water + corrosion inhibitor), then the as-milled samples have been compacted and sintered in vacuum. The investigations performed on the two components, carbide and binder, reveal different oxidation behaviour in water, with proven further effect upon the properties of the sintered product. The decrease of the saturation magnetisation of the compacts corroborated with the microstructure aspect reveals the more intense oxidation of the longer duration milled samples. The magnetic properties, hardness and microstructure suggest that the used corrosion inhibitor is effective for short milling durations.

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III.O.9

METAL/OXIDE MAGNETIC COMPOSITES OBTAINED BY REACTIVE SPARK PLASMA SINTERING

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Keywords: magnetic material, spark plasma sintering, ferrite, metal/oxide composite

Abstract: Soft magnetic alloys, such as Fe-based and Ni-based possess high magnetic permeability and induction, combined with low coercive field and low electrical resistivity. The soft magnetic ferrites are materials that are used at high frequency due to their high electrical resistivity, but they



Fig. 1. In-situ X-ray diffraction recorded in temperature up to 1000 °C.

have lower saturation magnetization and permeability as compared to the alloys. Alloy/ferrite soft magnetic composites can combine the advantages of the two mentioned soft magnetic materials classes in order to fulfil the gap between these classes [1-2]. Spark Plasma Sintering - SPS technique becomes of research and industrial interest for materials with special applications and also for replacing the classical sintering for some materials. The SPS technique present some major advantages such as lower energy consumption, higher

heating rates, lower sintering temperature or much shorter sintering time as compared to the classical sintering. These special features allow the sintering of multicomponent materials with controlling the reaction between the phases and grain growth. Metallic/oxide composites with various composition have been synthesized by SPS using mixtures of easily accessible precursors. High temperature X-ray diffraction (fig. 1) and differential scanning calorimetry have been used for checking the temperatures reaction of the precursor powders. It has been studied the formation of various metallic/alloy composite and also de material densification by SPS. It was evidenced the influence of SPS temperature and holding time on composites formation and density.

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III.O.10

EFFICIENT TYPE OF BATTERIES FOR ELECTRIC VEHICLES POWERING

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Keywords: electric vehicle, battery, LiFePO₄

Abstract: In this paper the authors present the results following the tests of a Lithium Iron Phosphate (LiFePO₄) type of battery used for electric vehicles powering. Batteries from the Lithium-ion family are used for multiple applications, LiFePO₄ being the most efficient from our point of view in electric



Fig. 1. LiFePO₄ batteries package from E-Smart electric vehicle [3,4]

mobility applications [1,2].

The measurements was made on E-Smart electric vehicle, obtained through conversion of a Smart ForTwo City vehicle by changing the internal combustion propulsion system to a system that uses an electric three-phase asynchronous motor [3,4].

The purpose of this study is to analyze the influence of the type of battery materials and the chemistry between them and the phosphate to improve the energy storage systems for electric vehicles.

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III.O.11

THE THEORY OF GENERALIZED THERMOELASTICITY WITH FRACTIONAL ORDER STRAIN FOR DIPOLAR MATERIALS WITH DOUBLE POROSITY

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Keywords: dipolar materials with double porosity, fractional derivative, generalized thermoelasticity

Abstract: In [3], a new theory of thermoelasticity has been derived based on fractional order of strain (fractional order Duhamel-Neumann stress-strain relation). This paper constructs a mathematical model for the materials introduced in [2] and follows closely the techniques described in [3] for dipolar materials which have a double porosity structure. The method requires the law of conservation of mass, the equations of motion, the first law of thermodynamics and the equations of the state of the thermoelastic materials in a special form, determined by the form of the fractional derivative with respect to time. The heat conduction is described by Cattaneo's equations. The results are the constitutive equations of the linear theory of thermoelasticity with fractional order strain. The equations are valid for anisotropic materials and are called the Duhamel-Neumann equations with fractional order. In conclusion, this paper continues the work on dipolar thermoelastic materials, which are a special case of multipolar continuum mechanics introduced in [1]. This theory allows a double porous structure: a macro porosity connected to pores in the material and a micro porosity which reflects fissures in the porous skeleton.

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III.O.12

EFFECT OF WALNUT SHELL REINFORCEMENT ON POLYMER MATRIX COMPOSITES

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Keywords: Polymer matrix composite, mechanical properties, walnut shell waste, polyester

Abstract: Turkey produces exceeding 200,000 tons of walnut annually as the fourth largest producer in the world. The walnut shell amounts to 40-60% of the total weight rendering over 100,000 tons shell per year, mostly as waste. A limited quantity is utilized in the fiberboard manufacturing, as solid fuel, scrubbing material in the chemical and metal industries. The purpose of this study is the development of low cost polyester composites with enhanced mechanical properties by the incorporation of walnut cellulosic fibers as the reinforcing phase in the polyester resin. The process not only makes the production more economical but also reduces the amount of waste. The shells are ground and the detailed characterization of waste fibers is carried out by scanning electron microscopy, X-ray diffraction, laser diffraction and He gas picnometer techniques. The effects of the filler/matrix ratio and the particle size, shape and chemical treatment of walnut fibers on the physical and mechanical properties of the composite are determined. The composites are characterized by the porosity, density, three-point bending strength, impact resistance and hardness measurements. Structure-property relationship of developed composites was discussed.



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III.O.13

THEORETICAL COMPARISON BETWEEN CARBON FIBER AND GRAPHENE COMPOSITES FOR AUTOMOTIVE COMPONENTS

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Keywords: composite materials, carbon fiber, graphene

Abstract: This paper represent the experience of authors in the field of design, calculation and experimental research for automotive components, steering column bracket, in composite materials based on carbon fiber and graphene. The paper reveals the comparison between the materials from



Fig. 1. Steel bracket and CF composite bracket [1]

the point of view of strength provided to automotive components, mix between different compounds, weight gain, price and chemical analysis. Making the steering column bracket composite showed that it successfully replace can traditional materials, but one of the problems raised by using this composite were related to production from splinters structure resulted in an impact. The idea of using graphene to solidify the composite structure appeared due to this problem. From the beginning of experimental research, graphene was the first candidate to achieve full automotive components due to its well-known characteristics but the

price, tensile tests, chemical analysis showed that it's better to use a mix between the composite materials. Strength calculation comparison between the materials, testing results of different specimens and conclusions will be exposed in full paper and come to complete technical and theoretical information.

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III.O.14

LIFE CYCLE COST COMPARISON BETWEEN STEEL, CARBON FIBER COMPOSITES AND GRAPHENE COMPOSITES

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Keywords: life cycle, carbon fiber, graphene

Abstract: This paper presents a detailed comparison between standard and new generation materials related to the use in automotive purposes. Automobiles require a very wide range of materials with properties differing by their means of use. Thus, this comparison is helpful in order to



Fig. 1. Carbon composite revenues in US\$ million in the automotive sector according to sub-segment

operations, properties of composite materials etc.

In the same time, to have a better view of materials advantages, it was made a comparison between BIW using the three evaluated materials.

Conclusions will be exposed in full paper and come to complete technical and theoretical information.

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achieve the customer requirements regarding the highperformance cars with low maintenance cost.

Different aspects related from the beginning in designing automotive components are pointed out such as: required energy to produce different components using the evaluated materials, required energy to produce the raw materials, cars fingerprint, technology, weight, life of the components,



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III.O.15

NANOPARTICLE EMBEDDED MIXED MATRIX PSF MEMBRANES CHARACTERIZATION AND MEMBRANE PERFORMANCE

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Keywords: Membrane; Polysulfone; Nanoparticles

Abstract: Polysulfone is very used in membrane fabrication because of the advantages it posesses,



Fig. 1. SEM image of a polysulfone membrane fabricated by phase inversion

such as chemical and thermal stability. Because of the hydrophobic characteristic of polysulfone, membranes are susceptible to fouling

[1] and it is of great interest to reduce it.

Various modification techniques had been reported, including the use of additives, chemical treatments, grafting components, and coatings [2].

In this article, the blending modifications of polysulfone membranes with nanoparticles were performed to increase the membrane flux as well as retention of metiloranj dye.

Flat sheet polysulfone (PSf) ultrafiltration membranes were fabricated by phase inversion process (Fig.1).

The blending modifications allowed for improvements in the membrane permeability and the structural features.

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III.O.16

OPTIMAL COMPOSITION OF MIXED MATERIAL FOR THE THERMOFORMING PROCESS

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Keywords: material, hardness, thermoforming process

Abstract. This research was conducted to design and analyze experiment to find the optimal composition of a mixed for thermoforming molds for plastic plates using the vacuum process. The experiments used the combined mixture process design method [1], which was used to determine the optimal ratio of Resin Aluminum Talcum (RAT), temperature and setting time there were 46 experiments for this design[2]. The optimized ratio gave a contents of 52 grams resin, 22 grams aluminum, and 26 grams talcum, a temperature between 70-85 degrees Celsius and a setting time 5 hours. The results support the predicted value, where the optimal ratio is the maximum hardness strength. Maximum hardness strength is 80 Shore D. The mixed material can save 30% of the cost[3] when comparing between RAT with metal are used for thermoforming molds in the plastic plate vacuum process.

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III.O.17

EFFECT OF SEQUENTIAL MILLING (MECHANICAL ALLOYING-CRYOGENIC MILLING) ON THE MICROSTRUCTURAL AND MECHANICAL PROPERTIES OF TIB₂ REINFORCED AL-12.6 WT.% SI MMCS

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Keywords: Mechanical Alloying, Cryogenic Milling, TiB₂ Reinforcing Particulates, Al-based Metal Matrix Composites

Abstract: In this study, effect of sequential milling - mechanical alloying (MA) followed by cryogenic milling(CM) - on the microstructural, physical and mechanical properties of the TiB₂ reinforced Al-12.6 wt.% Si metal matrix composites (MMCs) were investigated. Furthermore, different contents of TiB₂ reinforcing particulates (2, 5 and 10 wt.%) were incorporated into the Al–12.6 wt.% Si matrix in order to observe the correlation between TiB₂ amount and sequential milling. TiB₂ reinforced Al–12.6 wt.% Si MMCs were synthesized by MA from elemental AI, Si and TiB₂ starting powders. MA time was selected as 4 h based on the scanning electron microscope (SEM) and differential thermal analyses (DTA) of the Al-12.6 wt.% Si-2 wt.% TiB₂ powders MA'd for 1, 4 and 8 h. Afterwards, 4 h of MA'd powders were cryomilled for different durations (10, 20 and 30 min) in the presence of N_2 gas circulated externally around the milling vial. X-ray diffraction (XRD) analyses were performed to calculate the lattice strains and crystallite sizes of the milled powders. Relative densities of the MMCs were measured by Archimedes' method. SEM and energy dispersive spectroscopy (EDS) analyses revealed that TiB₂ reinforced AI-12.6 wt.% Si MMCs were successfully fabricated with a fine and homogeneous microstructure. MA followed by CM provided a significant contribution on the mechanical properties of the TiB₂ reinforced composites. Sequentially milled and sintered MMCs exhibited higher hardness and relative wear resistance values than those of only MA'd and sintered samples.

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III.O.18

ANALYSIS OF FLEXURAL PROPERTIES FOR METAL/PLASTIC COMPOSITES MADE BY A NEW HYBRID PROCESS

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Keywords: ABS, Copper mesh, Composites, Flexural testing, Hybrid process

Abstract: Metal/plastic composites have been receiving unprecedented attention from the academia, mainstream media, investment community and national governments around the world because of their unique properties.

This paper presents a new hybrid process for the production of high-quality metal/plastic composites. The process is a combination of Fused Deposition Modelling (FDM), vacuum forming and CNC machining.

The research aims to provide details of the proposed hybrid process, equipment used and the experimental results of the composites which have been produced by stacking copper mesh (99.99% pure) layers on ABS (plastic). They have been subjected to flexural loading with a different number of metal mesh layers to serve as a proof of the methodology. The composites have been compared to ABS samples made according to British and International standards.

The test results show that the Cu/ABS samples have higher fracture load values compared to the parent ABS sample. Furthermore, as the number of Cu mesh layers increase in a single specimen, the fracture loads also increased demonstrating the effectiveness of the new hybrid process. The initial results are very promising and open up new horizons for the production of high-quality metal/plastic composites in a cost-effective and proficient manner.



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III.PO.1

MICROSTRUCTURAL ANALISYS AND MECHANICAL PROPERTIES OF AL COMPOSITE MATERIALS REINFORCED WITH PARTICLES BASED ON FE ALLOYS

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Keywords: metal matrix composite, matrix aluminum, particles, mechanical properties

Abstract: Composite materials are defined as systems of two or more components whose properties complement each other, resulting in a material with superior properties of the specific component. The paper shows the characteristics of Al matrix composites reinforced with particles based on Fe alloys. The properties of the obtained composite depend on the matrix and on the nature and volume fraction of discontinuities phase (reinforcement). The paper shows the processing techniques, the mechanical properties, and the microstructural analysis of the obtained composite. Composite microstructure was analyzed by optical microscopy and scanning electron microscopy. There were used specific installations to characterize the mechanical properties of the obtained composite.

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III.PO.2

FEW-LAYER GRAPHENE ENCASPULATED Fe-Co NANOPARTICLES: SYNTHESIS AND CONTROL OF MAGNETIC PROPERTIES

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Keywords: few-layer graphene, magnetic nanoparticles

Abstract: Magnetic nanoparticles have unique physical and chemical properties in comparison to bulk magnetic materials. Magnetic nanoparticles based on pure metal phases exhibit better magnetic performance in comparison to their oxide counterparts. However, they have high chemical activity



Fig. 1. TEM image of product obtained from anode containing 15% Co and -85% Fe.

and can undergo undesired processes when exposed to ambient conditions, e.g. oxidation, corrosion and agglomeration. The specific physical and chemical properties can be preserved when one applies the so-called encapsulation procedure. Recently, it has been demonstrated that carbon coatings in a form of few-layer graphene can be regarded as the perfect encapsulation agent [1]. The fewlayer graphene coatings perfectly isolate the magnetic core against corrosion and oxidation, and moreover they are susceptible for further functionalization [2].

Herein the results on the synthesis of few-layer graphene encapsulated Fe-Co magnetic nanoparticles (GEMNs) are presented. GEMNs were synthesized via carbon arc discharge route [1]. The graphite electrodes containing various content of Fe and Co were used as initial precursors. The process was carried out under $Ar-H_2$ atmosphere at 60 kPa and the

discharge current of 70 A. It has been found that the phase composition of GEMNs, their magnetic properties and diameter distribution can be precisely controlled by a simple macroscopic parameter, i.e. content of the metal precursor in the anode. The synthesis procedure is selective and exclusively yields few-layer graphene encapsulated nanoparticles (Figure 1). The obtained GEMNs have the mean diameter of 30-40 nm and their saturation magnetization reaches even 160 emu/g. The phase composition studies revealed that the inclusion of Co minimizes the formation of undesired paramagnetic austenite phase.

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III.PO.3

PHOTOACTIVE GLAZED POLYMER-CEMENT COMPOSITES

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Keywords: glazing, polymer-cement composite, tannin, waste water

Abstract: Polymer-cement composites, known as macro defect free cements (MDF) are characterized by remarkably high mechanical properties [1]. Their flexural strengths are 20-30 times higher than those of conventional cement pastes, nearly equal to that of an ordinary steel. The main drawback of MDF cements is their sensitivity to water [2, 3]. This paper presents a method to both diminish the negative impact of water on MDF cements mechanical properties and to enlarge their application by conferring photoactivity. These tasks were solved by glazing MDF cement with an ecological glaze containing nano-particles of TiO₂. Efficiency of photocatalytic activity of this material was tested against methylene blue aqueous solution (4.4 mg/L). Influence of the photocatalyst concentration in the glaze paste, of contact time and wave length of the UV radiation on the photocatalysis process was studied. The best obtained photocatalysis yield was of 97.35%, after 8 h of exposure to an UV radiation with a wave length of 254 nm, when used an MDF glazed with 10% TiO₂ in glaze paste. Surface of glazed material was characterized by optic microscopy, adherence, SEM, XRD. All these properties were correlated with the aesthetic aspect of the glazed surface aiming to propose using of this material for sustainable construction development.

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III.PO.4

PHOTOACTIVE POLYMER-CEMENT COMPOSITES FOR TANNINS REMOVAL FROM WASTE WATERS

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Keywords: coating, polymer-cement composite, tannin, waste water

Abstract: TiO₂ nano-particles exhibit remarkable photocatalytic activity against organic pollutant from waste waters [1,2] but their separation from liquid media after photocatalytic process is a very difficult task The immobilization of TiO₂ on different supports could be a good solution to simplify cleaning stage after photocatalysis [3]. In this paper, two types of photoactive polymer-cement composites named macro-defect free cements (MDF) have been obtained both by embedding TiO₂ nanoparticles (Degussa P25) into the composite matrix and by photocatalyst deposition on the composite surface by dip-coating method (in two types of recipe). Composites surface has been characterized by SEM and XRD and photocatalytic efficiency was determined against tannins from waste waters coming from woodworking industry. Tannins concentration was monitored by VISspectrophotometry, using Folin-Ciocalteu method. All specimens evidenced photocatalytic activity. The process efficiency is dependent on the light wave length and of irradiation duration. The best results have been obtained when MDF containing embedded TiO₂ nano-particles was exposed to 254 nm light (92% after 6 h). Under higher wave length light irradiation (365 nm), the photocatalytic efficiency decreased at 48.2% for MDF containing embedded TiO_2 and at 39.85% for MDF coated with TiO₂ film. The obtained results are promising both from environmental protection point of view, taking into account the persistence of tannins in waste waters and the difficulties of their elimination by using classic methods and from the point of view of MDF properties improvement, taking into account its very large range of applications.

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III.PO.5

OBTAINING AND CHARACTERIZATION OF CALCIUM CARBONATE AND WOOD REINFORCED PVC COMPOSITES

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Keywords: poly (vinyl chloride), amorphous calcium carbonate, FTIR spectroscopy, surface energy

Abstract: The advantages of using amorphous calcium carbonate filler for poly (vinyl chloride) are related to the reduction of the overall costs of the material, non-toxicity, good stability, and low moisture content. However, due to the dissimilar nature of the components,



Fig. 1. Microstructure of the PVC-CaCO₃wood composite

the dissimilar nature of the components, compatibilizing/dispersing agents are necessary. Among the most frequently used are stearates and silanes, which are introduced either in the organic or inorganic phase by means of adsorption from their corresponding solutions in ethanol or toluene. This paper aims to determine the influence of wood sawdust addition in poly (vinyl chloride)-calcium carbonate composites, as means of improving the interfacial adhesion between the organic polymeric phase and the inorganic filler, eliminating the need for volatile solvents and toxic compatibilizers use. A good coverage degree of the calcium carbonate and wood fillers by the polymeric phase has been determined by optical microscopy (Fig. 1) and SEM in conjunction with a good mechanical performance

(hardness, wear resistance, compressive strength), resistance to solvents action (distilled water, diluted acidic solutions), by comparing to composites without wood in composition.

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III.PO.6

WOOD-POLYETHYLENE COMPOSITES WITH ALKYLPHOSPHONIUM AND ALKYLAMMONIUM IONIC LIQUIDS AS COMPATIBILIZERS

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Keywords: ionic liquids, HDPE, wood, surface energy, FTIR spectroscopy

Abstract: Wood-plastic composites (WPCs) are nowadays competing in the market with common lumber, due to their acceptable mechanical properties, good water and biological agents resistance. However, their performance (and price respectively) is determined by the addition of coupling agents (compatibilizers), due to the dissimilar surface polarity of the components, as well as of different additives [1,2].

lonic liquids have attracted great interest lately due to their cellulose plastifying, wood UVstabilization, fire-retardant and fungicide character. They have been recently used as compatibilizers between several biopolymers and synthetic polymers, making them attractive coupling agents for WPCs obtaining, due to their high affinity to the wooden material [2].

This paper aims to obtain WPCs starting from high density polyethylene (HDPE) and wood flour, using alkylammonium alkylphosphonium and ionic liquids as compatibilizers, namely methyltrioctylammonium bis(trifluoromethylsulfonyl)imide and trihexyltetradecylphosphonium bis(2,4,4-trimethylpentyl) phosphinate. The wood flour has been treated with different amounts of ionic liquids, mixed with a determined amount of plastic material, molded, and heated to different temperatures, in the 120-200°C interval. The composites present good stability to solvents action, as well as minimal leaching of the ionic liquid compound from the structure of the material. The resistance to solvents action is in good agreement with the low values of the composites surface energy, determined by the dominatingly hydrophobic character of the used ionic liquids.

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III.PO.7

INFLUENCE OF BIODEGRADABLE POLYMERS IN THE WASTE GENERATED FROM POLYPROPYLENE FOOD CONTAINERS.

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Keywords: Polypropylene, biodegradable polymers, recycled polymers.

Abstract: Recycle polymers is common due to the need of reduce environmental impact of this materials. Polypropylene (PP) is one of the polymers call 'commodities polymers' and it is commonly used in food packaging as well as in a wide variety of other applications, and that is why its generate



Fig. 1. Sample recycled PP with 15% PHB, x3500.

a large amount of PP residue that can be recycled [1].

introduction of biodegradable The polymers in the food packaging industry can negatively affect the properties of recycled PP [2,3]. For this reason, we analyzed the have influence that generates small amounts of biodegradable polymers (PLA, PHB and TPS) in the recycled PP properties.

The recycled PP and biodegradables polymers blends were evaluated by mechanical testing and measuring the VST, MFI and FTIR.

The mechanical properties, the VST softening temperature and the melt

flow index are negatively affected by the presence of biodegradable polymers in PP recycled. Although, FTIR technique can detect biodegradable polymers in the PP recycled, because the spectrum of the PP is modified between the band 1700-1800 cm-1 (band characteristic of the hydroxyl group) due to the presence or PLA, PHB or TPS.

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III.PO.8

AIMg/AIN OBTAINING THROUGH THERMODYNAMICS COMBINED WITH EXPERIMENTAL INVESTIGATION

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Keywords: AlMg, AlN, diagram, "in situ", matrix

Abstract: Basic material concept, technology and some results of studies on aluminum matrix composite with dispersive aluminum nitride reinforcement was shown. Studied composites were manufactured by "in situ" technique [1].

Aluminum nitride (AIN) has attracted large interest recently, because of its high thermal conductivity, good dielectric properties, high flexural strength, thermal expansion coefficient matches that of Si and its non-toxic nature, as a suitable material for hybrid integrated circuit substrates [2].

AIMg alloys are the best matrix for AIN obtaining. Al₂O₃-AIMg, AIN-Al₂O₃, and AIN-AIMg binary



Fig. 1 – TEM replica of AlN fracture surface

O₃–AIMg, AIN–Al₂O₃, and AIN–AIMg binary diagrams were thermodynamically modelled. The obtained Gibbs free energies of components, solution parameters and stoichiometric phases were used to build a thermodynamic database of AIN–Al₂O₃–AIMg system. Obtaining of AIN with Liquid-phase of AIMg

as matrix has been studied and compared with the thermodynamic results.

The secondary phase microstructure has a significant effect on the final thermal conductivity of the obtained AIN. Thermodynamic modelling of AIN–Al₂O₃– AIMg system provided an important basis

for understanding the obtaining behavior and interpreting the experimental results. Fig. 1 shows the effect of both temperature and time on the sintering of AlN for the compositions under consideration. Basically all compositions reach complete densification after sintering at >1850 °C for 1 h. The experimental observations it is explained by the phase assemblage diagram.

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III.PO.9

STUDIES ON NOVEL CYANATE-ESTER POLYMER BLEND CURING BY AID OF 'DIELECTRIC BROADBAND RELAXATION SPECTROSCOPY

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Keywords: cyanate-ester, polymer blend, dielectric relaxation, glass transition

Abstract: Broadband dielectric relaxation spectroscopy represents one of the most versatile methods deployed to assess the glass transition of polymeric materials with a wide dynamic range of frequencies [1, 2]. The herein study aims to present the relaxation curves from dielectric measurements carried by aid of a high precision dielectric analyzer (Alpha analyzer, Novocontrol Technologies) in combination with a control temperature system in the temperature range 25° - 180°



C, in steps of 5 K/min and consecutive isothermal frequency sweeps (10¹ – 10⁷ Hz). The novel polymer blend developed contains bisphenol A dicyanate ester pre-polymer supplied as 75% solid in methyl ethyl ketone followed by mixing with a diglycidyl ether of bisphenol F epoxy resin under a 70:30 (vol.%) ratio. An overview of various dielectric relaxation the processes are given in Fig. 1 for the dielectric loss ε " function of frequency during the heating step from 80° to 135° C.

Fig. 1 Dielectric loss vs. frequency for the novel polymer 1

Selective references:

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III.PO.10

COMPOSITE MEMBRANES USED FOR THE PROCESSING OF SOME BIOLOGICALLY ACTIVE EXTRACTS

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Keywords: cardiovascular disease, delivery of liquid chromatography (HPLC), membrane separation, mathematical models (neural networks).

Abstract: The purpose of this study was to obtain a food supplement formula for the cardiovascular system stimulation. This product was obtained from concentrated fruit extracts: Hawthorn (*Crataegus monogyna*), Chokeberry (*Aronia melanocarpa*), Bilberry (*Vaccinium myrtillus*) and Rrosehip (*Rosa canina*).

The composite membranes for processing biologically active extracts approach a current theme of the field of science and technology with direct applications in biochemistry, biotechnology and biomedicine. Using the separation processes through composite membranes (polysulfone) represents the isolation and purification of natural substances from vegetable raw materials. The supplement formula which was developed at laboratory level was morphologically and compositionally analyzed through various techniques and tested by various methods. In order to analyze the natural extracts and the new formula, we improved the analytical methods and adapted them for the new product.

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III.PO.11

STRUCTURAL AND MAGNETIC PROPERTIES OF QUARTZ SAND

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Keywords: sand quartz, iron impurity, electron microscopy, magnetism

Abstract: For being used in glass industry at crystal glass production, the quartz sand must contain less than 0.09 % iron. If the sand contains more than 0.09 % Fe, the iron must be removed. Removal of the iron depend on other hand on the form in which it appears (hematite, magnetite, ilmenite, chromite) and how it is distributed in a particle of a given size. In the present study, the particle size of the sand was analyzed and the particle size distribution was established. Using scanning electron



Fig. 2. Iron particle on a quartz particle.

microscopy (SEM) and energy dispersive Xray spectroscopy (EDX), the morphology and the elemental distribution on the particle was performed on several particle size classes. The SEM analysis has given a connection between the shape and size of chemical particle with elemental distribution, as presented in Fig. 1 and Fig. 2. The evolution of the iron contend versus the particle size was established, and a nonlinear evolution was found, with a minimum for 126 µm particles. Using X-ray diffraction, the phases appearing in sand were investigated. The phases are changing their ratio versus the particle size. The main phase is SiO₂ as quartz, accompanied by minor phases: iron oxides (Fe₃O₄, Fe₂O₃, and FeTiO₃) and some oxide of Al, Na, Ca, and K. In order to be able to remove the iron containing particles, magnetic field must be used and in order to obtain a value of the necessary field, magnetic measurement was performed. On the magnetic hysteresis, the complex and multiple iron phases was observed.

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III.PO.12

REMOVAL OF CRYSTAL VIOLET BY ADSORPTION ONTO CHARCOAL MODIFED DEVELOPED FROM PINE CONES

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Keywords: activated charcoal, adsorption process, dye.

Abstract: Discharge of residual dyes in surface water represents a problem for aquatic life. These wastewaters are loaded with pollutants such as, dyes, heavy metals, surfactants, and contain a variety of organic compounds (which are harmful and have carcinogenic properties), such as their



Fig. 1. SEM image

complete biodegradation to be slow or even impossible.

Charcoal prepared from wastes of the local forest activated with NaOH solution and with titanium oxide (TiO₂) was used as adsorbent and photocatalyst for the removal of crystal violet (CV) from wastewater. These materials were characterized by using atomic force microscopy for roughness surface and SEM Fig1 for morphology of surface. The energy dispersive X-ray and X-ray diffraction (XRD) indicate the existence of $nanoTiO_2$ on the charcoal surface. Additionally, the FT-IR spectroscopy

measurements illustrate that the alkali treatment develops hydroxyl groups on charcoal surface which could adsorb crystal violet and other pollutants via the synergistic effect. The micro-porosity and BET specific surface was carried out by N_2 adsorption at 77K using an Autosorb-IQ-MP, Quantachrome Instruments. The BET surface of modified charcoal is over 500m²/g support the adsorption process of the crystal violetThe activities of the charcoal (C), activated charcoal (CA) and of the mixture CA with TiO₂ (CA-D) depend on the contact time, dosage of adsorbent and pH. The adsorption kinetic data were tested using pseudo-first-order, pseudo-second-order and intraparticle diffusion models. Kinetic studies showed that the adsorption followed the pseudo-second-order reaction with regard to the intraparticle diffusion rate kinetics.

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III.PO.13

POROUS AIMg-SIC COMPOSITES STRUCTURE MODELING BY MEANS OF FRACTAL ANALYSIS

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Keywords: porous metallic composite, particles, fractal analysis

Abstract: This work is a continuation of the authors research in the field of ultralight metallic composite materials, based on AlMg10 alloy and SiC particles and obtained by salt dissolution method. We used for the fractal analysis the fractal geometry modeling by means of fractal dimension types of composites obtained from performed experiments. We achieved the following fractal dimensions for the samples: 1.37 (for 5% SiC sample), 1.41 (for 10% SiC sample) and 1.45 (for 15% SiC sample). Fractal analysis indicated that all the obtained samples have cells with a statistically regular form. We conclude that this kind of composite materials can be included in ultralights porous metal composite materials, with a tendency to a metal foam structure.

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III.PO.14

THE STUDY OF PHYSICO-CHEMICAL PROPERTIES OF PEROVSKITE TYPE LANTHANUM MANGANITE OBTAINED BY SOL-GEL METHOD HEAT TREATED AT DIFFERENT TEMPERATURES

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Keywords: perovskite, ceramic materials, sol-gel method, thermal treatment

Abstract: In the past few years, one-dimensional nanostructured materials such as nanotubes, nanofibers, nanowires and nanobelts [1], have received great interests due to their superior electrical, thermal, optical, magnetic and catalytic properties [2]. Perovskite LaMnO₃ and related materials are technologically important for many possible applications due to their unique electrical, magnetic, and catalytic properties. Of particular interest are these materials for their catalytic oxidation and reduction reactions associated with the regulation of waste gas emissions [3].

Perovskite type materials based on lanthanum manganite were synthesized by sol-gel method and heat treated at different temperatures. The crystallinity and microstructure of the powders were analyzed using X-ray diffraction (XRD), scanning/transmission electron microscopy (SEM/TEM) and Fourier Transform Infrared Spectroscopy (FTIR). The thermal behavior of the obtained materials was studied by simultaneous TG/DTA thermal analysis at different temperatures in air atmosphere. After achievement of investigations above mentioned, the morphology, particle size, structure and crystallization degree of obtained samples were compared.

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III.PO.15

MICROWAVE MELTING AND PROCESSING OF METALLIC POWDER MMC CASTINGS

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Keywords: microwaves, casting, melting, MMC, nickel

Abstract: In present work, the novel microwave energy has been explored in the melting and casting of metallic based powders [1, 2]. Experiments were carried out in microwave oven operating at 2.45 GHz frequency and 900 W. The melting and casting of nickel based metallic powders (EWAC-1004EN) with varying reinforcements (SiC, WC and Al_2O_3) were successfully processed within minutes of



Fig. 1. Back scattered electron image of Microwave processed Nickel + 10%SiC MMC [1]

microwave exposure. It was observed that addition of carbide reinforcements lowered the exposure times due to better absorption of microwaves. Developed castings were characterized by relevant techniques of XRD, SEM, EDS and hardness study. The microstructures nearlv revealed equiaxed cellular structures due to volumetric heating associated with the process.

The uniform distribution of reinforcements was observed, which may be attributed to the presence of convection currents in the molten pool. Higher hardness was observed in MMC castings; which was due to the formation of some intermetallic carbides and silicides [3].

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III.PO.16

SURFACE CHARACTERIZATION OF THERMALLY TREATED LIGNOCELLULOSIC PANELS

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Keywords: lignocellulosic panels, thermal treatment, FTIR spectroscopy, wear resistance, microscopy

Abstract: This paper presents a comparative study regarding the structural modifications that occur in thermally treated lignocellulosic panels comprised of different wood species, namely spruce softwood, and oak hardwood. Different thermal treatment regimens have been applied, varying the



Fig. 1. Microstructure of lignocellulosic panel

heating temperature between 180°C and 200°C, and respectively, the duration of the thermal treatment between 3 and 5 hours.

The structural modifications that occur in the lignocellulose samples have been assessed by using FTIR spectroscopy, optical microscopy, contact angle and SEM micrograph. It has been determined that the thermal treatment leads to a densification of the overall wood structure, by collapsing of the walls of the vessels (Fig.1), improving the panel hardness, overall thermal resistance and lowering of the water absorption coefficient. By FTIR spectroscopy it has been determined that several structural modifications

occur with cellulose and lignin macromolecules, namely oxidation, that promotes the formation of chromophore carbonyl and carboxyl groups and determining an increase in the surface energy of the samples. Our studies could be useful in determining the optimal thermal treatment regime of the wood panels, having as aim the improving of the panels resistance to outdoor conditions (humidity), without the addition of other compounds in the structure of the wood (resins, additives) [1].

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III.PO.17

CHARACTERIZATION AND OBTAINING OF SILICONE RUBBER REINFORCED WITH MAGNETIC FILLERS USED TO PROTECT THE HUMAN FACTOR

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Keywords: composite materials, silicone rubber, iron silicone (SI), plated nettling, electromagnetic shielding

Abstract: This paper presents results of research on processing, characterization and behavior of composite materials based on silicone rubber used for electromagnetic shielding.



Fig.1. Laying of siloxanic rubber and iron silicon without plated nettling (SSI- gray face); and view of both sides of the composite matrix siloxane rubber and iron powder- silicon

In this work silicone rubbers is reinforced with magnetic fillers (iron silicon). The obtaining of composite materials was with the aid of a doctor blade technique. Doctor blade technique is a coating technique widely used for producing thin films on large area surfaces. The tehnique to introducing the particulate in the polymeric matrix is a simple technology and ecological without environmental impact , contributing to environmental conservation. Recently, there has been

considerable interest in forming filler as a means to improve several properties of elastomers. The samples were processed in the laboratory using a plated nettling (PN), that was impregnated with a silicone rubber based composite containing iron silicon (SI). Measurement of the both parts of the materials obtaining, have shown an electromagnetic shielding effectiveness of the order of 3,35-41,62 dB (white face) and 3,53-41,42 dB (gray face), in the 1-18 GHz frequency range according to SR EN 50 147-1:1999. The character of the original paper, is to provide electromagnetic radiation absorbing material that attenuation characteristics of the electromagnetic field good, as compared to the absorbent materials studied to date. The work is trying to identify an absorbent material with the ability to protect the human being electromagnetics radiations that surrounds us every day.

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III.PO.18

NATURAL RESIN MATERIALS - SPECTRAL AND CHROMATOGRAPHIC INVESTIGATION

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Keywords: coniferous, natural resins, Raman, FTIR, chromatography

Abstract: Natural coniferous resins have been raw materials for various products in industry, and have been used as traditional medicines for skin wounds and infections. General speaking, the resin is a liquid hydrocarbon secretion of many plants, mainly composed of volatile and nonvolatile



Fig. 1. FTIR spectra of natural resins

terpenes and essential oils. Some resins, particularly in conifers, contain a mixture of diterpenic, organic carboxylic acids, called resin acids. The resin can be found in solid or semi-solid state, it is not soluble in water but is soluble in alcohol. By FTIR spectroscopy it is possible to see the resin composition. In figure 1, the C-H stretching vibrations of saturated hydrocarbons are seen below 3000 cm⁻¹, CH₃ and C-H deformations at about 1460 cm⁻¹ and 1380 cm⁻¹. Rocking and wagging of -CH₂- gives a clear peak at 720cm⁻¹. The main peak is that from 1700 cm⁻¹ which corresponds to а carbonyl (C=O) stretching vibration from free carboxylic

acid and from esters. But, these compounds are easier to be identified by Raman spectroscopy. Some extra organic compounds could be found in this wax seal, and this resin material need investigation by GC-MS. In all coniferous resins, alpha and beta pinene constituents are present, a wide range of fragrances, flavours, vitamins and polyterpene products, together with camphor, linalool, citral, citronellol, citronellal and menthol, present, too in these samples.

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III.PO.19

THERMAL BEHAVIOR AND STRUCTURAL PROPERTIES OF POLY (METHYL METHACRYLATE)/TIO₂ NANOCOMPOSITES FOR DENTAL APPLICATIONS

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Keywords: Poly (Methyl Methacrylate) (PMMA), nanoparticles TiO₂, Polymer composite, thermal properties, 3D printing

Abstract: The biocompatible polymer poly (methyl methacrylate) (PMMA) is widely used for complete denture manufacturing. However, important drawbacks related to mechanical resistance, bacterial contamination and thermal perception were recorded. Therefore, the PMMA characteristics could be improved by adding various fillers in the polymeric matrix. The present work was mainly dedicated to the development of a nanocomposite PMMA/TiO₂ for complete dentures 3D



Fig.1. Dental prostehsis 3D printed using PMMA- 0.4% TiO2 nanocomposite (with the support pins attached)

printing. Titania and poly (methyl methacrylate) (PMMA) nanocomposites have been obtained by mixing various amounts of TiO₂ nanoparticles and biocompatible polymer. Such nanocmposites have been characterized through thermogravimetric analysis (TGA) and Fourier transform infrared (FTIR) spectroscopy. The investigations performed proved that the PMMA – TiO₂ nanocomposites presented an improved thermal stability compared to the polymer itself. Nevertheless, the differences recorded for T_g (the glass transition temperature) of the PMMA polymer and nanocomposites have not been significantly. The FT-IR analysis put in evidence the presence of a specific vibrational frequencies at 810 cm⁻¹ in FTIR spectra which would show the presence of Ti-O-Ti interaction, proving an interaction between PMMA and TiO₂. The nanocomposite with 0.4% nanoTiO₂ in PMMA presented the most suitable thermal behavior sustained by the structural

characterization for a compound to be further used for stereolithographic technique to manufacture 3D printed complete dentures – Fig.1.

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III.PO.20

IRON(II)-EMBEDDED COMPOSITES BASED ON ACTIVATED CARBON USED AS FENTON-LIKE CATALYSTS

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Keywords: photocatalysis, composites, Fenton

Abstract: The aim of this work is to investigate the efficiency of a cheap Iron-impregnated activated carbon Fenton like catalyst towards the photo-degradation of organic compounds in aqueous solutions. Indigo Carmine (IC) is considered as model pollutant (1). Different Advanced Oxidation



Fig. 1. Degradation of Indigo Carmine by different AOPs

Processes (AOPs) are evaluated for the degradation of 100 IC (Fig.1). AC was directly contacted with FeSO4 solution using wet impregnation method (2). Thermogravimetric analysis, FTIR, X-ray diffraction, and transmission electron microscopy were employed to investigate the structural, textural, and micromorphology of the catalyst. UV light irradiation tests were carried out to determine the performance of the prepared Iron-impregnated composite towards the degradation of IC in aqueous solution using different conditions (17 watts UV lamps, with and without in-situ

generation of O_3 ; different concentrations of H_2O_2 , different initial concentrations of IC, different values of pH, different doses of NH_4 -OH enhancer). These tests were conducted after the adsorption equilibrium has been established. The obtained results emphasize an enhancement of IC degradation in case of the heterogeneous photo-Fenton process conducted with an O_3 generating UV lamp in the presence of ammonium hydroxide.

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III.PO.21

EFFECT OF DOPANT CONCENTRATION ON THE STRUCTURAL, MORPHOLOGICAL AND OPTICAL PROPERTIES OF NaNbO₃ NANOPARTICLES

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Keywords: ceramics, hydrothermal method, sodium zirconium niobate, lead-free

Abstract: Piezoelectric materials are commonly used in sensor and actuator technologies due to their unique ability to couple electrical and mechanical displacements, i.e., to change electrical polarization in response to an applied mechanical stress or mechanically strain in response to an applied electric field [1]. The research on lead-free piezoelectric ceramics as a possible alternative to widely exploited lead-based complex perovskites such as Pb (Zr,Ti)O₃ (PZT) and Pb(Mg_{0.33}Nb_{0.67})O₃-PbTiO₃ (PMN-PT)-based materials has been stimulated by the increased consciousness for health and environment protection. The group of materials based on $K_{0.5}Na_{0.5}NbO_3$ (KNN) has been intensively studied especially since the discovery of the compositions modified with lithium and tantalum with piezoelectric properties comparable to those of commercial PZT materials [2, 3].

In this study we report obtaining of Zr doped NaNbO₃ powders by the hydrothermal method at 200°C, for 12 hours. The morpho-structural and optical properties study for materials obtained was performed by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), UV-Vis spectroscopy and Scanning electron microscopy (SEM). The X-ray diffraction indicates that all samples have orthorhombic structure with a space group P n n m (58). From SEM images of the Zr doped NaNbO3 powders (1, 2 and 3%) is can see as the morphology of the particles it's changing with the dopant concentration, so: for Zr (1%) the particles have cubic shape, for Zr (2%) have wires form, and for Zr (3%) are in the form of bars.

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III.PO.22

MINERAL INVESTIGATIONS OF ROMANIAN WHEAT AND FLOUR

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Keywords: kernel, flour, mineral determination, ICP-AES.

Abstract: The wheat is one of the most consumed cereals by humans. The flour is used for the preparation of breads, cookies and other bakery products. For this reason, many papers have been

Mg	1402.36	Mg	1070.36
Ca	414.41	Ca	305.44
Na	263.10	Na	371.11
к	6426.57	К	5166.67
Fe	92.17	Fe	65.16
Mn	33.95	Mn	36.36
Zn	33.83	Zn	28.18
Cu	4.76	Cu	5.35

Fig. 1. The concentrations of the main elements from wheat and flour (mg.kg⁻¹)

written, treating the characterization and nutritional evaluation of this cereal and its flour. The kernel is the seed from which the wheat plant grows. Each seed contains three distinct parts that are separated during the milling process to produce flour. Na, K, Ca, Mg, Fe, Zn, Mn, Cu were determined with an inductively coupled plasma atomic emission spectrometer (ICP-AES).

Digestion was achieved by using a Berghoff digestor using 5ml HNO₃ and 2 ml H_2O_2 , provided by Merck. The materials used for each digestion, as well as the volumetric flask are 0.2 g. The calibration was achieved with a

standard solution (Standard ICP-AES multielement standard solution IV, Merck, 1000 ppm). The calibration curve was made with diluted solutions from the above multielement. The solutions for calibration were prepared using bidistillated water. The method validation (including digestion and determination) was performed using a certified reference material of wheat flour furnished by National Institute of Standards & Technology (NIST). The elements that have been identified on the wheat and flour have essentially the same concentrations in regular flour and wheat, Fig. 1.



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III.PO.23

FLY ASH CHARACTERIZATION FROM CET GOVORA USED IN MASONRY MORTARS

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Keywords: fly ashes, mortars, spherical particles

Abstract: For environmental protection, the fly ashes produced by coal burning from the power plant are separated by advanced filtration. [1]. Until the present, only a small fraction from the resulted ashes are exploited in different applications, concretes for constructions, construction materials [2],



Fig. 1. Fly ash

concrete road [3] etc. In this paper are presented the results from structural, micro structural and thermal characterizations of the fly ashes from CET Govora, used as addition in masonry mortars. The ashes from power plant were collected from CET Govora from the 3 and 4 fields belonging to electro filter. The fraction of ashes collected from electrostatic fields was followed by a gravimetric separation process. The mineralogical composition of the fly ash was characterized by X-ray diffraction (XRD). The chemical composition of fly ash was determined by XRF analysis and Raman

spectroscopy. The microstructure of the power plant ash was analyzed using a scanning electron microscope (SEM) and thermal analysis was performed by thermogravimetric analysis (TG-DSC). The fly ash which was investigated is composed of the spherical particles ($0,5\mu$ m - 50μ m) predominantly having a diameter of up to 5 mm (Fig.1).

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III.PO.24

ANALYZING THE BALLISTIC IMPACT PERFORMANCE OF LAMINATED COMPOSITES

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Keywords: ballistic impact, composite materials, damage

Abstract: Composite materials having superior properties must be used in engineering applications requiring advanced technologies such as automotive, aerospace, aircraft, gas turbines and especially defense industry. It is important to have renewable designs for machine and structural elements to have better resistance to penetration and puncture since these elements are subjected to dynamic loading especially due to high speed ballistic impact [1,3].

In recent years, scientists and engineers have considerable interest with an increasing trend in the potential engineering application of layered composite materials having high strength to low density ratio, low thermal expansion coefficient and high ballistic resistance. Since the materials used in these advanced technologies usually subjected to dynamic loads such as ballistic impact, their deformation and failure characteristics are quite different than the ones subjected to static loadings. For these reasons, in order to design composite materials for ballistic impact resistance, their high speed ballistic deformation mechanisms have to be well understood [4-6]. Therefore, in this paper finite element model is established with using micromechanics based material and cohesive element models to simulate the ballistic response of layered composite materials. In this respect, ballistic limit, perforation, penetration, and delamination deformation as well as energy absorption capacity of the target material are determined.

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III.PO.25

THERMAL AND THERMORHEOLOGIC CHARACTERIZATION OF DIFFERENT POLYOLEFIN WASTE FRACTIONS

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Keywords: polyolefins, melt flow index, density

Abstract: The design of polymer processing equipment, polymer processing optimization and trouble-shooting is dependent on the knowledge of the complete flow curve or rheogram of a polymeric melt depicting the variation of the melt viscosity over industrially relevant range of shear rate and temperature [1,2]. This is particularly useful for polymer wastes, where the presence of additives and possible small-molecular degradation byproducts could change their procesabillity in a significant manner. However, these data are usually obtained by expensive and time consuming rheometric method. Melt flow index (MFI) represents a useful and readily available parameter for plastics procesabillity characterization. By determining the values of MFI for different testing loads (different shear rates), the thermorheological behavior of the polymer could be estimated, using the glass transition values determined by differential scanning calorimetry [2].

In this study, MFI values from several fractions containing mainly polypropylene, low-density polyethylenes and high-density polyethylenes, coming from household wastes recycling (provided by Rom Waste Solutions S.A., Bucharest) were measured at 190 °C for polyethylene and 230 °C for polypropylene [3]. Higher values of MFI (low shear viscosities) have been reported, in comparison with the corresponding virgin plastics, probably due to the lower molecular mass of the polymer waste and/or the presence of surfactant compounds on the surface of the polymer flakes. Also, by extruding the same batch in different cycles at the same temperature values, the number of processing cycles on which the polymer could be recycled has been determined.

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SECTION IV Surface Engineering

Chairpersons:

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IV.K.1

PULSED LASER DEPOSITION OF THIN FILMS: A VERSATILE TECHNIQUE FOR STRUCTURE-COMPOSITION-PROPERTY STUDIES

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Keywords: thin films, pulsed laser deposition, protective coatings, transparent and conductive oxide

Abstract: The pulsed laser deposition (PLD) is probably the most versatile laboratory technique to grow thin films to investigate the interdependence between structure, composition and properties. Simple changes of the deposition conditions (substrate temperature, nature and pressure of the gaseous atmosphere, laser fluence, repetition rate, laser wavelength and deposition geometry) will result in the growth from a single target of films having a rather wide range of compositions, crystalline grain sizes, textures, stress levels and properties.

These advantages of the PLD technique will be illustrated with examples of nanostructured (SiC, ZrC and ZrN [1, 2, 3) or amorphous (indium zinc oxide) films grown by PLD and used to investigate the effect of radiations on their structure, composition and properties. The second example is the deposition on Ti surfaces of various coatings to improve the mechanical, chemical and biological properties of the surface [4]. We also deposited mixtures of compounds by a combinatorial pulsed laser deposition technique to reduce the grain size and obtain amorphous protective coatings.

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IV.K.2

PRODUCTION OF ZIRCONIUM OXYNITRIDE THIN FILMS BY MAGNETRON SPUTTERING: THE QUEST OF BREAKING THE CHEMICAL COMPOSITION AND COLOUR RESTRICTIONS

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Keywords: colour, decorative, sputtering, RBS, XRD

Abstract: Zirconium oxynitride coatings are very interesting for decorative/aesthetic purposes. They combine chemical stability, biocompatibility, good mechanical properties and a nice palette of achievable colours.

The preparation of these coatings by reactive magnetron sputtering is relatively simple, however, it has been very difficult to obtain some particular colours. The chemical composition of the obtained samples is located in a very particular region of the Zr-O-N ternary diagram. Probably this is one of the main reasons behind the limitations for the observed colours.

In this work, we will make a study of the Zr-O-N system, including an exhaustive comparison with literature. The approach is the exploration of the the deposition parameter space, by changing target power, bias and composition of the sputtering atmosphere. The chemical and phase composition and structure of the films are evaluated by RBS, XRD and SEM, respectively, and correlated with the synthesis conditions. Finally, the colour of the coatings is measured by spectrophotometry and interpreted in terms of the characteristics of the films.

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IV.O.1

MULTI COMPOSITE PROTECTIVE LAYERS FOR GEOTHERMAL CONDITIONS

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Keywords: thermal spray plasma deposition, geothermal turbines, SEM & EDX analyses.

Abstract: The subject of the present work is the development of a knowledge-based platform for the optimization of multi-composite technology and thermal spray deposition processes [1,2], in terms of accuracy, surface finishing, productivity and cost performance, in the purpose of upgrading and



Fig.1. View of plasma gun spraying in work position.

extending the geothermal turbine life cycle in superheated geothermal steam conditions. Geothermal steam contains H₂S and CO₂ and various solid particles resulting in turbine surface damage and scale deposits. Aggressive corrosion in steam geothermal conditions may be defined as an accelerated corrosion. Multi-composite materials applied as protective layers [3] are the actual option for corrective action against corrosion and have good reliability in geothermal conditions. The experimental procedure involves composite characterization and detailed information about surfaces, textures and

morphological modification of 23Ni20Co2.5Cr4Al0.5Y with 5%WC hard particles layer thermal plasma coating deposition. The results obtained are investigated by electron microscopy SEM with EDX and micro-mechanical analyses.

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IV.O.2

INFLUENCE OF THE SURFACE MANUFACTURING ON RUNNING-IN AND DURABILITY OF ROLLING MICRO-CONTACTS

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Keywords: surface manufacturing, running-in, plastic displacement, durability of micro-contacts

Abstract: The topography of the active surfaces in rolling contact depends on the surface manufacturing process technology (finishing, polishing, lapping, etc.) [1,2]. In fig. 1 is presented a specific topography similar to that obtained by the polishing process, where the average height of



Fig. 1. Simulated surface roughness [1,2]

roughness peaks R_a does not exceed the value of 0.25 μ m. The aim of this paper is to observe the influence of the surface roughness obtained by different surface manufacturing processes on running-in, surface geometry and durability of rolling micro-contacts. The simulated rough surfaces were introduced in a computer code as initial data, code developed in the framework of the plasticity model presented in [3] and durability of rolling micro-contact evaluated was by methodology described in [4]. To validate the obtained results the surface topography was measured with Taylor Hobson profilometer, was used AMSLER machine and an experimental setup with proper samples for rolling motion.

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IV.O.3

RELATIVE HUMIDITY EFFECT ON THE ADHESION ENERGY IN MEMS

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Keywords: Relative humidity, AFM, adhesion, pull-off force, capillary

Abstract: The adhesion energy was measured between atomic force microscope AFM probe and different size cantilevers from gold (fig. 1) in various relative humidities. The adhesive effect between AFM probe (Si_3N_4) and sample was evaluated using the force spectroscopy mode of AFM so-called force/distance measurements, in a clean environment (clean room). This type of measurements allows deducing the force of adhesion between nanoscale rough surfaces of microcantilever. This measurement provides correct information about surface energy and adhesion only if the contact between AFM tip and cantilever surface remains in the elastic range. Literature indicates that at 20°C



Fig. 1. Gold cantilevers samples

and 35%RH, there is no significant change in adhesion force when the load capacity is about 20nN to 250nN. Consequently, adhesion force measurements in this work were typically carried out in the low load regime (50nN) when the contact is elastic and when no material transfer occurred at these loads. The results showed that average pull-off force increased with relative humidity on size of cantilever. Tests showed that the pull-off force on one asperity was the same, but the number of contacting points between the flat probe and AFM probe increased with relative humidity. In this sense it is important to examine the relationship between adhesion strength and

surface topography. A part of the adhesion force is caused by surface tension of adsorbed water [1]. It is therefore important to clarify the effect of relative humidity on the adhesion force. It was obtained experimentally a increase of adhesion force with 20% - 25% when the humidity increases from 5% to 90%RH. Also we have obtained an increase in the adhesion while increasing the length of cantilevers. All the experimental data collected was imported into the XEI analysis software. The pull-in and pull-off forces value are directly provided by the software.

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IV.O.4

TRIBO-MECHANICAL CHARACTERIZATION OF NIOBIUM NITRIDE THIN FILMS DEPOSITED BY DC MAGNETRON SPUTTERING

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Keywords: niobium nitride, DC magnetron sputtering, atomic force microscopy, XRD analysis, nanocharacterization

Abstract: Niobium nitride is used in applications like microelectronics [1], diffusion barrier [2], and



nanoindentation

microelectromechanical systems (MEMS) (sensors) [3] and so on due to its chemical, mechanical, electrical and tribological characteristics. This paper aims at depositing niobium nitride thin films usable as diffusion barriers for manufacturing MEMS applications and to characterize them from the topographical, structural, mechanical and/or tribological point of view at nanoscale. The deposition was realized by direct current (DC) magnetron sputtering on silicon substrate. Different temperatures and nitrogen flows rates

were employed. The so-obtained films were mainly investigated by atomic force microscopy, nanoindentation (Fig. 1) and X-ray diffraction. The results pointed out an important influence of the deposition parameters on the determined properties at nanoscale. The change in properties is mainly due to the different growth of the films after certain preferential orientations.

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IV.O.5

COATING OF LIQUID WOOD SHEETS

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Keywords: liquid wood, thermal resistance, superficial coatings

Abstract: Liquid wood is one of the newly discovered green polymer materials, being a biodegradable and recyclable thermoplastic, made from cellulose, lignin and natural additives, currently used in commerce as ARBOFIL, ARBOROM and ARBOBLEND. The applications of this material (aeronautics, automotive, construction, consumer goods etc.) are limited by two major drawbacks: higher weight compared to ordinary plastic materials and low melting point (about 175 ° C).

In this work was studied the possibility of changing the temperature resistance of this material by applying superficial coatings. For this process were prepared specimens from two types of material: ARBOBLEND and ARBOFIL. The resulted samples were analyzed in terms of microstructural changes both longitudinally (in order to evaluate the quality of the deposited layer) and on cross section (in order to evaluate the changes produced at the interface substrate - coating). For this purpose were used the QUANTA 200 3D (FEI, Holland, 2009) scanning electron microscope and the X`PERT PROMD diffractometer (Panalitycal, Holland, 2009). Using the UMTR 2M-CTR Micro-tribometer were evaluated the elasticity modulus of the coatings through microindentation and its adhesion to the substrate through scratch method, applied both longitudinally and on cross section.

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IV.O.6

THE REACTION TO CORROSION IN STEAM OF A GEOTHERMAL POWER PLANT TURBINE BLADE

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Keywords: turbine blade, corrosion, cavitation, microstructure

Abstract: This paper presents the corrosion behavior in geothermal steam environment for a turbine



blade from Hellisadi geothermal power plant in Iceland.

The structural investigations were developed on two directions. The samples were examined for the macroscopic analysis using the stereo microscope Olympus SRZ. The quantitative and qualitative analyzes were realized using metallographic optical microscope and electric microscope.

During the research, there were identified the metallographic constituents, the blade's chemical composition, and the main fractographic aspects

of the corroded surface.

This case study allowed the main corrosion phenomena identification present during turbine blade operation and defining its mechanisms.

Finally a correlation was realized between the chemical composition, size cavity dips and operating conditions of the turbine.

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IV.O.7

INVESTIGATION OF DOUBLE-LAYERED RARE EARTH ZIRCONATES FOR THERMAL BARRIER COATINGS (TBCS) APPLICATIONS UNDER ISOTHERMAL OXIDATION CONDITIONS

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Keywords: Thermal barrier coatings (TBCs), Oxidation, Thermally grown oxide (TGO), Electron beamphysical vapor deposition (EB-PVD), Cold gas dynamic spray (CGDS), CoNiCrAIY, Rare earth zirconates

Abstract: Oxidation is an inevitable failure mechanism under the operation temperature in gas turbines. To avoid negative effects of oxidation, ceramic based materials having low thermal conductivity and high stability should be used to hot section components [1]. In accordance with this



Fig. 1. YSZ/Gd₂Zr₂O₇ TBC after oxidation

purpose, thermal barrier coatings (TBCs) are used in order to increase lifetime of gas turbine engine components which have not reached to desired levels yet [2]. Yttria Stabilized Zirconia (YSZ) has been used as conventional top coat material in TBCs. Increased turbine inlet temperatures promotes to researchers to try higher stable material such as rare earth zirconates [3]. In the present study, CoNiCrAIY metallic powders were sprayed using new emerging technique as called cold gas dynamic spray (CGDS) on Inconel 718 substrates. $Gd_2Zr_2O_7$ and $Gd_2Zr_2O_7/YSZ$ were

deposited by Electron Beam Physical Vapor Deposition (EB-PVD) technique as top coat materials. In high temperature furnace, both TBC samples were isothermally oxidized at 1000 °C under different time periods. TBCs were examined as microstructural before and after oxidation tests. Thermally grown oxide (TGO) layer forming at the interface during oxidation were investigated and compared for each TBC systems. Oxidation and TGO growth behaviors were discussed.

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IV.O.8

ULTRA-STABLE AQUEOUS SUSPENSIONS OF FEW-LAYER GRAPHENE ENCAPSULATED IRON NANOPARTICLES

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Keywords: few-layer graphene, magnetic nanoparticles, suspensions, ball-milling

Abstract: Few-layer graphene encapsulated iron nanoparticles (GEINs) are core-shell nanomaterials which are built of a magnetic core and protective coating. The few-layer graphene coating perfectly protects and isolates the encapsulated magnetic phase from the environment [1]. GEINs have unique properties and can be used in various biomedical applications, e.g. in construction of selective molecular imaging agents [2]. The surface of GEINs is highly hydrophobic and therefore the stability of GEIN aqueous suspensions is very poor. The objective of this study was to enhance the stability of aqueous suspensions of few-layer graphene encapsulated iron nanoparticles. Generally, the water suspension of various (nano)carbon materials are stabilized with the aid of desired surfactants, e.g. SDS, SDBS and polymers [3]. Initially, we have studied the sedimentation of GEINs suspended in water (100 μ g/mL) stabilized with polyvinylpyrrolidone (PVP). The suspension was prepared with the aid of ultrasonic treatment (80 W, 20 minutes). The suspension was not stable and after 15 minutes started to sediment. After 12 h the whole dispersed particles were on the bottom of the vial (see the



inset in Figure 1). Therefore, another approach has been tested, i.e. ball milling of GEINs/PVP mixtures (1/10 m/m) for 6, 24 and 48 h. The ball milled GEINs/PVP mixture is readily dispersible in water and no ultrasonic treatment is needed. The sedimentation stability was measured by turbidimetry using a 532 nm light source. The transmitted light was probed in the time interval of 1 minute. Figure 1 shows the sedimentation kinetics curves for obtained GEINs/PVP mixtures. The samples are ultra-stable, because more than 95% initially dispersed particles are present in the suspension after 12 h of sedimentation. The DLS studies revealed that the mean hydrodynamic size does not markedly change after 12 h of sedimentation.

The presented method is simple and can be used for other carbon nanomaterials.

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IV.O.9

OPTICAL, ELECTRICAL AND STRUCTURAL PROPERTIES OF SIC THIN FILMS DEPOSITED BY REACTIVE DC MAGNETRON SPUTTERING

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Keywords: silicon carbide, thin film, reactive DC magnetron sputtering, band gap, optical constants.

Abstract: Silicon carbide is a semiconductor that has great physical properties such as the wide band gap, high breakdown voltage, high thermal conductivity, high electron drift velocity, high surface hardness, high bulk plasticity and low density [1-3]. Also at high temperatures, silicon carbide shows high chemical inertness and excellent abrasion and radiation resistance [4,5]. These superior physical, chemical and mechanical properties makes silicon carbide ideal for many applications thus attracted a lot of scientific interest on various branches of technology in recent years [1-5].

In this study, silicon carbide thin films of variable compositions were deposited on glass and Si (100) substrates by reactive DC magnetron sputtering of high purity silicon target, using CH_4 as reactive gas. The composition and the properties of the coatings have been modified by the change in the reactive gas flow ratio from 5% to 50%. Spectrophotometer has been used to measure the optical transmittance and reflectance of silicon carbide thin films over the spectral range from 280 to 1000 nm. Tauc method was used to calculate the band gap values of SiC thin films with respect to the gas flow rate. Activation energy and room temperature conductivity values of the deposited films were also evaluated. Microstructural analyses of SiC films were realized on their cross-sections by FE-SEM observations. The results demonstrated that the transparency and opacity of SiC films could easily be tailored by modifying Si and C concentrations in the coating composition, for the same film thicknesses.

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IV.O.10

ISOTHERMAL OXIDATION AND THERMAL CYCLIC BEHAVIORS OF YSZ AND YSZ/La₂Zr₂O₇ DOUBLE-LAYERED THERMAL BARRIER COATINGS (TBCs)

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Keywords: La₂Zr₂O₇, thermal barrier coatings (TBCs), oxidation, thermal cycling, EB-PVD

Abstract: Thermal barrier coatings (TBCs) provide protection of super alloy substrates against harsh environments during service conditions in gas turbine engines [1]. A typical TBC consists of two main coating layer as called bond and top coat. MCrAIY is widely used as bond coat materials while YSZ is



Fig. 1. YSZ/La₂Zr₂O₇ TBC

conventionally deposited as top coat materials in TBCs [2]. Zirconates have attractive top coat material candidates due to their superior thermal properties instead of YSZ [3]. In this study, YSZ and La₂Zr₂O₇/YSZ as new promising material were deposited using Electron Beam Physical Vapor Deposition (EB-PVD) technique on CoNiCrAlY bond coats produced by High Velocity Oxygen Fuel (HVOF) technique. TBCs were exposed to isothermal oxidation tests in high temperature furnace at 1000 °C with different time periods and also, they were

subjected to furnace cycling tests at 1150 °C. After oxidation and thermal cycling tests, formation of thermally grown oxide (TGO) layers at the interface, crack surfaces and failure mechanism were investigated according to analysis results.

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IV.O.11

HOT CORROSION BEHAVIOR OF YSZ AND GD₂ZR₂O₇ THERMAL BARRIER COATINGS (TBCS) EXPOSED TO MOLTEN SULFATE AND VANADATE SALT

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Keywords: Thermal barrier coatings (TBCs), Hot corrosion, YSZ, Gd₂Zr₂O₇, Molten salt

Abstract: Thermal barrier coatings (TBCs) are mostly used on critical part components in aircraft's turbines inlet. Aviation fuels mostly have impurities such as vanadium oxide (V_2O_5) and sodium sulfate (Na_2SO_4) [1]. These impurities damage on turbines inlet at elevated temperatures because of



Fig. 1. EB-PVD TBCs with Cold Sprayed CoNiCrAlY Bond Coat's surface image After Hot Corrosion test chemical reaction [2]. Yttria stabilized zirconia (YSZ) is conventional top coat material for TBCs while $Gd_2Zr_2O_7$ is a new promising top coat material for TBCs [3]. In this study, CoNiCrAlY metallic bond coat was deposited on superalloy substrate material with a thickness about 100 µm using Cold Gas Dynamic Spray (CGDS) method. Production of TBCs were done with deposition of YSZ and $Gd_2Zr_2O_7$ ceramic top coating materials using EB-PVD method, having a total thickness of 300 µm. Hot corrosion behavior of YSZ and $Gd_2Zr_2O_7$ TBC systems were exposed to 45 wt.% Na₂SO₄ and 55 wt.% V₂O₅

molten salt mixtures at 1000°C temperature. Using scanning electron microscope (SEM), energy dispersive spectroscopy (EDS) analysis and X-ray diffractometer (XRD) TBC samples are investigated and compared. The hot corrosion failure mechanisms of YSZ and $Gd_2Zr_2O_7$ TBC in the molten salts were evaluated.

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IV.O.12

SUBSTRATE INFLUENCE ON THE TRIBOLOGICAL BEHAVIOUR OF DLC COATINGS WITH CRN INTERLAYER DEPOSITED ON HARDENED STEEL

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Keywords: DLC layer, PVD, tribology

Abstract: The purpose of this study was to evaluate the influence of substrate material type and processing parameters on the tribological behavior of DLC (diamond-like coatings). CrN interlayers and DLC top layer thin films were produced on hardened AISI 52100 bearing steel and AISI 5115 case hardening steel, by a magnetron sputtering PVD method. The PVD deposition was performed at three different temperatures: 180 °C, 200 °C and 250 °C. The present DLC was a typical DLC called as hydrogenated amorphous carbon (a-C: H). The hydrogen content was about 20 %. The chemical composition of the samples was assessed by RBS, the structure by XRD, and the surface morphology by AFM. The assessment of the way in which DLC layers behaved to different types of mechanical



Fig. 1. Friction coefficient as function of distance

stresses has been performed onto multiple directions: for the tribological characterization, a pin-on-disk rotational tribometer has been used; the adhesion to the substrate has been determined by micro scratch testing method; the thickness of the DLC film has been assessed by ball-cratering; hardness and the modulus of elasticity were obtained by nanoindentation. A strong correlation between the type of substrate material, but more importantly, of the deposition temperature, on one hand, and the mechanical characteristics, on the other hand, has been observed.

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IV.O.13

EFFECT OF PROCESS PARAMETERS ON THE TRIBOLOGICAL PROPERTIES OF PORCELAIN WASTE REINFORCED EPOXY COMPOSITES

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Keywords: Wear, epoxy composite, porcelain waste, friction

Abstract: In order to reduce resin cost and improve tribological properties porcelain waste added epoxy matrix composites were developed in different filler content and process conditions. Tribological behavior of these composites was evaluated in terms of friction, wear rates and wear mechanisms by ball-on-disk contact with dry and rotational sliding at room temperature against 6Co/WC ball with a diameter of 3mm. All wear tests were carried out at room temperature ($23-25^{\circ}C$) in air with a relative humidity of $40 \pm 5\%$ under dry-sliding conditions. The contact radius of cycles was set to 5 mm at 600 revolutions per minute (rpm) which corresponds to linear speed of 30cm/s for the geometry used in this study. In all the experiments, 3N of constant test load was applied at a frequency of 8 Hz and prolonged to 400m wear distance. During the tests, the friction coefficient of samples was continuously recorded online by the variation in the tangential force. The steady-state CoFs were changed in between 0,29-0,37. The dimensions of the wear tracks (depth and width) were measured as two-dimensional (2D) profiles by a stylus surface profilometer. The profilometric analyses, the wear volumes were calculated by integrating these 2D surface areas over the diameter. The microstructure, mechanical and physical properties and wear behavior correlation will be discussed in detail.







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IV.O.14

INDUCED SUPERHYDROPHOBIC AND ANTIMICROBIAL CHARACTER OF ZINC METAL MODIFIED CERAMIC WALL TILE SURFACES

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Keywords: Superhydrophobic ceramic tile surface, contact angle, antimicrobial surface, antimicrobial characterization, ceramic tile glaze

Abstract: Hydrophobic surfaces are also known to have antimicrobial effect by restricting the adherence of microorganisms on the surface. However, ceramic products are produced by high temperature processes resulting in a hydrophilic surface. In this study, an industrial ceramic wall tile glaze composition was modified by the inclusion of metallic zinc powder in the glaze suspension applied on the pre-sintered wall tile bodies with air spray gun. The glazed tiles were fired at peak temperatures ranging from 980°C to 1100°C for 5 and 30 minutes. The fired tile surfaces were coated with a commercial composition of 10% fluoropolymer, 60% alkoxysilane and 30% ethanol (ECC-4000) in order to avoid water absorption, and enhance the hydrophobic character. The surface hydrophobicity and the antimicrobial activity results were compared with that of unmodified uncoated gloss fired wall tiles. The surfaces were examined with SEM, XRD and the water contact angles were measured with a goniometer. The antimicrobial activity tests of the surfaces were conducted according to ASTM E2180-07 (Standard Test Method for Determining the Activity of Incorporated Antimicrobial Agent in Polymeric or Hydrophobic Materials).

For the tiles fired at 1000°C for 5 min, the surface contact angle of <30° of the unmodified, uncoated wall tile changed to 70° of the unmodified but coated tile, and to the superhydrophobic 145° of the Zn metal modified and coated tile heat treated at 1000°C. The antimicrobial tests indicated that the bacterial activity (cfu/mL) with *Staphylococcus aureus* of the unmodified but coated surface increased 15.5 folds while of the Zn modified and coated surface decreased by 99.68%, and with *Pseudomonas aeruginosa* of the unmodified but coated surface increased 1.17 folds while of the Zn modified and coated surface decreased by 86.7% as compared to the unmodified, uncoated surfaces. The increase of the hydrophobicity and the decrease in the antimicrobial activity of the Zn metal powder modified glaze compositions were attributed to the formation of willemite (2ZnO.SiO₂) crystals generating a nano-patterned surface topography as detected by XRD and SEM-EDX.



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IV.PO.1

SINTERING UNDER CONCENTRATED SOLAR RADIATION OF PEROVSKITE CERAMICS FOR ELECTRONIC APPLICATIONS

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Keywords: Concentrated Solar Energy, Horizontal Solar Furnace, Perovskite Ceramics

Abstract: Among all renewable energy sources such as wind, geothermal, tidal, solar energy has undeniable advantages: it is clean, free, safe, omnipresent and abundant. The core objective of our research is to sinter CaTiO₃, SrTiO₃, BaTiO₃ perovskite ceramics in horizontal solar furnace using



Fig. 1. Horizontal solar furnace at PSA

concentrated solar energy, which is an ecological, novel and original process. We performed the solar sintering of CaTiO₃, SrTiO₃, BaTiO₃ perovskite ceramics at *Plataforma Solar de Almeria*, Spain. This process takes place in a horizontal solar furnace (SF40-40kW) [Fig.1], constituted by a flat heliostat, a parabolic concentrator, a shutter and a test table. The working principle of SF40 is double reflections : the heliostat reflects the horizontal and parallel rays from the sun on the parabolic concentrator, which again reflects and concentrates them in the focus of the parabola [1,2]. We placed the pellets of CaTiO₃, SrTiO₃,

BaTiO₃ ceramics on zirconia support and solar sintered them in the temperature range of 1000-1100°C, between 2-60 minutes in air. Perovskite ceramic samples solar sintered in SF40 horizontal solar furnace at 1100°C for 1 hour, resulted in well-sintered, non-porous samples with good density : $3.85 \text{ g/cm}^3(\text{CaTiO}_3)$, $4.69 \text{ g/cm}^3(\text{SrTiO}_3)$, $5.46 \text{ g/cm}^3(\text{BaTiO}_3)$. These sintered samples were analyzed by XRD for phases determination and ferroelectric measurements as remanent polarization (Pr) and coercitive field (Ec) were carried out. The XRD patterns show that was obtained single phases of SrTiO_3 and BaTiO_3 respectively. In case of BaTiO_3 was obtained $\text{Pr=6.317} \,\mu\text{C/cm}^2$ and $\text{Ec=1.947} \,\text{kV/cm}$. The effect of processing parameters on microstructure and dielectric properties were investigated and will be presented.

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IV.PO.2

NANOCOMPOSITES THIN LAYERS FOR DENTAL IMPLANTS FUNCTIONALIZATION, OBTAINED BY ADVANCED LASER DEPOSITION TECHNIQUES

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Keywords: nanocomposites thin layers, dental implants, laser deposition techniques

Abstract: This work goal is the smart functionalization of dental implants by their covering with nanocomposites layers with complementary properties, in order to remove the problems that arise when introduce such an implant in the human body. We report on the transfer of novel polymerantibiotic-bioactive glass composites by matrix-assisted pulsed laser evaporation to uniform thin layers onto stainless steel implant surfaces. The targets were prepared by freezing in liquid nitrogen of mixtures containing polymer and antibiotic reinforced with bioglass powders. The cryogenic targets were submitted to multipulse ablation with an UV KrF* (λ =248 nm, $\tau \sim$ 25 ns) excimer laser source. Glow discharge optical emmision spectroscopy analyses, FTIR and UV-VIS analyses of surface emphasise the multiple advantages of these coatings which would allow to halt any leakage of metal and metal oxides to the biological fluids and eventually to inner organs, to speed up the osseointegration , to exert antimicrobial effects and possible to decrease the implant price.

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IV.PO.3

ADHESION CHARACTERISATION OF COMPLEX CERAMICS THIN LAYERS DEPOSITED ON METALLIC SUBSTRATE

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Keywords: ceramic complex, plasma spraying, SEM, EDS

Abstract: There was obtained superficial layer of ceramic (made of complex oxides 80% yttrium oxide stabilized zirconium $ZrO_2/8\%Y2O_3$ (Metco 204B-NS) and 20% alumina Al_2O_3 (Metco 105SFP)) by plasma jet deposition at a temperature of 12000°C of particles, on substrates of iron FC250. The layers were obtained by five successive passages (60 µm thick) on samples with different surface roughness and different processed (0.34, 2.47 și 4.25 µm). For the analysis of the adhesion of ceramic layers to the substrate scratch tests were carried out and the traces analyzed by scanning electron microscopy (SEM 2D and 3D), chemical analysis EDS, and profilometry.

In conclusion, it was obtained compact layers on the samples 2 and 3 with micro-cracks on the surface due to thermal gradient that occurs between the layers deposited during the five passes.

Regarding the resistance, the sample with the higher roughness (sample 3) resisted most to exfoliation the layer (15 N) in contrast to the sample 2 of 14N and only 10N for the polished sample.

It can be concluded that there is a dependency between the surface roughness and the thickness of layer deposited. Scratch marks presents no cracks, pores or adjacent exfoliation. The results show that the technique is suitable for obtaining thin layers of ceramic materials with a very good control of the thickness, very good adhesion to the substrate and a direct relationship between surface roughness and the quality of deposited layer.

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IV.PO.4

COMPARATIVE STUDY OF THE ADHESION OF TI-ME (ME = AL, CU, AG, AU) THIN FILMS ON POLYMERIC SUBSTRATES, AIMING BIOMEDICAL APPLICATIONS

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Keywords: Ti-Me thin films, adhesion, biomedical applications

Abstract: Recent investigations in sensors for biomedical devices showed the advantages in the use

of polymeric substrates, coated with conductive and biocompatible thin films such as titanium as bio-interfaces. Following the same research line, for the development of new thin film systems with improved capabilities, titanium-based thin films doped with different metals, Me, were produced [1-2]. When combined with other metals, like Al, Cu, Ag or Au important properties may result, such as low densities, excellent oxidation resistance, high strength and high stiffness, which are a direct result of the formation of intermetallic compounds [1-2].

Ti-Me intermetallic thin films, with different amounts of Me were deposited onto polymeric substrates of polyethylene terephthalate (PET) and polypropylene (PP) by DC magnetron sputtering, using a pure Ti target with different amounts of Me pellets. Although, the adhesion problems between the thin films and polymeric-based materials are widely known, the substrate surfaces were activated/functionalized by a set of low-pressure plasma treatments in order to increase the



interfacial adhesion. The influence of the polymer-base on the overall response of the thin film sensor devices was studied and correlated with the main physicochemical characteristics of the different systems produced as well as the chemical composition of each particular Ti-Me system. Preliminary results showed that increasing the amount of the Me in the films, decrease the crack density (Fig.1a and 1b), which is directly related with the shear strength and therefore with the films adhesion.

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IV.PO.5

ANTIFUNGAL PROPERTIES OF MAGNETRON SPUTTERED ZINC OXIDE THIN FILMS

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Keywords: ZnO thin films, antimicrobial surfaces, magnetron sputtering

Abstract: Disease outbreaks from food origin can cause a great socio-economic impact, due to the increasing of infectious diseases. Along with the increasing microbial resistance, it alarms the populations for the need of food security. Preventing these diseases rests in the cleaning and sterilization of every surface in contact with food, during its handling and processing, which reveals to be expensive and time consuming. The need of greater security at the industrial level leads researchers to the study of new methods to prevent contamination. Fungal food contamination is a serious problem since fungi produce mycotoxins that are versatile and a powerful cause of diseases. Previous works had shown that ZnO thin films, produced by means of magnetron sputtering, have antimicrobial properties, when tested against *Escherichia coli*, hampering the bacterial replication. In this work, different sets of ZnO thin films with different thicknesses and/or chemical compositions were deposited by reactive DC magnetron sputtering and tested for antifungal properties against a pathogenic fungus, *Candida albicans*. The yeast was cultured in liquid media on top of the thin films, in order to evaluate its growth capacity. The oxidative stress induced by the ZnO was also evaluated by determining yeast cell membrane integrity and mitochondrial enzymatic activity by flow cytometry. Results showed that for the film sets with thickness higher than 350nm, a decrease in the cellular growth was observed, supported by a decrease in enzymatic activity. These results showed that is of great interest to continue the study and development of these thin films by a variety of fungi testing food contaminant such as Fusaria, Trichothecium, Cephalosporium, etc. that contaminate grains and produce illness with symptoms such as vomiting, diarrhea, headaches, chills, dizziness, and blurred vision. In addition, with these promising results we may also develop this type of thin films to be used in different areas beside the food industry, to help preventing unwanted contaminations and also to reduce the costs of cleaning and sterilization of surfaces and materials.

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Acknowledgements: This work was supported by the Portuguese Foundation for Science and Technology (FCT) in the framework of the Strategic Funding UID/FIS/04650/2013 and UID/BIA/04050/2013 (POCI-01-0145-FEDER-007569)







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IV.PO.6

THIN FILMS COMPOSED OF METALLIC NANOPARTICLES (AU, AG) DISPERSED IN ALN MATRIX: INFLUENCE OF METAL CONCENTRATION AND THERMAL ANNEALING ON THE PLASMONIC ABSORPTION BAND

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Keywords: Thin Films; Noble Nanoparticles; AlN matrix; Localized Surface Plasmon Resonance.

Abstract: Nanocomposite thin films with plasmonic nanoparticles, such as Au or Ag, embedded in a dielectric matrix, have been receiving special attention in a wide range of technological applications, namely in plasmonic sensing (e. g. detection of gas molecules or biological agents), due to their unique optical properties. Most of these applications are based on Localized Surface Plasmon Resonance (LSPR) effect which is regulated by the geometric characteristics (distribution, size and shape) and concentration of the plasmonic nanoparticles, as well as the dielectric environment properties. The possibility of using AIN as a host matrix for LSPR thin films and how metallic nanoparticles concentration and distributions influence their optical response will be discussed in this presentation. Thin films of Au,Ag/AIN were deposited by reactive DC magnetron sputtering and submitted to annealing treatments at different temperatures in order to promote nanoparticles' formation with different size distributions throughout the AIN matrix. Thin films with different Au or Ag concentration and size distributions of metallic nanoparticles promoted by thermal annealing influence the plasmonic behaviour of the thin films, and they might present tuneable LSPR resonance peaks in the visible range and different absorption band characteristics.

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IV.PO.7

THE EFFECT OF IRON ON THE CORROSION OF AlTINICu AND AlTINICuFe EQUI-MOLAR COMPOSITION HIGH ENTROPY ALLOYS

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Keywords: corrosion, casting, heat treatment, diffraction, scanning electron microscopy

Abstract: The AlTiNiCu (Fe0) AlTiNiCuFe15 (Fe15) and AlTiNiCuFe20 (Fe20) high entropy alloys were prepared by induction melting method. Immersion tests and potentiodynamic polarization



Fig. 1. Surface appearances of Fe15 alloy before (left) and after (right) immersion test in $1M H_2SO_4$ solution.

measurements were conducted in 1M sulfuric acid solution (H_2SO_4) at room temperature to investigate the corrosion behavior of Fe0, Fe15 and Fe20. The microstructures of alloys were investigated by X-ray diffractometry and scanning electron microscopy. The results show that the alloys display very good general corrosion resistance because the alloys surface make it easy to form protective film (Al₂O₃ and TiO₂).

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IV.PO.8

INCREASING WEAR RESISTANCE OF POWER STEERING PUMP CAM USING NI-CR-FE AND NI-CR-B-SI-FE COATINGS

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Keywords: Plasma deposition, Ni-Cr-Fe powder, Ni-Cr-B-Si-Fe powder, morphology, XRD, scratch analysis

Abstract: Thermal depositions are very wide spread in the industry of coating techniques. The materials used as coatings for several applications must have the ability to produce a stable, slow-growing surface coating, in order to provide good service behavior. This paper presents a method to increase the wear resistance of steering pump cam, strongly stressed having premature wear effects. The method that the authors use is atmospheric plasma deposition with Ni-Cr-Fe and Ni-Cr-B-Si-Fe powders on steel substrate. It was investigated the morphology and physico-mechanical properties (scratch and micro-indentation analysis). Results showed a comparison between those two coatings with the metallic substrate. It has been found that the deposited coatings have an adherent, dense and uniform layer with a typically molten morphology. By increasing the coefficient of friction we can obtain higher wear resistance and recommend the optimum solution for further researches.

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IV.PO.9

MICROSTRUCTURAL ASPECTS OF TBC`S DEPOSITED ON INTERNAL COMBUSTION ENGINE VALVE MATERIALS

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Keywords: TBC, atmospheric plasma spray, internal combustion engines valve

Abstract The discs of the intake or exhaust valves are vital organs of internal combustion engines, being subjected to extreme operating conditions, thermal, mechanical and chemical types. One of the goals of researches in this area is related to thermal insulation of the combustion chamber of internal combustion engines, which could enhance their performance in operation.

In this article we analyzed the microstructural aspects of some coatings obtained from powders with thermal barrier role on specific materials for internal combustion engines valves. There were used as substrate samples of low alloy steels with Si and high alloyed steels with Cr, Ni and Mn. Using the facility SPRAYWIZARD 9MCE for atmospheric plasma spraying, two types of thermal barrier coatings were produced, from powders based on zirconia and alumina. The samples were analyzed in terms of microstructure using the QUANTA 200 3D scanning electron microscope and the X`PERT PROMD diffractometer. Observations were made both on the longitudinal surface of the coating in order to evaluate it and on the cross-section to evaluate the substrate-coating interface, the influence of deposition temperatures on the substrate and aspect/microstructure on its depth. XRD analysis revealed a cubic structure of aluminum oxide, respectively zirconium oxide. The identified morphology is a specific "splat" one for the ceramic coatings. Surface appearance shows tiny pores and cracks specific to the spraying method. The resulted coatings present a significant compactness and adherence to the substrate, which recommends them for further thermal behavior testing.

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IV.PO.10

STUDIES REGARDING THE WEAR BEHAVIOR OF THE BRAKING MECHANISM

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Keywords: disc brake, temperature, friction, mechanical and thermal stress

Abstract: During braking of any vehicle, due to the friction force created between the disc and the pad, occurs temperature rise with negative effects on the process of slowing down the vehicle. Braking system is subjected to a very high mechanical and thermal stress. Due to overheating brake discs and pads, can occur deformations or cracks and the material they are made can modify its structure. These defects lead to vibrations and noise during the braking, to reducing the friction coefficient, respectively decrease efficiency of the braking mechanism [1].

This paper proposes a study regarding the influence of thermal stresses on the cast iron disc brake [2], of a Dacia Logan car and a theoretical model to establish the friction temperature of surface between the disk and the brake pads [3,4].

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IV.PO.11

SURFACE CHARACTERIZATION OF MFE₂O₄ POWDER OBTAINED BY USING OLIVE OIL AS A SURFACTANT AGENT

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Keywords: Metalic powders, Olive Oil, Surfactant, Characterization



Abstract: This study highlights the synthesis of MFe_2O_4 nanoparticles by coprecipitation method [1] using olive oil as a in-situ surfactant. Structural and surface characterization were examined by Fourier Transform Infrared spectroscopy (FT-IR), X-ray diffraction (XRD), and scanning electron microscopy (SEM) analysis [2]. Magnetic properties are discused based on the magnetization plots for MFe_2O_4 compounds at room temperature [3, 4]. The results demonstrate that the surface of powders contains hydrophilic groups specific those of the olive oil.

Fig.1 Coated particle obtained by coprecipitation method

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IV.PO.12

IMPORTANCE OF POST-PROCESSING FOR CVD GRAPHENE

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Keywords: graphene, chemical vapor deposition, post-processing

Abstract: Graphene is considered the most amazing material, used for electronic and medical applications. At the present there are numerous studies that describe the development of synthesis of monolayer graphene using chemical vapor deposition, but no study shows the cleaning step before transfer process. Post processing of graphene sheets is a crucial step in graphene research. This study emerged as a necessity due to the formation of a film on the graphene layer surface which is caused by unreacted hydrocarbon.



In Fig. 1 is illustrated comparison before and after cleaning step with scanning electronic microscopy for CVD graphene on Cu substrate.

Fig. 1 SEM characterization for graphene/Cu sample: A. after cleaning step, B. before cleaning step

In this work we reported the importance of this cleaning protocol evidence involves two main steps: dry cleaning: boiling in acetone for 10 minutes, fast drying on hot plate and annealing step at 400 Celsius degree. Cleaning up graphene sample is used only in the transfer process, mainly for polymer removal. Techniques as scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) have been used to characterize the morphological and chemical structure, emphasizing the importance of this post processing step.

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IV.PO.13

NEW METALLO-PORPHYRINS -CHARACTERIZATION AND APPLICATIONS IN SOLAR ENERGY CONVERSION-

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Keywords: Porphyrins, FT-IR, RAMAN, UV-VIS, SEM, AFM

Abstract: Tetrapyrrole macrocycles play important roles that serve nature in various ways to energy conversion. Under such context, the tetra-aryl porphyrins derivatives with silicon, are known as one of the most efficient sensitizers in solar energy conversion. The investigations of spectral properties of the metallic complexes of 5,10,15,20-p-tetra-phenyl porphyrins, are achieved by: UV-VIS, FT-IR and RAMAN analysis. The surface morphology of the samples was probed by the atomic force microscope (AFM) and scanning electron microscopy (SEM). Spectral properties (absorption, fluorescence and excited-state lifetimes) of the studied porphyrins indicate that these materials could be perspective for potential photochemical applications.

In the work presented herein, porphyrin have widely-varied properties strongly linked to the central metal and axial ligand, proper for solar energy applications. Because they have high visible light absorption and stable physical properties, the tetra-aryl porphyrin derivatives were examined in conjunction with wide-bandgap semiconductive oxides TiO₂ and SnO₂. The molecules exhibit strong absorption in the visible and near-infrared spectral regions as well as interesting photophysical properties suggesting possible applications in light harvesting.

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IV.PO.14

NANOSTRUCTURED ANITICORROSIVE HYBRID MATERIALS OBTAINED BY PLD

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Keywords: porphyrin, PLD, anticorrosive.

Abstract: Intercalated thin layers made by PLD deposition from the pseudo-binary oxide $ZnTa_2O_6$ [1,2] and zinc meso-tetra(4-pyridyl)porphyrin (ZnTPyP, Fig 1) is expected to self-assemble into ordered nanoparticles and to exhibit uniform and adherent properties, proving the feasibility of the supramolecular approach and offering novel anticorrosive materials. It is already reported that the



presence of non metallated porphyrin TPyP decreased the corrosion rate of steel with over 50% efficiency [3], and we expect the novel proposed materials to be more effective.

Although the supramolecular chemistry of zinc meso-tetra(4pyridyl)porphyrin (ZnTPyP) in the presence of a surfactant was reported to give hexagonal nanoprism [4], we obtain triangular based prism aggregates of ZnTPYP.

Fig 1 STEM image of a large triangular based prism, generated from THF solution of bare Zn (II) tetra (4-pyridyl) porphyrin by H and J-type aggregation.

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IV.PO.15

SURFACE PROPERTIES OF LASER TREATED ARC-DEPOSITED CLADDINGS

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Keywords: welding; low alloy steel; coatings; laser treatment; surface energy; corrosion

Abstract: In this paper, several arc-generated Cr-Mn-Ni based claddings have been obtained on a low-alloy steel, with the help of ElCrMn2 and E48T electrodes. The claddings have been further submitted to laser treatment at two different powers, namely 2150 W and 2425 W.



Fig. 1. Water contact angle on the surface of the laser-treated sample

The laser treatment promotes the formation of martensite and chromium carbides on the surface of the samples, leading to a significant increase in the hardness of the coatings, as well as in their wear resistance. Laser treatment leads also to an increased stability of the claddings in aqueous environments, by their lower surface energies than those of the reference materials (Fig.1) and an improved overall compatibility with different types of water and oil-based paints and primers, lowering their specific consumption.

The overall high cost of the laser treatment could be overcome by the obtaining of performant hardcoatings through our proposed method, working under environmentally demanding conditions and having a prolonged life cycle [1].

The laser beam heating produces two kinds of regions inside the laser tracks. One region is composed predominately by martensite, and another region present is unchanged proeutectoid ferrite, martensite and some pearlite. The case depth varies with the laser power. The maximum hardened depth is 0.3 mm for a laser power of 2425 W. Under high power, the laser tracks partially overlap; therefore, some tempering occurs at the overlapped zones.

The current methodology shows a promising alternative to induction-hardened shafts and could be easily implemented within the production process. The method is rapid and allows treatment of specific surfaces on the piece.

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IV.PO.16

PRELIMINARY RESULTS ON EFFECT OF H₂S ON P265GH COMMERCIAL MATERIAL FOR GAS AND PETROLEUM TRANSPORTATION

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Keywords: H₂S, EIS, SEM, EDS

Abstract: A commercial Fe-C material (P265GH) used for natural gas delivery and transportation systems was analyzed in H₂S atmosphere in order to establish the corrosion resistance. In most of the industrial processes for gas purification the corrosion rate is speed up by the presence of S (sulfur) especially as ions (HS-, SO_3^{2-}) or different species like H₂S [1, 2]. The H₂S (hydrogen sulfide) is, beside a very toxic compound, a very active element in the acceleration of metallic materials deterioration. For experiments we used a three electrodes cell with Na₂SO₄ + Na₂S solution at pH 3 for two different temperatures, room temperature ~ 25°C and at 60°C in order to realize EIS (electrochemical impedance spectroscopy) and potentiodynamic polarization. The experimental samples surface was analyzed using SEM (scanning electron microscopy with VegaTescan LMH II) equipment and EDAX (X-ray energy dispersive spectroscopy, Bruker type) for structural and chemical analyses. The electrochemical tests were carried out at various H₂S compositions (0, 5, 10 and 15 ppm). XRD determinations on the expose surface at H₂S were done in order to establish the presence of FeS_x on the metallic material surface.

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IV.PO.17

COMBINED FAST LASER TEXTURING AND HVOF TiO₂ POWDER DEPOSITION SURFACE ENGINEERING TREATMENT FOR CORROSION PROTECTION

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Keywords: fast laser texturing, HVOF spraying, duplex surface treatment, anticorrosive.

Abstract: One of the advanced surface engineering processes used for obtaining thin to thick deposited layers for various (multi)purposes is High Velocity Oxy-Fuel Spraying. The process can be used for a wide range of applications due to its main advantages, e.g. lower porosities, harder and tougher coatings, thicker coatings due to less inter-layers residual induced stresses, etc. The use of



Fig. 1. SEM image of TiO₂ HVOF sprayed coating

laser processing proved to be a technology that can improve the HVOF technology [1] but also for applications related to corrosion resistance [2]. The paper presents the characteristics of TiO_2 coatings realized by HVOF spraying process by two processing paths: using the classical method of preparing the substrate by sandblasting and by applying a fast laser texturing of the substrate before the HVOF spraying respectively, i.e. a duplex treatment. The obtained coatings' morphology was characterized

by SEM / EDAX before and after electrochemical measurements in NaCl solution.

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IV.PO.18

IN-SITU XRD VS EX-SITU HEAT TREATMENT OF TANTALUM OXYNITRIDE THIN FILMS: SIMILARITIES AND DIFFERENCES ON THE STRUCTURAL EVOLUTION

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Keywords: additive manufacturing, powder bed fusion

Abstract: The purpose of this work is to discuss the main structural characteristics of a group of tantalum oxynitride thin films, prepared by magnetron sputtering, and to interpret and compare the structural changes, by X-ray diffraction, when the samples are heat treated in several different conditions: i) one sample of each deposition experiment is annealed at a different temperature, until a maximum of 800 °C, and the XRD patterns are obtained ex situ, at room temperature, after each annealing process; ii) the in situ XRD patterns are obtained, at certain temperatures, during the annealing process, in the same sample. Taking into account that the in situ XRD annealing was performed after the ex situ annealing, it was decided to evaluate the evolution of the structure of the in situ experiments up to 1000 °C; iii) in-situ DSC heating up to 600 °C, as well as in-situ DSC cooling, up to -200 °C, in argon atmosphere, at atmospheric pressure. It was found that films with around 32% of non-metal content exhibit the presence of fcc-Ta(N,O) crystallites, randomly oriented. The stability of the structure is maintained until 800 °C, and the fcc-Ta(N,O) is present until 1000 °C, but at 800 °C and above, it is observed the formation of Ta_2O_5 crystallites, mainly of the β -Ta₂O₅ phase. Furthermore, films with non-metal content around 50 % or higher, exhibit amorphous structure, stable until T = 700 °C (if N content is around 18% or higher) and T = 800 °C (if N content is lower than 18%). If the N content is 18% or higher, it is enough to create monoclinic TaNO crystallites when insitu anealed above 700 °C. If the N content is lower (~13%) it is not enough to promote the formation of TaNO and the O amount promote the formation of Ta₂O₅ crystallites above 800 °C.

Acknowledgements: We hereby acknowledge the structural funds project PRO-DD (POS-CCE, O.2.2.1., ID 123, SMIS 2637, ctr. No 11/2009) for providing some of the infrastructure used in this work. This work was also supported by the Portuguese Foundation for Science and Technology (FCT) in the framework of the Strategic Funding UID/FIS/04650/2013.






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IV.PO.19

SURFACE IMPROVEMENT OF COLD SPRAYED COPPER-ALUMINA COATING VIA FRICTION STIR PROCESSING

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Keywords: friction stir processing, cold spray, ceramic coating, metal matrix composite

Abstract: Particle reinforced metal matrix composites (MMC) have a wide range of industrial application due to their mechanical, tribological, electrical and thermal properties. A promising technology for MMC fabrication is cold spray deposition which uses low or high pressure gas to



Fig. 1. SEM image of copper-alumina powder

accelerate particles before impacting them into the metallic substrate. A review of cold spray deposition [1] indicates that major drawbacks, especially in case of low pressure cold spray, are non-homogenous structure of coating and limited particle-substrate bonding. Recent research [2, 3] demonstrated that applying friction stir processing (FSP) after cold spray deposition is a simple and economical method to overcome these problems.

In this respect, the present paper investigates the effect of friction stir processing parameters on microstructure and properties of aluminum samples coated with Cu/Al₂O₃ [Fig. 1.] by cold spray. Tests were carried on using a cylindrical alumina tool and rotation speed, travel speed and

normal load were varied during FSP. The evaluation of the processed zones demonstrated that a more refined and harder surface was obtained by combining cold spray with friction stir processing.

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IV.PO.20

STRUCTURE AND PROPERTIES OF BIODEGRADABLE CALCIUM PHOSPHATE COATINGS DEPOSITED BY MAGNETRON SPUTTERING ON BIODEGRADABLE MAGNESIUM ALLOYS

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Keywords: magnesium alloys, orthopedic implants, microscopy, corrosion, surface.

Abstract: Various studies have shown encouraging potential of the biodegradable magnesium alloys for biomedical applications but with serious concern related to their biodegradation rate in human body environment [1]. Due to their ability to generate a rapid bond with hard tissue, bioceramics like



Fig. 1. AFM images of the different layers coated on the MgCa0.8 magnesium alloy: SMO- $Ca_3(PO_4)_2$, SM1,SM2- $Ca_3(PO_4)_2$ with Si and Mg in different ratio. hydroxyapatite, bioglass and calcium phosphate materials are recommended if we intent to develop an orthopedic application [2]. The aim of this study was to evaluate the structure and properties of calcium phosphate coatings with Si and Mg in different ratio (SM0, SM1, SM2), deposited by magnetron sputtering on the experimental the Mg-Ca (0,8 wt%) alloy. The deposited layers (SM0, SM1, SM2) was characterized by X-ray Diffraction, Scanning

Electron Microscopy, Energy Dispersive X-ray spectroscopy, and Fourier Transform Infrared Spectroscopy, in order to obtain relevant data about their morphology, composition, and structure. Also, the surface properties and morphology were analyzed by profilometry, atomic force microscopy, and contact angle measurements. Biodegradation rate was evaluated by immersion tests in Simulated Body Fluid, following the hydrogen release rate, pH variation, weight loss, and the modification of the surface morphology, and by electrochemical methods. The results evidence that the calcium phosphate coating improves the biodegradation rate of the Mg-Ca (0,8 wt%) alloy.

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IV.PO.21

FUNCTIONAL COATINGS WITH PROTECTIVE PROPERTIES DEPOSITED BY COLD SPRAY TECHNOLOGY

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Keywords: cold spay, original mixture of micro-powders, carbon micro-powder, coefficient of friction

Abstract: Researches for elaboration of the functional coatings with protective properties (lubricant/ hardening/ anticorrosion/ thermal barrier, etc. properties), using the new Cold Spray technology, for deposition of 1-50 μm micro-powders in open atmosphere have been developed in this work for their tribological evaluation by pin-on-disk tribometer test. Original manufacturer mixtures of micropowders with protective properties, as also original mixtures of micro-powders combined with a top lubricant micro-powder (carbon/ graphite) were deposited [1]. Also, for proving of the cold spray technology capabilities, different deposition parameters, such as: 1. Concentration of the component micro-powders in the mixture; 2. Heating Temperature of carrier gas respectively of the micropowders; 3. Pressure of the Compressed Air (carrier gas) were used [2]. The deposition materials consisted in mixtures of micro-powders from Cu:Al₂O₃+Zn, Sn-83%+(Cu+Sb)-17% (Babbit-83) and Sn:Al₂O₃, with anticorrosion, lubricant, thermal barrier or hardening properties [1]. Also, compounds of carbon/graphite micro-powders e.g.: 90%Babbit-83+10%C and 90%(Cu:Al₂O₃+Zn)+10%C were deposited in this work [2]. The best coefficient of friction (COF) and wear rate coefficient (K) for the mixture of micro-powders were obtained for the Cu:Al₂O₃+Zn micro-powder mixture, when it was 0.18 - 0.44, respectively $(4.6-8,4)x10^{-4}$ mm³/Nm, while in the case of mixture of graphite micropowders (C) with manufacturer original micro-powders the best COF was obtained for the 90%Babbit-83+10%C compound, when the COF was reduced up to 0.18-0.20, for a sliding length test of 90 m and up to 0.30 - 0.38, for a sliding length test of 150 m. Using cold spray technology for deposition of original micro-powder mixtures with a top lubricant material (C micro-powder) an improvement in the tribological behavior of the coatings has been observed.

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IV.PO.22

IMPROVEMENT OF THE MECHANICAL AND TRIBOLOGICAL PROPERTIES OF THE METALLIC MATERIALS BY THER SURFACE NITRIDING IN OPEN ATMOSPHERE COLD PLASMA SURFACE TREATMENT

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Keywords: surface nitriding, open atmosphere, cold plasma, hardness, coefficient of friction

Abstract: This work presents preliminary researches for proving the capabilities of Open Atmosphere Cold Plasma Surface Treatment Method (OACP-STM) to improve the mechanical and tribological properties (hardness and coefficient of Friction) of the 304 stainless steel surfaces by their nitriding, in N₂ cold plasma, developed in open atmosphere without any heating to obtain α + ϵ equilibrium nitriding phase [1] and not classical nitriding temperature of 350-520^oC [2]. For the first experiments, the yellow-brune color (color well known for chemical compunds $TiN_x \& TaN_x$) of the Ti & Ta samples was visually observed on the nitrided surface after 0.1h. The capabilities of OACP-STM for nitriding of 304 Stainless Steel (304 SS) surface samples were proven by comparatively evaluation of the hardness (obtained by nano-hardness indentation test) and coefficient of friction (obtained by ballon-disk tribometer test) for 304 SS samples, un-treated or treated by OACP-STM for ~ 0.5 h, using nitrogen as working gas for cold plasma generation in open atmosphere. The preliminary tests have shown that the hardness of the 304 SS samples treated by OACP-STM with N_2 as working gas is higher (6.75 GPa) than the one of the un-treated surface (3.43 \pm 5.31% GPa) and it decreases rapidly with the depth increasing. Also, the coefficient of friction of 304 SS samples treated by OACP-STM with N_2 as working gas is much lower for the first part of sliding length test than that of the untreated surfaces.

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SECTION V Nanomaterials

Chairpersons:

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V.K.1

REGULAR SURFACE NANOPATTERNING WITH NANOSPHERE LITHOGRAPHY, BLOCK-COPOLYMER LITHOGRAPHY AND COMBINATIONS OF BOTH

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Keywords: nanopatterning, block copolymer lithography, nanosphere lithography, bottom-up techniques

Abstract: Nanosphere lithography (NSL) is a well-established technique to form regular surface patterns with periodicities in the range of several hundred nanometers. Here, self-arranged colloidal spheres act as shadow mask to modify the free interstices between the spheres. The technique can be easily applied to create patterns on large-area surfaces of several square centimeters. Recent examples will be given in which the NSL technique is used to direct the growth of semiconductor nanowires, to arrange proteins on a surface and to create novel plasmonic devices, mostly by combining NSL with physical vapor deposition PVD, chemical vapor deposition CVD and/or reactive ion etching RIE.

In order to achieve patterns which are 1-2 orders of magnitude smaller than the ones created by NSL, block copolymer (BCP) lithography is employed. This is an emerging self-organization technique in which the phase separation of BCP molecules is exploited to generate regular surface patterns the periodicity of which is governed by the length of polymer chains. Examples will be given where this is

used to achieve patterns which have a much higher areal density than in NSL and thus can be particularly useful in sensorics and photovoltaics.

The drawback of BCP lithography so far is the small length scale over which the periodicity is maintained. Therefore prepatterning of the substrate prior to the application of BCP lithography is required in order to provide a template for the self-assembly of BCPs. It will be demonstrated that the combination of NSL and BCP lithography is a promising low-cost attempt to form well-ordered surface patterns on wafer-scale surfaces with motive sizes in the sub-20 nm range. Emphasis will be placed on the formation of hexagonally arranged nanopores hierarchically ordered in sub-micrometer pores in metallic films.



Fig. 1 Hierarchically ordered nanopores

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V.K.2

THE ROLE OF HYDROGEN IN MATERIALS DEGRADATION

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Keywords: hydrogen, materials degradation, residual stress

Abstract: Development and validation of a lifetime prediction methodology for failure of materials used for hydrogen containment components is of significant importance to the planned hydrogen economy. With the prospect of transitioning to a hydrogen-based economy, many engineering components will be exposed to high-pressure gaseous hydrogen environments.

Hydrogen embrittlement is a severe environmental type of failure; when hydrogen is present, materials fail at load levels that are very low compared with those that a hydrogen free material can sustain.

We will review recent contributions to the understanding of mechanisms of hydrogen embrittlement. In this paper, we describe the role of hydrogen in different structural materials with an emphasis on steels.

Thermal desorption spectroscopy (TDS) was used to identify and quantify the types and strengths of the hydrogen trapping sites. TDS results support the notion that only the diffusible hydrogen through the lattice sites or the hydrogen residing at the traps with the lowest binding energy contributes to material embrittlement; the deeper traps were saturated in both hydrogen free and charged samples. Hydrogen trapping and diffusion will be discussed in relation with microstructure features and mechanical states. We present a model for hydrogen transport that accounts for trapping of hydrogen at microstructural defects and address the interaction of hydrogen solute atoms with material deformation.

The residual stress state in a material has an important role in the mechanism of cracking, induced or assisted by hydrogen. The hydrogen interaction with residual stresses is studied by synchrotron x-ray diffraction. The results will be discussed in detail.



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V.O.1

TiO₂/Pt-NPs PHOTOCATALYST SUPORTED ON ZEOLITES OBTAINED FROM FLY ASH

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Keywords: zeolite, nanocomposite, wastewater treatment

Abstract: The present of dyes at very low concentrations in surface water affect water transparency reducing light penetration and gas solubility in water, thus disturbing the ecosystem. Removal of pollutants like dyes with a complex structure (azo-, antrachinone, metal-complex dyes), heavy metals



Fig. 1. SEM image

cations, surfactants from wastewater was investigated by adsorption and photodegradation onto а lot of substrates. There are many materials obtained from fly ash and semiconductor oxides (WO₃, TiO₂,) used in removing pollutants from wastewater [1]. The zeolites obtained from fly ash [2] are an excellent support for TiO₂, thus a composite (fly ash with TiO₂) which could be efficient in the treatment of wastewaters loaded with dyes, by combining the adsorption and photo catalysis processes.

A novel composite was obtained through mild hydrothermal synthesis from Degussa P25 and activated fly ash. Platinum nanoparticles were added to the composite surface to increase the photocatalitic activity of the nano- composite powder. This material was noted FLY2NP.

The adsorption and UV photocatalytic activity of the composite (FLY2NP) were evaluated in removing of two textile dyes: Bemacid Blau (BB), Bemacid Rot (BR). In photocatalysis the results are over 80.70% for BB and 40.24% for BR at 300min optimum contact time.

The composites' surface properties were evaluated by (SEM) Fig. 1, and atomic force microscopy (AFM).

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V.O.2

SYNTHESIS AND PROPERTIES OF Cu₂ZnSnS₄ NANOPARTICLES

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Keywords: nanoparticle, solar cell material, band gap

Abstract: Cu_2ZnSnS_4 (CZTS) is identified as an efficient alternative for the widely used $Cu(In,Ga)Se_2$ absorber layer in solar cells. The band gap and absorption coefficient of CZTS are well suited for solar cell applications. Moreover, the constituent elements of CZTS are non-toxic and earth-abundant. Here, CZTS nanoparticles are prepared using wet-chemical synthesis. In comparison with other deposition techniques, the solution based method offers significant cost-reduction in CZTS cell fabrication. Another advantage is the viability in using different coating methods such as spray, spin coating, doctor blading and screen printing. In the present work, CZTS nanoparticles are synthesized using hot injection method. Particles are prepared at different durations such as 3, 6, 9 and 12h. Tetragonal kesterite phase of CZTS is prominent in all the films together with traces of wurtzite structure. Compositional analysis shows absence of Zinc with increase in the deposition duration to 6, 9 and 12 h which points to the possible formation of Cu₃SnS₄ phase. Raman analysis confirmed the presence of Cu_3SnS_4 phase with a peak at 316 cm⁻¹[1]. Also A1 mode of CZTS appeared at 332 cm⁻¹ [2]. In the sample synthesized in 3h duration, Cu₃SnS₄ phase is absent and hence duration is fixed at 3 h for further variation in composition. Band gap varied from 1.6 to 1.3 eV with increase in the synthesis time. The reduction in the band gap is due to the presence Cu₃SnS₄. Preferred composition (21.9:13.1:14.9:50.1) is achieved by reducing both Cu and Sn concentrations and increasing Zn concentration. XRD and Raman analysis of these samples confirmed the presence of Kesterite Cu₂ZnSnS₄ structure. As in the previous case, peaks corresponding to wurtzite structure of CZTS are also observed in these films. Secondary phases are absent in these samples and band gap of the film is ~ 1.5 eV. The prepared CZTS nanoparticles have a size of ~ 6 nm.

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V.O.3

CONTROLLING THE CHAIN ORGANIZATION AND THERMOCHROMIC BEHAVIORS OF POLYDIACETYLENE ASSEMBLIES BY VARYING SOLVENT MEDIA

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Keywords: polydiacetylene, chain organization, temperature sensor, self-assembling, solvent effect

Abstract: In this study, we investigate chain organization and thermochromic behaviors of polydiacetylene(PDA) assemblies, prepared from two types of diamidodiacetylene monomers, by utilizing various techniques including UV/vis absorption spectroscopy, X-ray diffraction, differential scanning calorimetry(DSC) and scanning electron microscopy. Chemical structures of PDA assemblies are modified by varying length of alkyl linkers, which are ethyl and hexyl, between the diamide groups.^{1,2} The preparation of PDA assemblies involved the dispersion in different solvent media followed by UV light irradiation. Various solvents including water, butanol, octanol, octane and decane were used. We have found that the change of solvent media significantly affects the chain organization behaviors of PDA assemblies. The use of water provides the PDA assemblies with irregular shape while the sheet–like structure is obtained from the systems of other solvents. The interlamellar d-spacing of PDA assemblies also varies with type of solvent media, indicating the difference of molecular arrangement. DSC study detects the variation of phase transition temperature. When the PDA assemblies are prepared in water, the color-transition temperature is lower compared to other systems. The increase of alkyl linker length also causes the increase of color-transition temperature.

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V.O.4

PROPERTIES ANALYSIS OF DEXTRANE MAGNETIC CARRIER COMPOUNDS WITH POTENTIAL APPLICATIONS IN ENVIRONMENTAL AND BIOMEDICAL FIELDS

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Keywords: Magnetic nanoparticles, organic compounds, biomedical applications, characterization techniques.

Abstract The paper describes the results obtained by synthesis and characterization of iron oxides nanoparticles coated with dextran for environmental applications. The experiments consisted in



Fig. 1: TEM image for Fe3O4 covered with Dextrane

synthesis of iron oxide nanoparticles, covering them with Dextrane and their characterization (Fig. 1). For the synthesis of iron nanoparticles the co-precipitation technique was used, while for the characterization were used techniques such as scanning electron microscopy, transmission electron microscopy, X-Ray diffraction spectrometry and Fourier transform infrared spectroscopy. The morphology experiments certified the obtaining of iron oxide nanoparticles covered with an organic compound (dextran) with an agglomeration tendency. Also, the nano dimensions of the analyzed sample were confirmed. The obtained nanoparticles can be used in the environmental engineering field, as adsorbent particles for heavy metals removal [1], likewise in biomedical fields a matrix in which drugs [2], radionuclides or genetic material can be dissolved; thus the magnet-coating system can perform as

"carrier" to transport useful material to the targeted area.

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V.O.5

CARBON COATED METALLURGICAL GRADE SILICON DECORATED GRAPHENE ANODES

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Keywords: metallurgical grade silicon, graphene, CNT, anode, li-ion battery

Abstract: Silicon (Si) is a very attractive candidate anode material for secondary lithium ion batteries because of alloying with lithium. Thus it exhibits the highest theoretical energy storage capacity of 4200 mAh/g which is more than 10 times higher than graphite anode material (372 mAh/g) [1,2]. On the other hand the poor cycling stability of the silicon based anode materials because of the severe volume expansion during the intercalation/deintercalation process inhibits its application. There are different methods to overcome the volume expansion problem. Using different nanostructures of Si materials such as nanowires [3], nanotubes [4], nanofibers [5] and porous materials [6], thus getting enough vacancies for the volume change during the lithiation and de-lithiation process is one of methods. On the other hand using of silicon-carbon hybrid structures such as CNT or graphene are integrated into the Si material as anode is the second method [7]. Because of metallurgical grade silicon is cheap and readily available, using of this type silicon can be a solution for the development of li-ion batteries. In this work, we reported the synthesis of Silicon/Graphene/MWCNT composite structures which were produced via high energy ball milling and vacuum filtration technique. Firstly the large metallurgical grade silicon particles were converted micrometer silicon particles via high energy ball milling method. Then the samples were coated with carbon and etched by using hydrofluoric acid. Graphene oxide was produced by Hummer [8] method and reduced to graphene with hydrazine hydrate solution. Silicon/Graphene/MWCNT papers were produced with vacuum filtration technique. The structure and morphology of the nanocomposite electrodes were characterized by X-ray diffraction, Raman spectroscopy, scanning electron microscopy. The electrochemical characterization tests including galvanostatic charge/discharge, cyclic voltammetry and electrochemical impedance spectroscopy (EIS) measurement of the electrodes were carried out by using an CR2032 test cell. These high performance graphene supported composite electrodes provide competitive properties relative to other electrode materials for Li-ion batteries.

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V.O.6

INFLUENCE OF CARBON NANOTUBES USED IN CARBURIZING HEAT TREATMENT OF SAE 3310 STEEL

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Keywords: CNT network, carburizing, mass transfer

Abstract: The properties of carbon nanotubes, especially thermal conductivity [1] allowing their use in carburizing process with with huge possibilities. Obtaining carbon nanotubes relatively easy [2,3], led to a new carburizing heat treatment in controlled atmosphere full with carbon nanotubes. In this



Fig. 1. SEM micrograph with used CNT network

aper is presented experiments on samples of AE 3310 carburized in presence of different lantity of carbon nanotubes in furnace. Were easured hardness at the surface layer and epth of carburizing in same conditions, time 30min., and temperature T=980°C. The sults were compared with the results otained on samples carburized in normal ondition. Use of carbon nanotubes for irburizing requires less time and best ffusion. The hardness is higher than the ardness obtained in regular carburizing eatment. Experiments made on samples of eel SAE 3310 has demonstrated the role of

presence of carbon nanotubes in carburizing atmosphere. It has developed the conclusion

that the carburized layer properties depend on the amount of carbon nanotubes, during carburizing.

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V.O.7

DEPOSITION OF TiO₂ NANOSTRUCTURES BY MOCVD WITH METALLOCENE CATALYSTS

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Keywords: MOCVD, TiO₂ nanostructures, metallocene

Abstract: Within the Nanoform Theme, Nanosciences Ax, ICB, we realize the growth of 1D, 2D and 3D hybrid TiO₂ nanostructures by Metal Oxide Chemical Vapour Deposition (MOCVD) technique. Our work focuses on the theoretical aspect of the formation of these nanostructures (growth and kinetic models, structure and texture) and the study of photoelectric, optical and physicochemical properties as well as the development of applications of these structures in the field of physics (random laser), photo catalysis, energy (Ion-Ii batteries, solar cells ...). [1]

Under certain conditions, original reproducible structures, which are not clearly described in literature, were obtained (membranes, crucibles, forests, coaxial heterostructures...). They were obtained in one step on silicon (100) wafers in the MOCVD reactor using titanium isopropoxide as a precursor and metallocenes as catalysts.



Fig. 1. TiO₂ membranes (left); TiO₂ forests (center); Fe-TiO₂ COHN (right).

Our studies have led us to believe that the cause of these structures is using ferromagnetic catalysts in the presence of an induction heating. [2] In order to confirm this theory and to study the growth mechanism, parameters like: deposition time, temperature, catalysts etc. were varied [3]. The differences on the thin films morphologies and structures were registered and analyzed.

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V.O.8

CHARACTERIZATION OF VC REINFORCED Cu, Fe AND Ni COMPOSITE POWDERS PRODUCED VIA MECHANICAL ALLOYING

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Keywords: transition elements, vanadium carbide, mechanical alloying, nanocrystalline materials, Williamson – Hall method, Lorentzian function

Abstract: In this study, VC powders were added to Cu, Ni and Fe powders to constitute 75Cu-25VC (wt. %), 75Ni-25VC (wt. %) and 75Fe-25VC (wt.%) powder mixtures. The mixtures were mixed in laboratory scale tubular blender for homogenization and then the mixtures were mechanically alloyed against time. After mechanical alloying (MA), powders were characterized using analytical technics. For this purpose apparent densities, true densities, green densities, and particle size and distributions (PSD) of composite powders were measured and data obtained compared with the initial values of the Cu, Ni, Fe and VC powders. Structural analysis of the powders determined by Xray diffraction (XRD) analysis, and the microstructure of composite powders were characterized by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) supported analysis. In this sense, the changes in the lattice parameter (a), crystallite size (D) and lattice strain (ϵ) of the VC reinforced Cu, Fe and Ni matrixes during MA were also investigated. Fundamentals parameters approach (FPA) and Williamson – Hall (W-H) plot analysis coupled with Lorentzian (L) function were applied in order to estimate ε and D of matrix phases and XRD peak shifts and amatrix were determined at various MA durations. Furthermore, effect of annealing temperature on the final powder properties was investigated. In addition, thermal behaviours of composite powder were examined via differential scanning calorimetry (DSC) and vertical dilatometry supported studies were performed to determine the sintering temperature of composite powders.

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V.O.9

MORPHOLOGICAL ANALYSIS OF MAGNETIC NANOMATERIALS THROUGH COMPARATIVE METHODS

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Keywords: AFM; magnetite; nanopowder; SEM; XRD

Abstract: The analysis of particles morphology and structure is one of the most important factors for characterizing a material, powder or substance. Several different methods are available in the scientific environment to study material properties such as Scanning Electron Microscopy (SEM), X-



Figure 3. AFM 3D image of the dispersed powders with profiles

Ray Diffraction (XRD), Energy Dispersive Spectroscopy (EDS), Atomic Force Microscopy (AFM). Material analysis involves the study of particle/grain size, structure, elemental composition or other properties such as magnetism, electrical conductivity or phase changes. Powder size classifications vary from macroscopic micro-submicron to and nanometric particle sizes. ultimately Measurements done by using different methods can give various results on particle

size or morphology depending on the accuracy of the equipment used. In this study, a magnetic powder made of magnetite (Fe_3O_4) was synthesized through a co-precipitation method [1,2] and it was analyzed by using XRD, SEM and AFM in order to determine the particle/agglomeration sizes of the powders. The synthesized powder is nanometric in scale. The method used for measuring the particle size through XRD is the Scherrer equation, which allows for the calculation of crystallite size based on XRD patterns. For comparison, particles were analyzed by SEM to determine agglomeration sizes in a 2D format and then they were compared to the AFM results of the same powder in a 3D spectrum. The purpose of the analysis is to determine the level of accuracy between the methods when analyzing magnetite nano-powders.

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V.O.10

IN SITU SYNTHESIS OF SNO₂/GRAPHENE NANOCOMPOSITE ANODE AND ITS ENHANCED LI–ION BATTERY PERFORMANCE

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Keywords: tin oxide, graphene, anode, Li-ion batteries

Abstract: Rechargeable Li-ion batteries are aroused a lot of interest because of their high voltage, high energy density, stable cycling, and environmental-friendly properties. Nowadays, the continuous miniaturization of electronic devices such as implantable medical devices, smartcards, microelectromechanical systems (MEMS), remote sensors, and RFID tags is led to the growing demands of micropower sources. Rechargeable microbatteries are recently become the topic of widespread research for use in low power applications and energy storage systems for electronic devices. The general requirements of the microbatteries for these applications are high specific energy, wide range of temperature stability, low self-discharge rate and flexibility of cell design. SnO₂ is very attractive electrode material because of its high theoretical capacity (1491 mAhg⁻¹), good cyclability and high columbic efficiency. During charge/discharge process, volume expansion and pulverization have occurred in the SnO₂ electrodes materials in addition to their superior properties. To overcome this problem, forming composite structure with carbon based materials is a efficiency way. Carbon with different forms (graphene, carbon nano tube, carbon fiber etc.) is suitable materials in electrochemistry applications because of its mechanical properties and its high chemical stability in acidic or basic solutions at a wide temperature range [1-3]. In this study, in situ one-step solution based chemical synthesis method was carried out for preparation of SnO₂/graphene nanocomposite. Graphene oxide was produced Hummer methods using flake graphite. Further loading of SnO₂ on GO was carried out with an ultrasound assisted solution-based synthesis route by using SnCl₂.2H₂O. The reduction of GO and the in-situ formation of SnO₂ nanoparticles were achieved in one-step. The structure and morphology of the nanocomposites were characterized by X-ray diffraction, scanning electron microscopy and thermogravimetric analysis. The electrochemical characterization tests including galvanostatic charge/discharge, cyclic voltammetry and electrochemical impedance spectroscopy (EIS) measurement of the electrodes were carried out by using an coin type CR2016 test cell.

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V.O.11

EXFOLIATION OF GRAPHITE IN CARBON ARC DISCHARGE

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Keywords: exfoliation, graphite, few-layer graphene, carbon arc discharge

Abstract: Exfoliation of graphite is an important process, because it allows to obtain the well-defined



Figure 1. SEM image of exfoliated graphite, test under He, 15 kPa.

from the inner walls of the reactor.

precursor of graphene. This process can be carried out in several ways, as for example via intercalation of graphite with alkali metals [1-2] or via ultrasound treatment of graphite aqueous suspensions containing surfactants [3-4].

In this work the solid graphite rod was exfoliated in carbon arc discharge. The anode and the cathode were made of pure graphite. The process was carried out under helium or nitrogen-helium (1:1 v/v) atmosphere. The starting pressure of the buffer gas was in the range between 5 and 50 kPa. The pressure during the process, which took 15-50 minutes, increased 1.6-2.0 times and this effect was connected with subsequent heating of the buffer gas. The discharge was conducted using a constant current of 30 A. Two types of products were collected in each test: (i) the solid deposit from the cathode and (ii) soot-like powder

In all tests carried out under N_2 -He atmosphere, the electrode erosion rate (0.41 – 0.99 mg/s) increased monotonically with the starting pressure. The erosion rate in tests under He atmosphere was almost identical in all experiments (0.11±1 mg/s), and the pressure increased 1.2-1.5 times. The cathode deposit obtained in tests under N_2 -He atmosphere contained carbon microspheres, graphite flakes and nanotubes, and their relative content varied with the pressure. The soot-like product comprised of few-layer graphene and carbon nanoparticles (50-100 nm in diameter). The higher content of few-layer graphene in soot was found in products obtained at lower pressure. The amount of soot in tests carried out under He was less than 5 mg. The dominant product was the cathode deposit and its morphology changed with the pressure. The highest selectivity was found for the starting pressure of 15 kPa and in this case the deposit contained mainly exfoliated graphite (Figure 1).

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V.O.12

WASTEWATER TREATMENT USING ZEOLITES NANOMATERIALS

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Keywords: zeolite nanomaterials, wastewater treatment, heavy metals removal

Abstract:

Zeolites used in wastewater treatment are efficient because of multiple reasons such as: high internal surface areas, ability to lose and gain water reversibly and to exchange some of their constituent elements without a major change of structure, high efficiency for retaining various compounds in their structure since will provide a high accessibility of them into the structure and at the active sites (adsorbtion), the possible regeneration and reuse of the zeolite, the recovery of the pollutant compounds after regeneration of the zeolite (without creating new waste) [1, 2, 3].

Zeolites display unique physical and chemical features and have a variety of industrial applications. The purpose of this research was the study of ZSM-5 type zeolites for wastewater depollution by removal of two heavy metals (Pb²⁺ and Cd²⁺). The synthesis and characterization of Na- ZSM-5 and NH4- ZSM-5 were done by X-ray diffraction (XRD) and transmission electron microscopy (TEM). Also, nitrogen adsorption-desorption isotherms were measure for revealing the specific surface area values and the pore size distribution.

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V.PO.1

COMPOSITE MATERIALS FOR IBUPROFEN REMOVAL FROM WATER BY CATALYTIC OZONATION

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Keywords: Ibuprofen; catalytic ozonation; core-shell catalysts; endocrine disruptor compounds.

Abstract: Endocrine disruptor compounds are widely present in surface and ground water and encompasses a variety of chemical species both natural and with anthropogenic origin. Among them ibuprofen represent an important threat to aquatic ecosystem since interfere in activity of several hormones through steroidogenic pathways in both vertebrates and invertebrates [1].

Catalytic ozonation is an eco-friendly alternative for removal of ECD due to the efficiency and versatility of this method [2,3]. Core-shell type nanomaterials with magnetic properties seem to be a viable choice since both the core and the shell have tunable characteristics. In this work nanoparticles with core-shell morphologies have been prepared and used in the treatment of aqueous solutions contaminated with different concentrations of ibuprofen. This composite material is made on mixed ferrites core which allows us to retrieve easily the particles from the solution and is coated with materials which give catalytic properties in the ozonation process.

In the study of ibuprofen oxidation several parameters were investigated: initial pollutant and ozone concentrations, catalyst dose, evolution of oxidation by products and pH. All prepared catalysts are active in the liquid phase oxidation of ibuprofen. The experiments indicates that degradation rate of ibuprofen increases significantly in the presence of catalysts and the pollutant removal rate follows a pseudo-first order kinetics model. It was also found in radical scavenger experiments that demonstrate that catalytic ozonation of ibuprofen by core-shell catalysts follows a hydroxyl radical reaction pathway.

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V.PO.2

ACTIVATED CARBON AC35 NANOMATERIAL TEMPERATURES LIMITS FOR SOLAR ADSORPTION COOLING SYSTEM USING METHANOL AS REFRIGERANT

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Keywords: Temperatures limits, Activate carbon AC35 nanomaterial, Solar adsorption, Performance coefficient, Cold production

Abstract: The knowledge of the limits temperatures of adsorbent nanomaterial is more important for solar adsorption cooling system performance. The COP (coefficient of performance) depends on these parameters, which are affected by adsorbent/adsorbate working pair. A numerical evaluation for the limits of temperatures of the thermodynamic cycle of adsorption refrigeration machine using the activate carbon AC35 nanomaterial and methanol as working pair is given in this paper. The cold production was better for the lower adsorption temperatures of activate carbon nanomaterial and the higher temperatures of generating. For each 01 Kg of activate carbon nanomaterial it may produce 01 Kg of ice for an adsorbent heating from 40°C to 100°C according to the solar irradiance and the ambient temperature. The considered lower temperature of the adsorption is 10°C, which may allow producing 09 Kg of ice for higher temperature of the solar collector. The considered higher temperature of the approximation is 10°C, which may allow producing is 10°C, which may allow producing is 10°C, which may allow producing 3.5 Kg of ice for higher temperature of the generating up to 144°C, and this needs 622 KJ of solar irradiance for 01 m² of the solar collector.



Fig. 1. Generating temperature of carbon AC35 nanomaterial versus the daily global irradiance for different values of adsorption temperature.

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V.PO.3

CONDUCTION COMPONENT OF DIELECTRIC LOSSES IN FERROELECTRIC PHASE OF TGS

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Keywords: Triglycine sulphate crystals, relaxation mechanism, temperature dependence of conduction

Abstract: Triglycine sulfate crystal (TGS for short) is a ferroelectric with a second order transition at 49.2 °C. It is the most sensitive material used for IR detection, i.e. almost one order of magnitude higher sensitivity than other materials. In this contribution we present the frequency dependence of TGS conduction at temperatures in ferro and in paraelectric phase. The temperature dependence of conduction is very high near the Curie point and become almost insignificant towards zero Celsius or down. There are two type of losses and only the conduction losses has been estimated with the specific program of Novocontrol-Alpha Analyzer on the frequency range $(1-10^7)$ Hz and the temperature range from 65 °C in para phase to -40 °C, crossing down the Curie point. Temperature dependence of conduction in para / ferroelectric phase was analyzed and the activation energy was estimated.



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V.PO.4

MAGNETIC MATERIAL WITH NANOSIZE DIMENSIONS: SYNTHESIS, CHARACTERIZATION AND TESTING

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Keywords: nanostructured material, scanning electron microscopy, X-Ray Diffraction, Atomic-force microscopy, heavy metals

Abstract: The aim for this study is to present the synthesis, characterization and removal efficiency of a magnetic nanostructured material with potential to reduce heavy metals. The nanostructured material was obtained by co-precipitation method. In order to avoid dissolution of the obtained nanostructure an acidic resin was used as material protecting. The structure and morphology of the material was examined by scanning electron microscopy (SEM), Atomic-force microscopy (AFM) and X-Ray Diffraction Spectrometry. The aim of this material preparation was to demonstrate the applicability of such nanostructured material. Testing for removal of heavy metals such as Cd, Pb and Zn was performed together with testing of some standard materials such as acidic resin and magnetic nanomaterial. In order to establish the mechanism of heavy metals removal, the structure and surface properties were studied before and after testing. The results demonstrate that synthesis nanostructured material was a good option for removal at approximately 90% of heavy metals.

Acknowledgements: The work has been funded by the PNCDI III Program, Program 2: "Increase of the competitiveness of the Romanian economy through research, development and innovation" through the Financial Agreement ID Project: 7PTE / 2016.







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V.PO.5

A NOVEL ROUTE FOR THE PREPARATION OF CoCr₂O₄/SiO₂ NANOCOMPOSITE STARTING FROM Co(II) - Cr(III) CARBOXYLATE COMPLEX COMBINATIONS

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Keywords: cobalt chromite, nanocomposite, modified sol-gel method, thermal stability

Abstract: This study reports a summary on the preparation and characterization of cobalt chromite nanoparticles embedded in silica matrix [1]. We have used the original, modified sol-gel method for the preparation of $CoCr_2O_4/SiO_2$ spinel nanocomposites. Thus, we have prepared gels with different $CoCr_2O_4/SiO_2$ ratios (20 and 50 wt% $CoCr_2O_4$) starting from Co(II) nitrate, Cr(III) nitrate, tetraethylortosilicate (TEOS) and 1,3-propanediol (1,3PG). The obtained gels were dried at 40°C and heated at 150°C, when the redox reaction took place between 1,3PG and the nitrate ions (NO₃⁻). In the reaction resulted a Co(II) - Cr(III) carboxylate type complex dispersed within the gels matrix, which was further used as precursor in the preparation of $CoCr_2O_4/SiO_2$ [2,3]. The heat treatment of the samples obtained at 150°C was carried out at temperatures in the range 300 – 1000°C in order to establish the evolution of the phase composition and crystallite size. The Co(II) - Cr(III) carboxylate type complex decomposed at 300 °C leading to the corresponding metal oxides (amorphous Cr_2O_{3+x} , CoO), which are uniformly distributed inside the silica matrix. On further heating at 400°C, Cr_2O_{3+x} turns to α -Cr₂O₃ which interacts with CoO resulting in the desired spinel phase (CoCr₂O₄).

The samples were characterized by thermal analysis, FTIR and XRD. The mean particle size was in the range 5-20 nm.

The results showed that the modified sol-gel method can be used effectively for the preparation of $CoCr_2O_4/SiO_2$ nanocomposites.

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V.PO.6

REMOVAL OF PHENOLIC DERIVATIVES FROM WATER BY CROSSLINKED β-CYCLODEXTRIN

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Keywords: cyclodextrin; adsorption processes; phenol derivatives; water treatment.

Abstract: Phenols are widely encountered in surface water as a result of spilling from various industrial sources and due to toxicity to aquatic environments these pollutants must be removed. Several methods can be applied for water cleaning and a good candidate is represented by adsorption processes. In order to maximize the removal efficiency new type of adsorbents must be prepared [1-3].

This work reports on the preparation of β -cyclodextrin polymers and the removal of several phenol derivatives by prepared solids. The characterization of the prepared bio-polymers was accomplished using X-ray diffraction, Fourier Transform Infrared Spectroscopy, Raman spectroscopy, electron microscopy and thermal analyses. The removal of phenols derivatives from aqueous solution raw industrial phenolic wastewater with the β -cyclodextrin was carried out either in a batch mode or in fixed bed mode. Influence of several important parameters (temperature, pollutant concentration, adsorbent dose etc.) was also assessed [1, 2].

The prepared adsorbent show good sorption capacities toward phenols. The monolayer adsorption capacity was around 76.5 mg/g. The adsorption capacity increased as the dosage of the material increased. The straight lines in plot of t/qt versus t showed good agreement of experimental data with the second order kinetic model for different initial sorbent concentration.

The results showed that the experimental data were correlated reasonably well by the Freundlich adsorption isotherm.

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V.PO.7

TUNED APATITIC MATERIALS: SYNTHESIS, CHARACTERIZATION AND POTENTIAL APPLICATIONS

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Keywords: apatitic materials; synthesis; characterization; antimicrobial properties; anti-biofilm activity.

Abstract: Inorganic antimicrobial materials are usually made of metal ions having biocide action and a substrate. Copper and silver are known to be one of the multifunctional inorganic nanoparticles with antibacterial activity [1]. Among the substrates, one of the calcium phosphates, synthetic hydroxyapatite (HAP, $Ca_{10}(PO_4)_6(OH)_2$), is the most promising, mainly due to its high affinity for the pathogenic microorganisms. These materials can be viable for multiple applications (related to its use for new buildings with special requirements related to microbiological loading, such as hospital buildings and for consolidation of cultural heritage constructions); also the use of substituted hydroxyapatites for protection of stone artefacts against environmental factors (acidic rain) and biodeterioration it's an option to no longer use of toxic substances [2].

This paper presents methods of synthesis and characterization of the material from the point of view of the obtained structures and final applications. The materials were characterized using modern analytical techniques (XRD, XRF, ICP-AES, TEM). Antimicrobial activity was tested against filamentous fungi strains and pathogenic bacteria strains, using both spot on lawn qualitative method (on agar medium) and serial microdilution quantitative method (in broth medium). Further, it was evaluated the anti-biofilm activity of the tested samples toward the most important microbial strains implicated in biofilm development, using crystal violet stained biofilms microtiter assay, followed by spectrophotometric quantitative evaluation [3].

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V.PO.8

PHOTOVOLTAIC PERFORMANCE ENHANCEMENT OF HYDROTHERMALLY GROWN ZNO NANORODS FOR DYE SENSITIZED SOLAR CELLS: THE EFFECTS OF SEED LAYER

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Keywords: ZnO, nanorods, hydrothermal process, seed layer effects

Abstract: One-dimensional ZnO is among the most promising nanostructures due to their exceptional properties in wide range of applications such as electronic, optoelectronic, electrochemical, electromechanical and photoelectrochemical devices [1]. Dye sensitized solar cell as the third generation of solar photovoltaic device has attracted considerable interest during the past two decades, due to its low fabrication cost, simple manufacturing process and higher energy conversion efficiency. ZnO has been considered as one of the most proposed photo-anode materials [2] with wide bandgap (3.37 eV) and high exciton binding energy (60 meV) [3]. A perfect nano-structure of ZnO is required, which could provide the direct pathway for electron transmission. The typical one dimensional nano-structure of ZnO such as nanowires, nanorods and nanotubes, which can be beneficial to electron transport and can reduce the probability of charge recombination [2]. The ZnO nanorods have been synthesized by hydrothermal method. The seed layer properties are vitally important to control the structural, morphological, and optical features of the ZnO nanorods [1]. In this study, the effects of seed layer on the growth of ZnO nanorods during hydrothermal process and also on the photovoltaic properties of ZnO nanorod-based dye sensitized solar cells have been examined. The ZnO seed layers have been deposited on FTO coated glass substrates by sol-gel dip coating and preheated at 400°C. Zinc acetate dihydrate as precursor, monoethanolamine as an additive and ethanol as an solvent have been used to provide the sol to synthesize the seed layers. ZnO nanorods have been produced by hydrothermal route from precursor solution contains Zinc nitrate, hegzamethilenetetramine and water. General morphologies and detailed structural characterizations have been obtained by using scanning electron microscope, X-ray diffractometer, Raman spectroscopy, UV-Vis spectroscopy. Open-circuit photovoltage measurements have been performed to investigate the photoelectrochemical characteristics of ZnO nanorod-based dye sensitized solar cells.

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V.PO.9

GRAPHENE SUPPORTED α -MnO₂ CATHODES FOR Li-O₂ BATTERIES

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Keywords: Graphene, MWCNT, Manganese dioxide, Free-standing, Li-air batteries

Abstract: In this study, α -MnO₂/MWCNT/Graphene nanocomposite electrodes were produced by a facile method as flexible and free-standing. Graphene oxide was produced by Hummer method and reduced to graphene with hydrazine hydrate solution. Multi-wall carbon nano tubes were added at 1:1 (w_{GO}/w_{MWCNT}) ratio between graphene layers to prevent from aggregation of graphene layers and increase interlayer distance, surface area and pore volume. α -MnO₂ was synthesized as nanorods by using KMnO₄ and MnSO₄. A series of α -MnO₂/MWCNT/Graphene nanocomposite containing different values of α -MnO₂ were produced and used as electrode for Li-O₂ batteries. The structure and morphology of the nanocomposite electrodes were characterized by X-ray diffraction, scanning electron microscopy and N₂ adsorption/desorption. The electrochemical characterization tests including galvanostatic charge/discharge, cyclic voltammetry and electrochemical impedance spectroscopy (EIS) measurement of the electrodes were carried out by using an ECC-Air test cell.

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V.PO.10

INFLUENCE OF SURFACTANTS ON THE CHEMICAL SYNTHESIS OF Cu-DOPED ZnO NANOPARTICLES

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Keywords: Cu doped ZnO, nanoparticles, chemical synthesis, surfactants.

Abstract: These last years, techniques for nanomaterials synthesis have been a topic of considerable scientific interest in materials science, due to the necessity to obtain some materials with controllable properties (size distribution, morphology, composition of nanoparticles, etc.). Nowadays, there is a continued concern to intensify the research activity, in order to obtain some materials with improved properties for applications in the various fields of activity for domestic and industrial uses.

In the present work, Cu-doped ZnO nanoparticles were synthesized using a relative and simple chemical co-precipitation method with zinc nitrate $(Zn(NO_3)_2)$, copper acetate $(Cu(CH_3COO)_2)$ as basic precursors, sodium hydroxide (NaOH) as precipitator material, in the absence and presence of cationic and non-ionic polymer surfactants, such as cetyl trimethylammonium bromide (CTAB), poly(ethylene oxide) (PEO). The characterization of powders samples was carried out by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and Fourier transform infrared spectrometry (FT-IR). From the results obtained, it can be considered that the surfactants have a significant influence on the physical and chemical properties. Also, the use of surfactants in synthesis of particles suggests a control over surface morphology, size, degree of agglomeration and chemical structure of the particles to yield specific properties.

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V.PO.11

PHOTOVOLTAIC PERFORMANCE OF ULTRASMALL Fe DOPING CuCrO₂ NANOPARTICLES FOR P-TYPE DYE-SENSITIZED SOLAR CELLS APPLICATION

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Keywords: delafossite materials, 2H-CuCrO₂, DSSCs, hydrothermal method

Abstract: Delafossite materials like CuCrO₂ are promising p-type semiconductor materials [1], because they have an excellent optical transparency in the visible range and high electrical conductivity, a very promising alternative to NiO in p-type dye-sensitized solar cells (DSSCs). In this paper, we report the successful hydrothermal synthesis of Fe³⁺ doping nanocrystalline 2H-CuCrO₂ with very small size and a high surface area of 151 m²/g for undoped CuCrO₂. The specific surface area of 2H-CuCrO₂ nanocrystalline is the biggest ever reported compared with other materials with delafossite structure used for dye sensitized solar cells (DSSCs) applications. Until now, the low circuit density (Jsc) for CuCrO₂ was still too low for the commercial applications, because of the low surface areas which limited dye adsorbing amounts. Therefore, it is crucial to explore more on the synthesis of delafossite CuCrO₂ materials with small size and their application features in p type-DSSCs. The structure of Fe³⁺ doping nanocrystalline 2H-CuCrO₂ has been determined by X-ray diffraction (XRD) and high-resolution transmission electronic microscopy (HRTEM). Optical measurements attest of the quality of the material, in line with the literature. The performance of p-type DSSCs based on Fe³⁺ doping nanocrystalline 2H-CuCrO₂ photocathode was made using P1 dye and I⁻/l₃⁻ dissolved in acetonitrile as redox couple.

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V.PO.12

Low-temperature hydrothermal synthesis of the copper metaborate CuB₂O₄

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Keywords: delafossite materials, CuB₂O₄, hydrothermal method

Abstract: In order to obtain the copper metaborate CuB_2O_4 by hydrothermal synthesis, the yield diagram for Cu-B-H₂O system has been built and studied. We had first synthesized directly the copper metaborate CuB₂O₄ by a one-step low-temperature hydrothermal method. The soft hydrothermal synthesis method offers the lower synthesis time and a higher purity in comparison with other synthesis methods. The tetragonal structure of CuB2O4 has been determined by X-ray diffraction (XRD) and high-resolution transmission electronic microscopy (HRTEM). Optical measurements attest of the quality of the material, in line with the literature. The thermal stability of CuB₂O₄ has been investigated by means of high temperature X-ray diffraction and DTA-TG. In order to obtain the copper metaborate CuB_2O_4 by hydrothermal synthesis, the yield diagram for $Cu-B-H_2O$ system has been built and studied. We had first synthesized directly the copper metaborate CuB₂O₄ by a one-step low-temperature hydrothermal method. The soft hydrothermal synthesis method offers the lower synthesis time and a higher purity in comparison with other synthesis methods. The tetragonal structure of CuB_2O_4 has been determined by X-ray diffraction (XRD) and high-resolution transmission electronic microscopy (HRTEM). Optical measurements attest of the quality of the material, in line with the literature. The thermal stability of CuB₂O₄ has been investigated by means of high temperature X-ray diffraction and DTA-TG.

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V.PO.13

MODULATION OF TVA DEPOSITION PARAMETERS FOR THE Fe-Ni-Cu-Ta THIN FILMS INTERDIFUSIVITY AND GMR EFFECT INFLUENCE

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Keywords: thin film, GMR effect, interdifussion, surface parameters

Abstract: Thin layers with GMR effect are lately analyzed due to their application possibilities in high tech fields like spintronic. The achievment of notable performances is conditioned by the fine control of layers' deposition parameters, regardless the chosen obtaining method. In this paper, this issue is addressed from the perspective of the thermionic vacuum arc (TVA) obtaining method for multilayers (cooper, nickel and iron with tantalum layer deposition) with GMR effect. Multilayered structures were obtained from layers with 3 and 5 nm thickness. The most important tuned parameter for the layers' structure and composition modification (by interdiffusion) was the energy of the impinging adatoms. The layers with total thickness between 12 and 20 nm were characterized by high resolution complementary methods for the determination of composition, morphology, structure, electric properties and GMR effect: SEM (in cross-section), AFM (surface parameters), XRD (structure by grazing incidence determination), STEM/EELS (cross-section layers' line profile and mapp), HRTEM (nanolayers internal structure), MOKE (magnetorezistance). The results analyze led to the conclusion that the thin layers' magnetic anisotropy is highly influenced by the microstructure and the interfacial effects like roughness, interdiffusion and layers thickness. The interface general profile spreads into the entire film thickness and highlights the substrate importance in the morphology of the deposited layer. Due to the high asymmetry on atomic mobility (Ni atoms diffuse more rapidly in Cu areas than the Cu atoms in those of Ni) and function of deposition selected parameters, a layer by layer or interdiffused growth, can be observed.

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V.PO.14

PHYTO-MEDIATED METALLIC NANO-ARCHITECTURES VIA *Melissa officinalis* L.: SYNTHESIS, CHARACTERIZATION, CYTOTOXICITY AND ANTIMICROBIAL PROPERTIES

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Keywords: *Melissa officinalis*; metallic nano-architectures; characterization; cytotoxicity; antimicrobial properties.

Abstract: Development of obtaining techniques for different materials with antimicrobial properties, based on green chemistry principles has been the target of research over the past years. In this context, metallic nanoparticles (either single metal or bimetallic) proved to be a viable solution for several applications [1]. The present paper describes the phyto-mediated synthesis of metallic nanoarchitectures (gold, silver) via the ethanolic extract of *Melissa officinalis* L. (obtained by accelerated solvent extraction). Analytic methods (XRD, XRF, UV-Vis, electron microscopy, chromatographic techniques – GC-MS, HPLC) were applied for the evaluation of the extract composition, as well as for the characterization of the phyto-synthesized materials. The cytotoxicity of the synthesized materials was evaluated by *Allium cepa* assay [2], while the antimicrobial activity was evaluated using both qualitative and quantitative methods. Qualitative screening was performed by diffusion method (adapted from CLSI standard methods) and quantitative analysis was performed by binary serial micro-dilution method in liquid medium, in order to determinate the Minimal Inhibitory Concentration values. Microbial strains included in the present study belong to different genera and species: molds *Aspergillus niger* and *Trichoderma viride*, yeast strains *Candida albicans* and *non-albicans*, bacteria strains *Staphylococcus aureus*, *Escherichia coli*, *Pseudomonas aeruginosa* strains.

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V.PO.15

SYNTHESIS AND CHARACTERIZATION OF CeO₂ DOPED ZrO₂ CERAMICS BY A SIMPLE SOL-GEL ROUTE

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Keywords: Sol-gel preparation, ZrO₂, CeO₂, Characterization

Abstract: It is well known that sol-gel technique is a simple method to produce nano sized ceramic powders [1,2]. In this study, cerium oxide doped zirconia samples, with 10 mol%-12mol% and



Fig. 1. ZrO₂ nano-sized powders

14mol% CeO₂, were synthesized by solgel technique and characterized. The surface morphology, elemental composition, microstructure, and phase analysis, of the sintered CeO₂ doped ZrO₂ ceramics were characterized by field emission scanning electron microscopy (FE-SEM), energy dispersive X-ray (EDX) analysis, and X-ray diffraction (XRD) respectively. X-ray diffraction for samples sintered at 1550 °C for 4h revealed that the zirconia ceramics have a cubic phase structure. The addition of CeO₂ can raise the content of the cubic phase, but the minor metastable tetragonal phase exists even at the CeO₂ content as high as 10 mol%. The effect of dopant concentration on the lattice parameter, average primary

crystallite size and microstrain was studied. Relative densities for CeO_2 doped ZrO_2 ceramics varied between 95% and 99 %, depending on the CeO_2 addition.

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V.PO.16

SOLAR RADIATION SYNTHESIS OF FUNCTIONAL CARBONACEOUS MATERIALS USING Al₂O₃/TiO₂-Cu DOPED CATALYST

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Keywords: solar synthesis, carbonaceous materials, catalyst

Abstract: Besides the traditional methods, solar synthesis of carbonaceous materials (CM) through solar vapor deposition represents a promising method due to the inexpensive solar energy. One of the major advantage of using the solar reactor for CM synthesis is the scaling-up possibilities which could be reflected in the productivity and the price of the CMs. Functional carbon based micro- and nanomaterials have attracted great interest due to their unique optical, electrical, energy-storage and catalytic properties. Compared to inorganic-based materials, carbon materials provide a wide variety of structures and textures, are processed more easily and possess higher compatibility with other types of materials.

Several studies present the synthesis of carbonaceous materials using solar radiation. Flamant et al. [1, 2] presents the synthesis of fullerenes in a solar reactor and reports a pronounced effect of pressure and gas flow rate on the reaction process. Furthermore, he optimized the design the solar reactor for carbon product processing by vaporization. Alvarez et al. [3] report that the catalyst type does not influence the growth mechanism of single wall carbon nanotubes but has a clear influence on their structure, electronic properties and yield.

Our work describes the obtaining of different types of functional carbon particles starting from graphite and Pd/C targets using an alternative and "green" approach, namely solar vapor deposition on the surface of a novel Al_2O_3/TiO_2 -Cu doped catalyst, obtained by thermal spraying on a copper base material, through varying the synthesis parameters (reactor temperature, gas flow, etc.).

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V.PO.17

SYNTHESIS AND CHARACTERISATION OF CREDNERITE CuMn_{1-x}B_xO₂ (B=Mg, Al; x=0 - 0.08) TYPE MATERIALS

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Keywords: Transition metal oxides, synthesis, structures, electrical and magnetic properties.

Abstract: There is a renewed interest for delafossite type oxides (with ABO₂ formula) due to their potential applications in the domains of transparent p-type oxides [1], oxygen storage [2] etc. The ABO₂ delafossite structure, where A=Cu and Ag, and B a transition element, belongs to the R-3m space group and is characterised by O-Cu-O dumbells linking layers of edge sharing BO₆ octahedra. However, in this class of materials, crednerite CuMnO₂ occupies a unique place due to the Jahn-Teller (JT) distortion of the Mn^{3+} ($t_{2g}^{3}e_{g}$) which leads to a monoclinic structure (C2/m space group at room temperature) and to a different topology of the magnetic triangular lattice and out-of-plane stacking sequence compared to delafossite structure. In this research work, a series of $CuMn_{1-x}B_xO_2$ (B=Mg, Al; x=0 - 0.08) crednerite nanomaterials were prepared by using hydrothermal synthesis. The effect of Mg²⁺ and Al³⁺ substitution on the structural, morphological, optical, thermal and magnetic properties of crednerite samples was investigated. It has to be mentioned that for these compounds beside the size effect, the disorder induced by the cations with different sizes and electronic configurations sitting on the same crystallographic site play an important role. X-Ray diffraction study showed that the solubility limit was around 4% with a very distinct behavior of unit cell parameters in function of x. Transmission electron microscopy indicated that the average crystallite size decreased with increasing x for both Al and Mg substitution samples. Moreover, the susceptibility curves, obtained from magnetization measurements, show that the obtained samples present an interesting magnetic behavior, different from the CuMnO₂ polycrystalline samples (obtained by high temperature solid state reaction).

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V.PO.18

DEVELOPMENT OF BROADBAND ULTRASONIC SPECTROSCOPY FOR COLLOIDAL SUSPENSIONS CHARACTERIZATION

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Keywords: ultrasonic spectroscopy, FFT, colloidal suspension, ferromagnetic particles,

Abstract: The progress in computing and digital techniques transformed ultrasonic spectroscopy from a complicated measuring procedure to a conventional method for materials characterization [1,2]. More recently the intense development of drug-delivery products drew a particular interest for using ultrasonic spectroscopy as a characterization tool in the pharmaceutical industry [1]. Furthermore, its general advantages can be extrapolated for appropriate characterization of optically opaque and highly concentrated dispersed systems without sample alteration [3,4]. This study targeted the development of broadband ultrasonic technique for characterization of colloidal suspensions based on nanometric and micrometric magnetic particles. The results were obtained with pulses generated by broadband transducers of various spectral content using the pulse-echo technique. The spectral characteristics were obtained by Fast Fourier Transformed analysis (FFT) of the echo signals received from the cell bottom containing the liquid suspension after one sonic path (distilled water was used as reference system). For all broadband transducers and all applied input pulses respectively, results concluded that only limited spectral response can be received and used for spectral analysis. This outcome contributes to further key-investigations and clarification of encountered experimental limitations and to find solutions to increase the sensitivity and spectral range of broadband ultrasonic spectroscopy.

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V.PO.19

NANOAGGREGATES OBTAINED FROM ECO-FRIENDLY SILVER NANOPARTICLES AND PHTHALOCYANINE DERIVATIVES

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Keywords: nanoaggregates, eco-friendly silver nanoparticles, phthalocyanines, photophysics

Abstract: Silver nanoparticles (AgNPs) are nowadays among the most studied noble metal nanoparticles especially since the eco-friendly methods used to obtained them gained more and more importance. Therefore, different plants and plant parts are used to obtain silver nanoparticles



Figure 1. The chemical structure of AgNPs - Pcs

by mixing their aqueous extracts with silver nitrate (AgNO₃) 10^{-3} M solution [1]. Phthalocyanines (Pcs) are very versatile macrocyclic compounds with excellent photoactive properties and various applications in different scientific field. The conjugation between silver nanoparticles and phthalocyanine derivatives (Figure 1) enhances the photochemical activity of Pcs on one hand and increases the stability of AgNPs on the other hand [2].

This paper presents the biosynthesis of AgNPs from *Sea buckthorn* aqueous extract, the synthesis of tetracarboxamido-zinc phthalocyanine

 $ZnPc(CONH_2)_4$ and octacarboxamido–zinc phthalocyanine $ZnPc(CONH_2)_8$ and their physical – chemical characterization. These two components are further used to obtain the two nanoaggregates whose formation is confirmed by means of spectroscopic analyzes (e.g.: UV-Vis, FTIR, DLS, SEM, TEM, XRD).

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V.PO.20

THE USE OF GOOSEBERRY AND CHOKEBERRY FRUITS FOR THE SYNTHESIS OF **METALLIC (Au, Ag, Pt) NANOPARTICLES**

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Keywords: metallic nanoparticles, fruits, antioxidant activity

Abstract: Scientific studies have demonstrated that the vegetable material extracts act as potential precursors for the synthesis of nanomaterial using eco-friendly ways. Due to the fact that the plant extracts contains various secondary metabolites, they act as reducing and stabilizing agents for the



Fig. 1. a) Gooseberry (Ribes uva-crispa) and b) Chokeberry (Aronia melanocarpa) dried fruits

bioreduction reaction for synthesis of novel metallic nanoparticles. In the present study, are presented some of our work results in the field of nanomaterial phytosynthesis, using extracts obtained from different types of native fruits (Ribes-uva-crispa and Aronia melanocarpa). The total content of phenolics, flavonoids, tannins and terpenoids in the fruits, have been determined and correlated with the antioxidant activity (using DPPH method). The green method for obtaining

metallic nanoparticles (PtNP, AgNP, AuNP) and the nanoparticles investigated by spectral methods (UV-VIS, FTIR, RAMAN) are shown in this research.

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V.PO.21

REMOVAL OF HEAVY METALS FROM SINGLE- AND BINARY-METAL AQUEOUS SOLUTIONS USING HYDROXYAPATITE NANO-ADSORBENTS

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Keywords: metallic toxic heavy metals, removal, lead, synthetic wastewater

Background and aims: Environments contaminated with metals have been extensively studied in recent years due to the negative effects of the heavy metals on living organisms (plants, animals, humans) [1, 2]. The aim of present study was to investigate the up-take capacity of different hydroxyapatite nanoadsorbents, from single and binary-metal synthetic aqueous solutions. *Methods:* HAP nanoparticles (n-HAP) were synthesized on the laboratory scale by a wet chemical precipitation method. Batch experiments were carried out using different synthetic nano-hydroxyapatites. Powders were tested for lead/zinc removal process from aqueous solutions, under different conditions, i.e. initial metal ion concentration, pH of the solutions and contact time. The experimental data of metals removal were analysed using the pseudo-first and pseudo-second order models. **Results:** Our results shown that ion adsorption capacity increased with decreasing crystallinity and increasing specific surface area. The kinetic experimental data were fitted by both pseudo-first, and pseudo-second order models. The thermodynamic equilibrium sorption experimental data are well fitted by Freundlich adsorption isotherm for all tested adsorbents, better than Langmuir's. Conclusions: Preliminary experiments for testing the uptake ability of the sorbents for Pb²⁺/Zn²⁺ ions primarily showed the uptake capacity dependence with physico-chemical and morphological characteristics of the sorbents. These findings are in accordance with the literature data. The contact time of approximately 300–360 min was necessary to reach the thermodynamic equilibrium, and the optimum pH was 6.0-6.5. These results showed that all nano-adsorbents evaluated exhibited high adsorption capacity, and can be successfully used to remove heavy metals from wastewater.

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V.PO.22

Si@Void@Carbon Yolk-Shell Anodes for Lithium Ion Batteries

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Keywords: silicon, lithium-ion, yolk-shell

Abstract: Rechargeable lithium-ion batteries are energy storage devices which are the most widely utilizable in electronic market due to their many advantages. Although attaining great field of usage, they have insufficient sides such as answering the high energy density and good rate performance needs of electrical vehicles [1]. Silicon is one of the most considerable candidates which can alter the performances of lithium-ion batteries since it has the highest theoretical lithium storage capacity (4200 mAh.g⁻¹) and when compared with commercial carbon anodes (372 mAh.g⁻¹) silicon becomes obviously superior over the currently used lithium-ion batteries negative electrodes [2]. Despite of theoretically excellent in terms of capacity values, vital drawback hinders silicon anodes to be commercially applicable. During cycling, huge volume variations occur (> 300 %) which causes cracking and pulverization of anodes, consequently leading the loss of electronic contact and rapid capacity fade followed by failure of anode. In order to overcome mentioned problems there are many efforts have been devoted by researchers such as using amorphous silicon, thin films, reinforcing nanotubes and nanowires etc... which were not come up with solution for capacity fading of silicon anodes so far [3,4]. In this work we present a study in order to refer a solution or improvement for silicon anodes via yolk-shell structures which is presented in figure 1 that have space to accommodate volume variations during lithiation .



Figure 1 . Demonstration of yolk-shell structure and its production procedure.

X-Ray diffraction analysis and RAMAN spectroscopy techniques were used for phase characterization of anodes. Scanning electron microscopy analyses were conducted for morphological characterization. The electrochemical performance tests were carried out by means of galvanostatic charge/discharge, cyclic voltammetry and electrochemical impedance spectroscopy.

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V.PO.23

CONTROLLING SELF-ASSEMBLING AND THERMOCHROMISM OF POLYDIACETYLENE/ZINC OXIDE NANOCOMPOSITE BY VARYING PH

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Keywords: polydiacetylene, color transition, temperature sensor, self-assembling, surface charge

Abstract: This research presents a simple method to control thermochromic behaviors of polydiacetylene (PDA)/zinc oxide (ZnO) nanocomposite [1-3]. Surface charge of ZnO nanoparticle and degree of dissociation of the carboxylic head group of diacetylene (DA) monomer are altered by adjusting pH of the aqueous suspension. This directly affects assembling of DA on ZnO nanoparticles, and then, concentration and thermochromic behaviors of PDA/ZnO nanocomposite after photopolymerization. Concentration of the nanocomposite prepared using 10,12-tricosadiynoic acid (TCDA) is found to increase with increasing pH of ZnO suspension up to 11. The poly(TCDA)/ZnO nanocomposites prepared from ZnO suspensions of pH 7 to 11 change from blue to red at ~60 °C and exhibit completely reversible thermochromism. On the contrary, ZnO suspensions of pH 1 to 5 yield the nanocomposite with lower color transition temperature and irreversible thermochromism.

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V.PO.24

PROPERTIES OF THE YELLOW EMITTING PHOSPHORS FOR WHITE LED MANUFACTURING

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Keywords: yellow phosphor, YAG:Ce, TAG:Ce, white LED, sol-gel process

Abstract: The phosphors as luminescent materials with applicability in emissive optoelectronics have been important from an industrial viewpoint with the introduction of fluorescent lamps, and experienced a spectacular comeback with the first white LED. Although there are numerous studies in the field of phosphors, achieving different types of luminescent materials withemission in red, green, blue, etc., the yellow phosphors are the only that have proven industrial applicability in the development of white LEDs.

In this work we report obtaining of the A₃B₂C₃O₁₂ yellow phosphors doped with rare-earth based onyttrium and terbium respectively. Cerium-doped terbium aluminum garnet (TAG:Ce) and cerium-doped yttrium aluminum garnet (YAG:Ce) were synthesized by sol-gel method and sintered at 1100°C. The morphology, chemical structure and luminescent properties of phosphors were studied. For the chemical structure study, the Fourier transform infrared spectrometry was used to determine the bond configuration, X-ray diffraction for phase identification and energy dispersive X-ray spectroscopy for the elemental composition analysis. This study demonstrated the transition from amorphous to the crystalline phase and the formation of M-O bonds in the garnet structure. The presence of the dopant in the crystalline structure was also demonstrated. Scanning electron microscopy was used for morphology and microstructure study of the phosphors showing nanostructured particles with smooth surfaces. The luminescent properties were studied by fluorescence spectroscopy indicating YAG and TAG may be used as materials to generate white light in an LED.

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V.PO.25

INFLUENCE OF CdS LOADING UPON TYPE PdS/CdS-ZnS PHOTOCATALYSTS

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Keywords: photocatalysis, PdS/CdS-ZnS, water splitting, hydrothermal method

Abstract: CdS based photocatalysts are today the most efficient photocatalysts for the water splitting reaction in the presence of sacrificial agents [1] when are using the blue light region of solar spectrum. When CdS is in intimate contact with ZnS, the photocatalysts efficiency may increase due to a better separation of hole-electron pair [2, 3]. In our previous work [4] we proved that when PdS / CdS-ZnS type photocatalysts are obtained by hydrothermal synthesis using ZnS as sulfur source, the photocatalysts efficiency is depending on the precursor crystallinity. In the aforementioned system PdS serves as a co-catalyst. In the present study we aimed at evaluating the efficiency of PdS / CdS-ZnS type photocatalysts for various degrees of co-catalyst loading. Reaction product morphology was assessed by SEM/EDX analysis, phase/composition identification by XRD and the band gap value was determined from UV-VIS-NIR spectra. Photocatalysis experiments were conducted using simulated sunlight as a source of radiation.

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V.PO.26

LOW-COST SYNTHESIS OF PdS / Zn_{1-x}Cd_xS USING BATTERY INDUSTRY WASTE

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Keywords: photocatalysis, water splitting, hydrothermal method

Abstract: Hydrogen energy has been regarded as the most promising energy resource in the near future due to that it is a clean and sustainable energy [1]. Until now, cadmium sulfide (CdS) is the most representative material, for hydrogen production in visible light. In this paper we present a simple process for the inexpensive production of a high crystallinity $Zn_{1-x}Cd_xS$ and PdS / $Zn_{1-x}Cd_xS$ with high efficiency using hydrothermal method. The Cd precursor from battery industry waste, the low temperature (up to 200 ° C) and hermetic synthesis system (teflon autoclave) have provided a low-cost and ecological production of $Zn_{1-x}Cd_xS$ and PdS / $Zn_{1-x}Cd_xS$.

The structure of $Zn_{1-x}Cd_xS$ and PdS / $Zn_{1-x}Cd_xS$ has been determined by X-ray diffraction (XRD), the morphology was assessed by TEM / STEM analysis, and the band gap value was determined from UV-VIS-NIR spectra. Photocatalysis experiments were conducted using simulated sunlight as a source of radiation.

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Acknowledgements : This work was carried out through the Partnerships in priority areas - PN II program, developed with the support of MEN - UEFISCDI, project no. PN-II-PT-PCCA-2013-4-1708.



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V.PO.27

BIOSYNTHESIS OF ZINC OXIDE NANOPARTICLES USING ENTEROBACTER

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Keywords: biosynthesis, Enterobacter, zinc oxide nanoparticles;

Abstract: In this study, zinc oxide nanoparticles (ZnO NPs) were synthesized through biosynthesis



Fig. 1 SEM image of synthesized ZnO NPs

using microorganisms. $Zn(NO_3)_2$ was used as zinc oxide precursor. Nanoparticles are synthesized when the *Enterobacter* grabs target ions $(Zn^{2+} - O^{2-})$ from their medium and then turn the metal ions into the element metal, through the enzyme generated by the cell activities. The synthesis of zinc nanoparticles has taken place extracellulary according to the location where nanoparticles were formed [1].

The synthesized ZnO NPs were characterized by ultraviolet-visible spectroscopy which shows a peak at 368 nm. Energy Dispersive Spectroscopy analysis confirms the elemental composition of ZnO nanoparticles. Scanning Electron Microscopy presents images of ZnO nanoparticles, revealing their size smaller than 100 nm and with a spherical shape

(Fig.1). The size and shape of the nanoparticles vary with: the microorganism that can be employed, and by the conditions, like pH, temperature and precursor concentration.

These ZnO NPs are safe and eco-friendly and may have potential applications in medicine, cosmetic industry, and biochemical sensors [2]. The challenges during biosynthesis include optimal production and minimal time to obtain the desired size and shape, to enhance the stability of nanoparticles and optimization of specific microorganisms for specific application.

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SECTION VI Welding Engineering

Chairpersons:

Dragoș BUZDUGAN, Politehnica University of Timișoara, ROMANIA **Radu IOVĂNAȘ**, Transilvania University of Brasov, ROMANIA



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VI.O.1

CHARACTERIZATION OF FUSION LINES OBTAINED BY LASER WELDING ON PREHEATED DUCTILE IRON PLATES

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Keywords: Ductile iron, laser welding, scanning electron microscopy, spectrometry, diffractometry, microstructure, microhardness.

Abstract: Ductile iron (DI) is a type of cast iron, invented in 1943 by Keith Millis, and researchers are concerned to discover new joining methods. Qiang Wu et al [1] have studied the variation of deep penetration and width by using CO₂ laser on high- strength galvanized steel. For achieving the tests they used two types of shielding gas (Ar, N₂) and variation of laser power- welding speed. N. Farabi et al [2] have studied the relation between mechanical properties and microstructure from heat affected zone. For achieving the tests laser welding technology was used on two types of steels. The present research is dedicated to weldability of DI using laser welding technology. The fusion lines were done using an Yb: YAG continuous laser, with a maximum power of 6 kW, the samples were preheated at temperatures between 200°C and 250°C using a heating plate. During this tests, have been tested several parameters to identify their influence on compactness seams, geometry of fusion lines or even the behaviour of mechanical point of view. Each fusion line obtained was examined visually and was used X-ray (EDS) to determine the chemical composition of samples. Vickers method was used to determine microhardness in three specific areas: base material, heat affected zone and deposited metal. After experimental tests on DI plates results the power-welding speed diagram for possible domains of laser welding.

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VI.O.2

CONTINUOUS WAVE LASER WELDING OF COPPER

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Keywords: laser welding, copper

Abstract: The increase in the demand for materials with good mechanical strength, high corrosion resistance, electrical and thermal conductivity has attracted the interest in using copper and its alloys as contact material or layer in almost all electrical-bearing components of batteries and micro-contacts applications. The disadvantage of copper represents its low weldability due to its high thermal conductivity. Friction stir welding (FSW) offers good results in term of mechanical behavior of joints by using different geometry of the tool pin or by using water cooling during the stir process, with satisfactory results obtained only in case of thicker parts [1,2]. Besides the FSW, the laser welding could provide high quality joints of pure copper. Copper high reflectivity and thermal conductivity require an increased heat input for obtaining a full penetration laser welded joint [3]. Preheating of the base material or using a green laser (500 nm wavelength) represent a few alternatives to improve the absorption of the laser radiation and consequently the quality of the welded joint.

This study presents the laser welding of pure copper grade of 3mm in butt geometry. A continuous wave Trudisk TRUMPH laser of 8 KW and a TRUMPH welding head with 100 mm focal length was used for the welding process. Different laser power and welding speed were used to obtain full penetration welding. Optical and SEM microscopy and EDS analyses were carried out to characterise the welded bead. It has been determined that the weld bead, microstructure and the hardness is influenced by the welding speed and power density. Depending of the parameter used and due to the high cooling rates of the weld zone pores formation can occurs during welding of copper sheets.

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VI.O.3

INCREASING THE QUALITY OF WELDED JOINTS OF AI-Si 6082 ALLOYS BY INTRODUCING FORCED VIBRATIONS

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Keywords forced vibrations, aluminum alloys, welding technology

Abstract: The solidification process of aluminum alloys welded area has a major influence on the quality of the joining material. Based on this fact, the introduction of forced vibrations during welding aims to prevent the formation of increase tensions inside the welded structure and diminish the presence of



pinholes or pores as a result of material contraction of alloys containing metals with a different melting point / solidification point.

To avoid this happening it is essential to know and control how the melted material is evolving during the solidification. This step requires that the Al-Si alloy to be welded on a surface with longitudinal vibration composed of a platform whose support can be controlled depending on the required frequency and un M.I.G welding equipment with mechanized welding torch setup to weld, the test peace, above the platform surface. Welding in a mechanical forced vibration environment, results in the

formation of a more uniform and finer grain size microstructure with a more advanced diffusion in the affected zone. The diffusion process occurs faster resulting in decreased structural tensions. The presence of voids and pores inside the material structure is inferior for the oscillating welding process in comparison with the classic solution.

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VI.O.4

INFLUENCE OF THE LASER CLADDING PARAMETERS ON THE MORPHOLOGY, WEAR AND CORROSION RESISTANCE OF COMPOSITE COATINGS

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Keywords: coatings, laser cladding, wear, corrosion, cermet

of Brasov

Abstract: In order to improve wear and corrosion resistance of low carbon steel, cermet composite coatings are usually produced on its surface by different deposition techniques [1]. Laser cladding compared to the traditional surface techniques such as electroplating, thermal spraying and arc welding, has many advantages to produce such cermet coatings. It has been considered as a competitive technique for the traditional methods to deposit surface coatings with low dilution, fine microstructure and superior properties [2].

The paper presents the parameters optimization process of the laser cladding method in order to obtain dense and defect free Metco 439 NS coatings deposited onto a low carbon steel substrate. The properties of the optimized coating in terms of microstructure, tribological behaviour and corrosion resistance were investigated. The microstructure of the Metco 439 NS coating was investigated by scanning electron microscopy (SEM), the sliding wear resistance was determined by pin-on-disk method and the corrosion behaviour was evaluated by means of electro-chemical methods.



Fig. 1 Morphology of Metco 439 NS powder (left image) and laser cladded coating with dendritic structure (right image)

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VI.O.5

MECHANICAL PROPERTIES IMPROVEMENT OF Cu10AI LASER CLADDED ON ALUMINUM

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Keywords: laser cladding, aluminum, mechanical properties

Abstract: It is well known that certain components are made of aluminum and its alloys due to their high degree of ductility, formability and weight ratio. Nevertheless, several applications demand specific mechanical properties like wear, corrosion, fatigue and hardness resistance [1,2]. To prevent possible malfunction of aluminum components due to high temperature oxidation, it is necessary to develop a thermal cladding technology with Cu based powders [3]. This type of powder is frequently used in to restore the damaged areas, high-temperature particle erosion resistance and oxidizing atmosphere resistance below 800 °C.

The chosen method to clad the Cu base layers is laser and coaxial/radially injected powder cladding. Laser and radially injected powder cladding is the most efficient and precise procedure of metallic surface cladding. The cladding ends for radial injection are composed of three conic, concentric components that allow targeting the laser, the powder provided by the transporting gas and the gas used to shape the powder jet. These conic cladding modules, made of copper, are water cooled to prevent the powder to adhere on the nozzle surface.

By using this cladding method new coatings with advanced mechanical and physical properties, employing aluminum 2024 series as base material and Cu10Al powder as cladding. Cu10Al is an easily workable powder, ideal for soft bearing applications, with high resistance to wear, cavitation and suitable degree of resistance to abrasion and fraying.

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VI.PO.1

INDUCTION BRAZING OF COPPER COMPONENTS USING AN AMORPHOUS BRAZING ALLOY

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Keywords: induction brazing, amorphous alloy, copper components, SEM

Abstract: Brazing alloys with amorphous structure have excellent technological properties (stretching, wetting), mostly having a self-fluxing character. These alloys provide good mechanical properties and corrosion resistance to the brazed joints [1].



Fig. 1. SEM Image in longitudinal section of brazed joint

In this work it was elaborated an amorphous brazing alloy in ribbons form for induction brazing of pipe type copper components. In the case of induction brazing, the heating and melting of the brazing alloy is achieved as a result of the heat resulting from the induction heating of the base material [2]. Therefore, in this case there is a heating of the base material to a temperature above the melting temperature of the brazing alloy. Consequently, it was aimed to establish the process parameters of induction brazing in order to achieve the melting of the brazing alloy, without the partially melt or too

much thermal affectation of the base material. The brazed joints were metallographic analyzed by optical and electron microscopy. By optimizing the process parameters, in a longitudinal section of the brazed joint part it can be observed a dendritic structure consisting of a copper solid solution and the joint is geometrically homogeneous (Fig. 1). The developed brazing alloy has good stretching and wetting properties and self-fluxing character.

The main technological parameters are voltage of brazing current and brazing time and the optimization of these parameters leads to a single-phase structure in bonded joints.

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VI.PO.2

CHARACTERIZATION OF THE SURFACES OBTAINED BY GOUGING

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Keywords: gouging, concentration of microparticles, EDAX, SEM, S275JR

Abstract: In this scientific paper, we intend to present the influence of the gouging procedure on the raw material, and the characterization of the surfaces obtained by various gouging procedures. In the framework of the experimental researches, some constructional steel with a low carbon content was used as raw material. The gouging procedures used in the experimental framework were the following: Arc-Air Gouging, Manual Metal Arc Gouging, Plasma Manual Gouging, and Plasma Mechanized Gouging. These procedures are classified by a series of processes, according to the European standard EN ISO 4063:2009. The gouging operation was followed by the measurement of the surface rugosity. During the gouging, the concentration of microparticles was measured by means of the detector MicroDust 880nm. The results obtained after the gouging operation were analysed with a scanning electron microscope, in order to characterise the surface of the notches. With a view to characterising the gouged surfaces, a scanning electron microscope of the type VegaTescan LMH II, equipped with an energy-dispersive X-ray analysis system, was resorted to.

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VI.PO.3

SOLID STATE WELDING TECHNOLOGIES FOR AEROSPACE APPLICATIONS

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Keywords: welding, pressure welding, solid state, friction welding, aerospace components

Abstract: Solid state welding technology is an attractive method to weld metals in solid state. This type of welding is performed below the melting temperature of the materials and in many cases with microscopic force or pressure. Since solid state welding is formed from atomic migration across an interface without a liquid phase, the welding interface is homogeneous and hence mechanical properties and microstructure at the bonded region show better properties than that from conventional fusion welding. In hot press welding, enough time is required with applied pressure at elevated temperature. In friction stir welding, high temperature can be obtained from the frictional heat induced from mechanical rotating motion between two materials. In this study friction stir welding and hot press welding processes are used to manufacture aerospace components without localized distortion. Pressure welding of superalloy was successful without any foreign materials. Press welding of dissimilar alloys of copper alloy and stainless steel was demonstrated to fabricate complex shape of combustion chamber for liquid rocket engine. The optical and scanning electron micrographs show that there is no notable distinction or foreign phase at the interface and the strength of the bonded interface is evaluated. Manufacturing of aerospace components with complex shape is demonstrated with solid state welding processes.



Figure 1. Application of diffusion bonding process to manufacture a liquid rocket chamber

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VI.PO.4

A STUDY ON FRICTION STIR WELDING PROCESS OF AA2219-T87 AL-CU ALLOY

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Keywords: friction stir welding, AA2219, microstructure, mechanical properties

Abstract: Friction Stir Welding (FSW) is a solid-state welding process, and welding takes place with a rotating tool to provide a combination of frictional heating by the tool shoulder, and stirring of the soft material by the pin along the welding line. It is important to avoid overheating since the temperature must be maintained to be below the solidus of the equilibrium phase diagram for the materials being joined. The main advantage is its ability to join metals without melting precludes the risk of traditional defects found in fusion welds such as liquidation cracking, solidification cracking, or oxide formation. The objective of this paper is to investigate the effect of friction stir processing on mechanical and microstructural properties of aluminum alloy 2219-T87. The plates were joined with friction stir welding process using different tool rotation speeds and welding speeds. The mechanical properties were characterized and microstructures of the material in the stirred zone were investigated. The present work represents the strength at each process condition and the optimum friction stir welding process parameters.



Figure 1. Micro-hardness measurement (a) measurement location in cross-sectional view, (b) welded at 400 rpm,

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VI.PO.5

OVERVIEW OF JOINING DISSIMILAR MATERIALS: METALS AND POLYMERS

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Keywords: laser welding, dissimilar materials, polymers, metals, joining

Abstract: The increasing demand in manufacturing hybrid structures and components for engineering applications has determined a growing interest in dissimilar materials joining. These joining methods are: mechanical fastening, adhesive bonding and welding [1]. The objective of this paper is to analyze and summarize the results from the reference literature, concerning different techniques for joining



Fig. 1. Steps of the laser based metal-polymer connection

dissimilar materials, with emphasis on polymers and metals. This literature overview could serve to further understand the processes involved and to optimize the processes for obtaining metal to-polymer hybrid joints. Among the different methods used for hybrid joining, laser welding offers the maximum for production of various components for automotive, aerospace, electronics, and bio-medical applications

[1]. Using laser welding technology, different types of joints could be generated, that are difficult to generate in comparison with conventional joining processes, such as adhesive-bonding, mechanical fastening and thermal pressing. Among the advantages of laser welding, shorter processing times, minimal degradation of the materials (when joining polymers) and minimal thermal stress could be noted [2]. The laser can be also use for microstructuring of the metal surface (fig. 1) in order to increase the boundary surface creating undercuts that fasten the clamping [3].

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Acknowledgements: We hereby acknowledge the structural founds project PRO-DD (POS-CCE, O.2.2.1., ID 123, SMIS 2637, ctr. No 11/2009) for providing the infrastructure used in this work.



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VI.PO.6

OVERLAP LASER WELDING OF THIN POLYCARBONATE SHEETS

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Keywords: laser welding, polycarbonate, joining

Abstract: Due to technological limitation of the polymer molding, it is preferable to make single components and to join them to do the final products. Polymers joining methods can be classified in three main categories: mechanical joining, adhesive bonding and welding [1]. As innovative joining



Fig. 1. Overlap laser welding of polycarbonate 2 mm

technique for plastics, laser welding offer advantages that cannot be achieved with any other process at this time: one step quick welding, desired flexible welding contour, non-contact, easy automated, high stability process, low thermal and mechanical stress. Laser plastic welding requires one part to be transmissive to a laser beam and either the other part absorptive or a coating at the interface to be absorptive to the beam [2].

The goal of this paper is to study the overlap laser welding of thin polycarbonate sheets. As testing material was used 2-3 mm transparent polycarbonate. The joining was done using Coherent HighLight 1000F directdiode system laser with Precitec YC52 welding head manipulated by a CLOSS 7 axes welding robot (fig. 1). After welding with

different welding speeds and laser powers, the samples were analyzed using optical microscopy and tensile stress.

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VI.PO.7

INVESTIGATIONS ON DISSIMILAR BRAZED JOINTS BETWEEN TUNGSTEN CARBIDE AND STEEL

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Keywords: brazing, AgCu filler material, tungsten carbide, steel

Abstract: Joints between different materials such as steel and tungsten carbide are often made by brazing. For asphalt cutters, quick solutions are required to obtain dissimilar joints, at acceptable costs, given the very short working period of these parts.









In this paper are presented some results obtained during brazing dissimilar joints between tungsten carbide and steel, by using different type of filler material from AgCu system, alloyed with P, Sn and Si. The brazing techniques used were oxygen-gas flame and induction joining. The brazing behavior was analyzed in cross sections by optical and electronically microscopy. Metallographic analyze enhanced adhesion features, length of penetration in the joining gap and the wetting angle. The melting range of the filler materials has been measured using thermal analyze.

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VI.PO.8

INNOVATIVE RESEARCH IN THE FIELD OF BRAZED JOINTS FOR DRILL BIT

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Keywords: drill bit, coated rods, brazing

Abstract: The research followed the development of deep joint brazing joints with economic efficient materials between the reinforcement, made from sintered wolfram carbide, and the support, drill bit made from low alloy steel with chromium, from the earthmoving equipment.

The brazing procedure selected is heating until the semi-products of the addition materials reach melting temperature, with oxyacetylene flame slightly carburized.

The pressure of reducing manufacturing costs for drill bits highlighted the need to replace brazing alloys Ag156 according to SR EN ISO 17672 with coated rods type VIAg140R, which are less expensive and successively deposit two layers in one melt, one buffer layer and a filler layer.

The buffer layer has a high moistening capacity of sintered wolfram carbides; it contains Ag156 that comes from the coat of the covered rods, in which it participates with up to 10%. The filler layer of the brazed joint is made out of Ag140.

The global chemical composition of the deposited metal with VIAg140R complies with prescriptions SR EN ISO 17672 for Ag140.

Metallographic analysis and sclerometric tries of specific area of the brazed joints did not highlight any embrittlement imperfections, which lead to the possibility of suggesting the new technology for brazing and extending it to a large number of similar joints

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VI.PO.9

ASPECTS REGARDING REPETITIVE MAINTENANCE CONCEPT IN PUSH BLADES FOR LOADING EQUIPMENT

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Keywords: push blades, loading equipment, repetitive maintenance

Abstract: Preventive repeated maintenance concept provides elaborating, developing and reconditioning, repeatedly, in economic efficient conditions, of the pushing blades that equip bulk loading machines, namely leveling them.

Applying the solution implies developing and elaborating active areas in direct contact with processed materials by loading through welding. To this end the base material of the blades is chosen to comply with the addition material afferent to the selected process, for predetermined circle tries, approx. 5 in the present moment.

Research conducted to validate the loading through welding technology had primary objectives to obtain material couples base/addition that have a high resistance to intense bending demands, under load, combined with a good abrasion usage resistance or high pressure. The chemical composition of the basic materials in the pushing blades, is afferent to high resistance steels, with a maximum of

1,6%Mn, max. 1,5%Cr, max.2%Ni, max. 0,7%Mo and max. 0,005%B.

The chemical composition of the deposited material, through welding, falls into steel prescription, type Fe-25%Cr-5%W-Nb-Ti-B, with metallographic structures that have a high content of complex carbides and hardness of minimum 60HRC.

Sclerometric tried and metallographic research of specific area of the deposited metal/base metal ensemble did not highlight any imperfections such as cracks or white sports in the thermal influenced areas.

Research regarding exploitation behavior, of basaltic aggregates in loading conditions or cereals, have shown a good resistance at wear.

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VI.PO.10

RESEARCH REGARDING THE POST-CLADDING HEAT TREATMENT APPLIED ON METCO 12C LAYER OVER C45 STEEL

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Keywords: laser cladding dissimilar materials, polymers, metals, joining

Abstract: The requirements of tough superficial layers on usual materials and various products repairs demand the using of hard coatings. Applying a laser surface-clad Ni-base alloy, the hardness of the stainless steel increased from 230 HV to 500–700 HV, and heat treatment enabled the microhardness of the clad layer to be further increased to 850–1120 HV [1]. Post-cladding heat treatment increase the dilution having a favorable effect at the melted zone-HAZ interface and thermal fatigue resistance [2].



This study investigates the effect of postcladding temperature on the microstructure and hardness of METCO 12C layer deposited on C45 steel (fig. 1). The cladding, with different powers, was done using Coherent HighLight 1000F direct-diode system laser with Precitec YC50-F200 coaxial cladding head manipulated by a CLOSS 7 axes welding

robot. After cladding the samples were tempered at different temperatures using BMI-FOURS V12 furnace. The samples were inspected using optical microscopy and Vickers hardness. The tests reveal the reducing of the clad layer hardness with laser power increasing.

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VI.PO.11

DISSIMILAR LASER WELDING OF PURE ALUMINIUM AND COPPER

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Keywords: dissimilar welding, laser

Abstract: Nowadays there is an increasing demand of heterogenous joints between alloys with dissimilar mechanical and chemical proprieties. Copper and aluminum represent a potential dissimilar couple that raises compatibility problems in terms of brittle phase formation and unmixed region into the welding bead. The high thermal conductivity and the high reflectivity of the two metals represent a major



Fig. 1. Dissimilar laser welding of copper to aluminum

drawback in the laser welding process. Moreover, the absence of the filler material has a negative influence on the ductility of the welded joint.

Friction stir welding and ultrasonic joining are among the most common methods used for welding of aluminum to copper.

The rapid evolution of the laser technology in terms of power, emission type, wavelengths and frequency allows the development of different laser welding techniques for joining of the copper/aluminum tandem.

This study addresses to the continuous wave

laser welding of copper/aluminum in a butt joint configuration. Electronic grade copper and high purity aluminum (> 99.5%) with a thickness of 1 mm were used for the experimental tests. A TRUMPH Trupulse laser and 7 axes robot was employed for the welding tests. The geometrical and microstructural analysis of the welds were carried out using optical and SEM microscopy. The EDS mapping of the samples cross-sections reveals the mixed and unmixed areas in the weld bead.

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SECTION VII Safety Engineering

Chairpersons:

Dragoș BUZDUGAN, Politehnica University of Timișoara, ROMANIA **Radu IOVĂNAȘ**, Transilvania University of Brasov, ROMANIA TRANSILVANIA

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VII.O.1

ECOSOCIAL (ENVIRONMENTAL AND SOCIAL) SAFETY IN METALLIC MATERIAL INDUSTRY

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Keywords: Ecosociology, pollution, professional disease, metallic materials.

Abstract: The paper deals with a subject of *industrial eco-sociology* represented by the interactions of three systems: natural-ecological (pollution with particulate matters), technological (generation of polluting particulate matters in a metallurgical plant) and social (health status under the negative impact of the polluting particulate matters).[1, 2]

The negative influence on health of the particulate matters is investigated based on the particle size, $d_p[\mu m]$, concentration, c_p [$\mu g/m^3$], granulometric fractions $f_g[\%]$, elemental chemical composition and granulometric structure. The following aspects are approached:

- The importance of industrial ecosociology concerns for sustainable development of the human existence sphere;
- The engineers' need to know the *industrial ecosociology* targets;
- The ranking (classification) of metallurgy sectors (sintering plant, blast furnace, LD steel plant, EAF steel plant, and rolling mills) depending on the severity of diseases induced by the impact of particulate pollutants.

If we take into account only the disease dependence on concentration, the *disease probability increases in the following order*:



If we take into account *only the granulometric structure* in those two situations of granulometric shifting, the conclusions are:

• the probability of inducing diseases with third degree of severity, BGG III, increases with increasing smaller fractions (shifting to the right of the granulometric spectrum), in the order:



• the probability of inducing diseases with first degree of severity, BGG I, increases with increasing large fractions (shifting to the right of the granulometric spectrum), in the order:



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VII.PO.1

VISION OF SAFETY – RESEARCH ABOUT FIRE RESISTANCE OF MATERIALS

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Keywords: vision of safety, fire, materials, clubs.

Abstract: In this paper we present throughout legislation in the field of fire protection and outlined an indicative basis in this area for all those who want to promote recreational activities in nature clubs.



Fig.1. – Fire resistance of wood wainscot

Fire - human guide throughout history, but also the demon who transforms lives and property into ashes. The fire is the oxidation of a combustible material, which results in heat, light, and various reaction products, such as carbon dioxide and water.

Being a random phenomenon unfolding in time and space is practically impossible to describe and specify the evolution of a fire.

In the concept of "VISION OF SAFETY" the initiators of this work have established as to achieve a guide to deliver support in the field of defense against fire, current and future entrepreneurs in recreational activities carried out in establishments defined as "clubs".

Experimental research proposed the creation of experiments that enable us to issue conclusions on how certain materials commonly used in the interior design of the clubs react to fire.



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SECTION VIII Magnesium Science and Engineering

Chairpersons:

Nir MOSCOVITCH, R&D and Business Development, Dead Sea Magnesium, ICL, ISRAEL

Bela VARGA, Transilvania University of Brasov, ROMANIA







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VIII.K.1

EFFECT OF TUNGSTEN INERT GAS WELDING ON THE MICROSTRUCTURE AND THE FATIGUE RESISTANCE OF ZE41 MAGNESIUM ALLOY

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Keywords: magnesium alloys, welding, microstructure, tensile properties, fatigue.

Abstract: ZE41 magnesium alloy is a Mg–Zn–RE–Zr alloy showing good mechanical properties, creep resistance and excellent castability too [1,2]. Currently this type of light alloys is used in automotive and aeronautical industry, for manufacturing of different mechanical components, like aircraft components and transmission housing. Generally, at industrial level, welding technologies are used to remove and repair the casting defects and in particular Tungsten Inert Gas (TIG) welding technology is one of the processes applied to achieve this goal [3,4]. In the present paper, the microstructure and the fatigue resistance of ZE41 magnesium alloy before and after welding, carried out by TIG technology have been evaluated. In case of the TIG welded samples, a grain refinement in the fusion zone and a grain coarsening in the heat-affected zone appears. The micro-hardness evolution reveals the microstructure variations occurred in the welded samples. A slight decrease of both the yield strength and the ductility of the samples after TIG welding appears, but at the same time a slight increase of the ultimate tensile strength, compared to the un- welded samples was observed. The fatigue strength is not affected negatively by the welding; a fatigue limit of 92 MPa and 97 MPa, for the un-welded and TIG welded samples, respectively were obtained. The fracture mode in the crack growth region shows trans-granular features and the fatigue cracks preferentially growth by cutting straight across the grains. In the final fracture region, the fracture is mainly intergranular. For both type of the investigated samples, isolated planes of the cleavage zones and oxide particles have acted as nucleation site for the fatigue cracks. Cavity defects, developed during the welding process, favoured the nucleation of fatigue cracks. The obtained results have demonstrated that the alloy has maintained excellent fatigue resistance after the welding process.

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VIII.K.2

NANOCARBONS-Mg COMPOSITES FOR HYDROGEN STORAGE

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Keywords: hydrogen storage, spillover, magnesium, graphene, CNT

Abstract: Magnesium is one of the most investigated materials for hydrogen storage applications; it has a very high hydrogen capacity (7.7wt% and $101gH_2/L$) while being relatively cheap. However, this metallic hydrogen acceptor has a poor de/hydriding kinetics. Loading the Mg by light carbonaceous additives, such as CNT or grapheme, may assist in hydrogen atom diffusion in solid mixtures through spillover mechanism¹; this phenomenon describes the transport of H atoms, adsorbed on one surface (e.g., metal catalyst) onto another surface.

We explored the hydrogen storage kinetics of Pd-Mg composites upon addition of different carbonaceous spillover agents² (activated carbon, AC, a wide spectrum of Carbon Nanotubes and graphene types). We found that the de/hydriding kinetics is strongly dependent on the nanocarbon



Figure 1: (a) t_{50} for 50% MgH₂ dehydriding versus t_{50} of Mg hydriding for several composites containing Pd/CNTs and Pd/AC-based additives. T=300°C. [Pd]=0.25wt%. (b) Pd/CNT(>50nm) network (arrows), SEM and (c) discrete Pd/CNT (~10nm) agglomerate on Mg matrix, SEM.

morphology and configuration (e.g., length, diameter and distribution, Figure 1). We defined a figure of merit (FOM) quantifying the de/hydriding performance of composites previously reported in literature and those investigated in the present study. The FOM demonstrates that the fastest kinetics is obtained for our Pd-decorated CNT (Pd/CNT) having the largest diameter. We found a clear structure-function relation between the spillover agent properties and the Mg de/hydriding rates.

The nanocarbon properties and the Pd concentrations may be used for tuning the kinetics when nanocarbons are integrated into Mg-based composites. The FOM analysis emphasizes the significant effects of our

synthesized nanostructures. These findings confirm that nanocarbons are applicable as an alternative to expensive metal catalysts in light-metal hydrogen storage devices and fuel cells.

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VIII.O.1

CHARACTERIZATION OF SOME MASTER Mg-X SYSTEM (Ca, Mn, Zr, Y) ALLOYS USED IN MEDICAL APPLICATIONS

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Keywords: Optical microstructure, SEM, XRD, Micro-Hardness, Electrochemical tests



Fig.1. Microstructure of Mg-30Y

Abstract: Ultralight magnesium alloys are wide used in the medical field, especially for biodegradable implants. Although they are wide used, magnesium has low corrosion resistance. To improve this resistance, different types of alloys based on magnesium and Ca, Mn, Zr and Y can be developed. The main goal of the present paper is to investigate the properties of some master alloy based on Mg-X system (Ca ,Mn, Zr, Y) used in the development of biodegradable based alloys of Mg. The surface morphology was characterized using scanning electron microscopy (SEM), X-ray diffraction (XRD) and optical microscopy. After the

XRD analysis, there was observed that some specific compounds were formed of Mg_2Ca , $Mg_{0.97}Mn_{0.025}$, MgZr, Mg_2Y , $Mg_{24}Y_5$ having the main Mg phase formed in the hexagonal structure. There were also evaluated the master alloys micro-hardness values in the range of 58.41 HV (Pure Mg), 67.97 HV (Mg-3Mn), 85.12 HV (Mg-25Zr), 131.8 HV (Mg-15Ca) and 291.45 HV (Mg-30Y). The corrosion resistance was developed using electrochemical testing in specific medium and there is shown that the corrosion rate increased significantly for the master alloys investigated, rather than pure magnesium. As a final conclusion structural properties of these alloys recommend them for usage as medical implants.

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VIII.O.2

THE EFFECT OF ZIRCONIUM ADDITION ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF GRAVITY CAST MG-4ZN ALLOY USING GRAPHITE AND SHELL MOULD

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Keywords: Mg-4Zn alloy, graphite-silica mould, thermal analysis, zirconium, wear, hardness, mechanical properties

Abstract: In this study, the effect of zirconium (0, 0.5, 1, 1.5, 2 and 4 Zr wt%) on the microstructure, mechanical and wear properties of a magnesium-based alloy (Mg–Zn4 wt%) were investigated. The Mg–Zn4-(x)Zr alloys were melt by controlled sheilded gas atmosphere and produced by a gravitiy-casting process using two type of mould materials such graphite and shell mould. Both type of materials were machined by The results show that the addition of Zr element modified the microstructure and decreased the grain size for the both mould materials [1]. The maximum tensile strengh and hardness were achived by %1 Zr (169 MPa) and %4Zr (100 HrB) addition respectively using graphite mould. After casting, two type of machining route were aplied on the surface of samples to make the surface flat and less rough, first route was wet grinding and the second was dry machining by milling machine. The highest hardness value were measured after milling operation. However there was no increasing on the tensile test. The results of hardness, tensile and wear indicate that the hardness, tensile and wear resistance of Mg–4Zn alloy increased by adding Zr up to 1 wt% and then is relatively constant with increasing Zr. The wear rate of Mg–4Zn alloy decreased with increasing alloying elements and machning process.

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VIII.O.3

THE DEVELOPMENT OF SPECIAL MAGNESIUM ALLOYS BY ULTRARAPID SOLIDIFICATION

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Keywords:ultrarapid solidification Mg-base alloy, nanocrystalline structure, industrial application.

Abstract:The range of the specific cooling rate in ultrarapid solidification is capable to produce really new constitutional and structural modifications (sometimes at the limit of amorphous structures). The cooling range of 10^5 K/s can be considered as a limit, non-equilibrium being obtained beyond it. This makes the



Fig.1.TEM image of nanocrystalline structure in Mg-base alloy.

ultrarapid solidification a means to create absolutely new structures.The nanocrystalline and nanostructure magnesium materials offer radical improvements of properties or new functions. This actual work in the development of engineering aterials refers to the production of the ultrarapidly solidified Mg-base alloys: 90Mg5Al4Zn1Cu or 87Mg9Al0.4Zn3.6Pb and 91.5Mg6.6Al1.4Zn0.5Cd in the attempt to obtain a maximum increase of the structural homogenization and a substantial improvement of the physical-mechanical and chemical stability. The ultrarapid solidification of magnesium base alloys has shown interesting phenomena, such as the appearance of some new type

of quasicrystalline and nanocrystalline structures and also some special chemical and electrochemical characteristics for possible application in the energy conversion field. The micro-structural, mechanical and electrochemical characteristics of the newly developed alloys were analyzed using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The corrosion behavior was evaluated by immersion test and potentiodynamic polarization analysis in order to select the alloys suitable for industrial applications.

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VIII.O.4

EXPERIMENTAL INVESTIGATION OF USING FRICTIONAL SIGNATURE FOR

STRUCTURAL HEALTH MONITORING

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Keywords: frictional heat generation, structural health indicators, condition monitoring

Abstract: A typical industrial application of thermal based conditioning monitoring on a dynamic mechanical component (compressor motor) is presented in Fig. 1, where the abnormal surface temperature is an indication of a probable flaw [1]. Generally, frictional generation occurs whenever



Fig. 1. A typical practical setup for thermal based condition monitoring [1]

interacting mechanical dynamic components undergoes a relative motion [2]. Hence, its analysis can result to a quick and reliable indication of the structural health monitoring (SHM). Therefore, in order to explore this, an experimental investigations were carried out on AISI 304 steel of both healthy and defect induced cantilever beam-like structures coupled with a lacing wire and subjected to forced excitations, while infrared cameras capturing the thermal images on the frictional interfaces. Subsequently, the temperature and vibration frequency indicators SHM were

analysed from the interface thermal imaging. The analysed frequencies from frictional temperature evolution time-domain waveform using a Matlab FFT algorithm compared well with accelerometer acquired, the maximum relative error being 0.30%. Moreover, the beam with induced defect indicated an abnormal temperature of 8.1°C compared to the healthy, as well as multiple spectral peaks around the dominant frequency, hence, successful discrimination.

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