

BOOK OF ABSTRACTS

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Contents

Plenary presentations	1
Functional Materials	8
Oral	8
Poster	31
Structural Materials	48
Oral	48
Poster	79
Design and Processing	110
Oral	110
Poster	123
Manufacturing and Recycling	131
Oral	131
Poster	138
Energy, Transportation and Environment	145
Oral	145
Poster	154
Life, Science and Health	159
Oral	159
Poster	172
Modelling and Characterization	179
Oral	179
Poster	206
European Master Thesis Award	214
FEMS Communication Award for Excellence in MSE for 2019	222

Plenary presentations

Bioactive materials and biofabrication for tissue engineering: progress and challenges

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A comprehensive overview about 3D scaffolds for tissue engineering will be presented and key results on the development and characterisation of nanostructured bioactive glass and polymer-bioactive glass composite scaffolds aiming at clinical applications will be discussed. Technologies available to fabricate such scaffolds will be introduced, including foam replica method, additive manufacturing and electrospinning techniques. Novel approaches involving the coating and infiltration of bioactive glass based scaffolds by biodegradable polymers containing functionalized nanoscale inorganic particles for in situ drug delivery will be presented, as an effective concept to merge tissue engineering with drug delivery matrices. As an important advantage of bioactive glasses in comparison with other biomaterials, specific glass compositions doped with metallic ions with biological activity can induce favourable cell behaviour in relation to osteogenesis and angiogenesis [1]. Indeed the vascularisation potential of bioactive scaffolds incorporating angiogenic ions will be discussed in the context of vascularised bone based on *in vivo* results. Areas of future research will be discussed, focusing on regeneration of (complex) tissue interfaces, e.g. osteochondral defects, wound healing and soft tissue regeneration [2]. Novel additive manufacturing approaches for production of multifunctional scaffolds based on cell encapsulation and biofabrication (bioplotting) technologies using bioinks incorporating bioactive glass particles [3] will be introduced and the challenges ahead, which must be tackled to make tissue engineering a reality in clinical settings, will be highlighted, discussing the expanding opportunities in the field.

References

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Mechanical properties of nanostructured materials

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Mechanical properties of nanostructured materials are being massively employed because of their properties departing from their bulk counterpart. This expanding field will be addressed from the mechanical point of view. The mechanical properties at small scales will be discussed, namely size effects on elastic, plastic and brittle responses of nano-objects, thin films and polycrystals. We will then examine both ex-situ and in-situ experiments that operate in the SEM, TEM and under synchrotron x-ray beam. A few examples will be further discussed in terms of samples preparation, test procedure, interpretation and errors identification.

New insights into metallic alloy microstructural evolution by in-situ characterization

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Structure, processing, property, and performance relationships are the cornerstone of materials science and engineering. Yet, we are usually left to infer what, when, how, and why performance degrades and failures occur. Today, state-of-the-art characterization techniques available in the laboratory and at national user facilities are enabling unprecedented, real-time studies of metallic alloys during processing, deformation, and simulated service and manufacturing. Solidification is critical to processes like casting and additive manufacturing and the manufacture of metallic alloy components we use in our everyday lives. Here the use of x-rays, protons, and electrons to study multiscale solidification dynamics in metallic alloys during processing is highlighted. These experimental results are used to inform, develop, and validate computational models at the same length- and timescales. The impact of solidification on subsequent solid-state microstructural evolution is also explored. The new knowledge gained by in-situ characterization will aid the prediction and control of metallic alloy microstructures and properties by design with advanced manufacturing. Opportunities for young professionals in the U.S. in Materials Science and Engineering are also highlighted. This work was supported by the U.S. Department of Energy, Office of Science, Basic Energy Sciences, Materials Sciences and Engineering Division.

Biography

Amy J. Clarke is an Associate Professor in the George S. Ansell Department of Metallurgical and Materials Engineering, Site Director for the Center for Advanced Non-Ferrous Structural Alloys (CANFSA), and an affiliate of the Advanced Steel Processing and Products Research

Center (ASPPRC) at the Colorado School of Mines (CSM). She is also a Guest Scientist at Los Alamos National Laboratory (LANL). Her current research focuses on making, measuring, and modeling metallic alloys during processing, including x-ray, proton, and electron imaging of multi-scale solidification dynamics at national user facilities, the study of phase transformations and microstructural evolution, and non-ferrous and ferrous physical metallurgy. Amy earned her B.S. degree from Michigan Technological University and her M.S. and Ph.D. from CSM in Metallurgical and Materials Engineering. Prior to joining CSM, she was a Scientist and Seaborg Institute Postdoctoral Fellow at LANL and Senior Engineer – Development/Research at Caterpillar Inc. Amy has received a U.S. DOE Office of Science Early Career Research Program Award and a Presidential Early Career Award for Scientists and Engineers (PECASE). Amy serves on The Minerals, Metals & Materials Society (TMS) Board of Directors, is Chair of Argonne National Laboratory's Advanced Photon Source Users Organization Steering Committee, and is an Editor for Metallurgical and Materials Transactions A. She has also served on the Association for Iron and Steel Technology (AIST) Board of Directors.

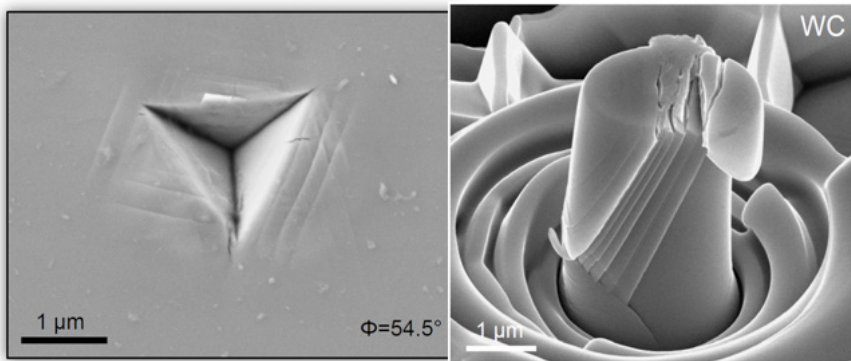
Nano-mechanical testing of advanced ceramics

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The deformation and damage characteristics of differently oriented WC grains/crystals in WC – Co, Si_3N_4 grains/crystals in reaction bonded Si_3N_4 system and ZrB_2 grains/crystals in ZrB_2 polycrystal were investigated. Depth-sensing nano-indentation and scratch tests of grains and micro-compression tests of micropillars prepared by focused ion beam from oriented facets of grains were studied. Electron backscatter diffraction (EBSD), atomic force microscopy (AFM) and scanning electron microscopy (SEM) investigations were performed to determine the grain orientation and to study the surface morphology and the resulting deformation and damage mechanisms around the indents and in micropillars.

The hardness and scratch resistance of the differently orientated grains showed significant angle dependence from the basal towards the prismatic directions. A strong influence of the grains orientation on compressive yield stress and rupture stress values was found during the micropillar test, too. The active slip systems for individual ceramics have been recognized. The different properties of the basal and prismatic planes was found to be connected with the different deformation mechanisms – slip and dislocation activities.



Slip lines around the indent in ZrB_2 and deformation of micropillar from WC grain.

Defect structure characterization in advanced materials

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The type and densities of lattice defects (e.g., vacancies, dislocations and twin faults) have a significant effect on some important properties, such as electrical and corrosion resistance, yield strength and ductility of advanced materials [1]. The processing conditions of these materials influence their lattice defect structure, therefore the properties can be tailored by an appropriate selection of the production methodology. In this presentation, the correlation between the processing conditions and the defect structure is overviewed for nanomaterials processed by either bottom-up production methods (such as sintering of nanopowders or electrodeposition) or top-down routes (i.e., by severe plastic deformation techniques). The defect structure is studied by different methods, such as electron microscopy and X-ray line profile analysis. The latter method is very effective in studying the density and arrangement of dislocations, as well as the twin-fault probability [2]. The effect of additives and alloying elements on the defect structure in nanomaterials is discussed in detail. In addition, the influence of post-processing annealing on the lattice defects is investigated in this presentation. The correlation between the defect structure and the mechanical performance of advanced materials is also revealed.

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Materials characterization down to the atomic scale using electron microscopy

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Electron Microscopes have become one of the most important instruments used in materials characterization. By using a beam of accelerated electrons as a source of illumination electron microscopes are capable of far higher resolving power than light microscopes as the wavelength of an electron can be up to 100,000 times shorter than that of visible light photons. As such, the electron microscope can reveal the structure of objects on the nanoscale.

Previously, the ultimate performance of electron microscopes was limited due to both imperfections in the electron optical lens and the lack of a monochromatic electron source. However, these spherical and chromatic “aberrations” were overcome in the 1990’s with the development of the aberration corrector. By correcting these aberrations scientists were able to visualize atoms and even measure individual atomic positions with picometer precision. The possibility to correlate atomic-scale structure with the macroscopic physical properties constituted a major breakthrough in materials science, with implications for many areas of science and technology.

Within less than five years of the commercialization of aberration-corrected electron microscopy, more than 200 corrected microscopes were installed in university and industrial research laboratories all over the world, pointing to the central role that state-of-the-art electron microscopy plays in 21st century research and putting an end to decades of stagnation in this key area of scientific instrumentation and industrial technology.

This lecture will focus on the development of the electron microscope and the role state of the art transmission electron microscopes are playing in advanced materials characterization around the world.

The European Institute of Innovation And Technology’s aims and impact

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The European Institute of Innovation and Technology (EIT) is a unique EU initiative that drives innovation and entrepreneurship across Europe. We bring together leading universities, research institutions and companies to form dynamic European partnerships, known as EIT Innovation Communities. Through these Communities, our partners develop innovative products and services, start new companies, bring research to the market and train a new generation of entrepreneurs. Each EIT Innovation Community focuses on a different chal-

lenge, from sustainable energy production to active ageing and mitigating climate change. To date, we have created over 6 000 highly skilled jobs and supported more than 800 ventures. These ventures have raised more than € 600 million in external capital, showing that venture capitalists have confidence in our entrepreneurial activities.

The EIT's education activities – including Master and Doctoral programmes and Massive Open Online Courses (MOOCs) – provide graduates with an entrepreneurial mind set. Over 1 200 students have already graduated from EIT-labelled education programmes; by 2020, we project that 5 000 more will hold an EIT-label degree. These graduates combine excellent theoretical skills with the entrepreneurial and creative skills needed to become a successful entrepreneur. Available evidence indicate that this combination of skills is highly valued on the labour market: more than 90% of EIT InnoEnergy alumni find employment within six months of graduation, and their initial remuneration is calculated to be 15% higher than non-EIT graduates.

CRM-EXTREME COST Action activities and future perspectives

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Difficulties in the access to critical raw materials (CRMs) are expected to depress industrial sectors vital to Europe.

The CRM-EXTREME COST Action focuses on the substitution of CRMs (like Cr, Co, Nb, W, Y) in high value alloys and metal-matrix composites used under extreme conditions of temperature, loading, friction, wear, corrosion, in Energy, Transportation and Machinery manufacturing industries. Thanks to the COST networking tools, the Action aims to set up a network of expertise to define the state of knowledge and gaps in multi-scale modelling, synthesis, characterization, engineering design and recycling that could find viable alternatives to CRMs and promote the industrial exploitation of substituted materials.

The Action envisions a fully Sustainable Value Chain approach for:

- Alternatives for Co and W in WC/Co cemented carbide wear resistant tool materials
- Alternatives for chromium- and tungsten-alloyed tool steels
- Reduction of Cr and Y in high-strength steel alloys
- Alternatives for Cr and other CRMs by hard, wear and corrosion resistant surface coatings
- Alternatives for Nb in high-strength low-alloy (HSLA) steel (Automotive)
- Alternatives for high-temperature Ni-based superalloys (Aerospace)
- A four-year Action oriented to strengthen collaboration between active researchers working in the different areas of investigation involving CRMs, is the most suitable initiative to seed the initial catalytic nucleus of growth for EU excellence in strategic CRMs substitution.

Establishing the industrial leadership of europe in advanced materials for the energy union – The role of innovation

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The aim of the EMIRI contribution is to inform stakeholders on the key enabling role that the Industry of Advanced Materials plays in facilitating the development & deployment of clean energy & clean mobility technologies in Europe while creating growth & jobs in Europe.

After a short introduction to EMIRI, the presentation will address global trends in clean energy & clean mobility technologies, will highlight elements of the European sector of Advanced Materials and will outline recommendations to solve the European energy challenges and ensure Industrial Leadership of the sector. The contribution will also present the key priorities from Industry for more effective & efficient Innovation on Advanced Materials for clean energy & clean mobility in Europe.

Functional Materials

Oral

Flame retardants (FRs) based on hybrid organic-inorganic materials

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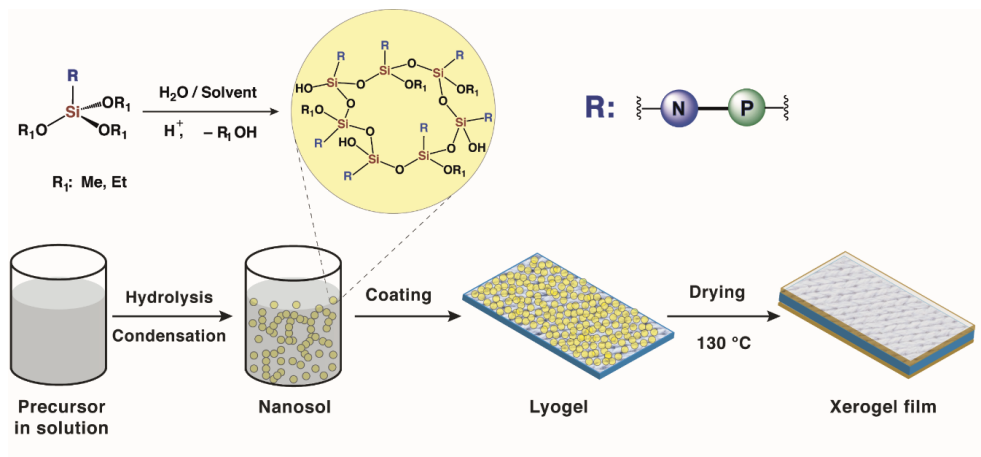
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Keywords: flame retardants, hybrid materials, sol-gel coating, textile

The requirement of halogen-free flame retardants for fire protection of fibers and textiles have recently received much attention because of the toxicity and the bioaccumulation of the presently used commercial halogenated-FRs. Therefore, they are listed by REACH regulations as substances that will be forbidden for application in the near future. In contrast, hybrid organic-inorganic FRs containing phosphorus, nitrogen and silicon groups are environmentally friendly and less toxic materials. Among several strategies that can be exploited, the incorporation of these groups onto textile can be achieved by sol-gel coating technique, as illustrated in the figure below. In addition to enabling multi-functionality, flame retardant finishing of textile using sol-gel method has a lot of advantages, such as fast and simple processing method, applicability on various type of textiles very good fastness and washing resistance. In this work, several silicone-based FRs containing phosphor and nitrogen have been synthesized. The FRs were evaluated by testing the flammability of different textiles according to EN ISO 15025 (protective clothing). One of our aim is also to study the synergistic effect of phosphor/nitrogen functional groups on the properties of flame retardancy as well as to analyse the behaviour and hence evaluate the mechanism of the synthesized FRs materials using different facilities.



Acknowledgments

The research projects (IGF Nr. 19617N) of the Forschungskuratorium Textil e. V. was funded by the Bundesministerium für Wirtschaft und Technologie within the program Industrielle Gemeinschaftsforschung (IGF) by the Arbeitsgemeinschaft industrieller Forschungsvereinigungen e. V. (AiF).

Al₂O₃ protective thin film

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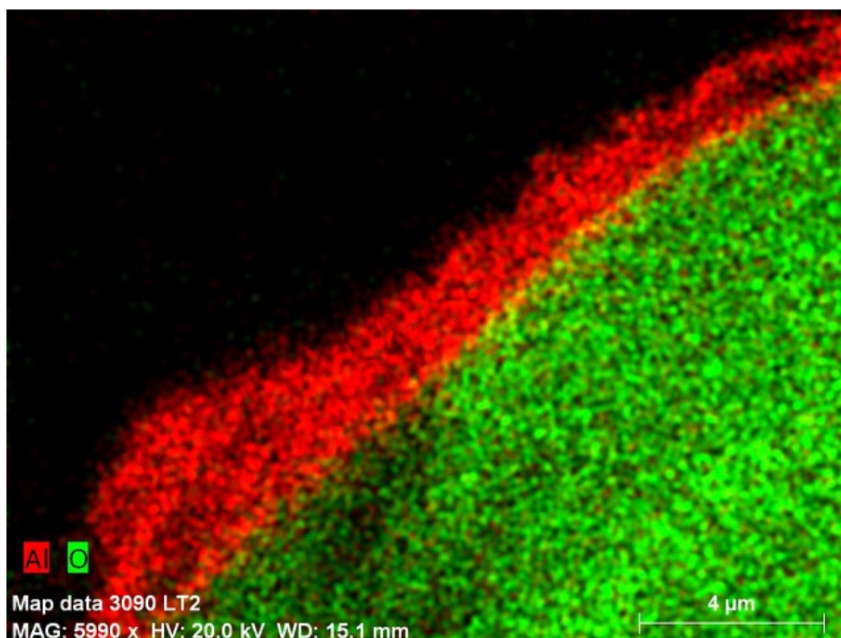
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Gamma aluminum oxide is one of the most important oxides and also the most common form of activated alumina for adsorptive and catalytic application. γ -Al₂O₃ can be utilized for the purification of drinking water, *e.g.* for the removal of arsenic ions by activated-alumina adsorption. The other possibility for the release of arsenic is a new photochemical oxidation method by means of vacuum-UV lamp irradiation emitting both 185 and 254 nm lights. Over that γ -Al₂O₃ film has direct application as catalyst carriers, ultra-filtration and separation films as well as abrasive-resistant and optical coatings.

In this study, transparent γ -Al₂O₃ thin layers have been produced by dip coating technique from various systems; hydrogel, alcogel, and diluted colloid solution such as sol. Starting materials were aluminum nitrate, basic aluminum acetate, and boehmite. Propanol and water were provided as solvent. The gel systems have been prepared by sol-gel method. The diluted colloid dispersion was obtained by partial dissolution of boehmite powder. The evaporation of solvent content from layer was carried out at 70–80 °C. In order to develop the gamma Al₂O₃ crystalline phase, a heat treatment of 600 °C must be applied.

The products of various syntheses were compared regarding their transparency and layer quality. The transmittance was determined by uv spectroscopy. The transparency strongly depends on the concentration of colloid solutions. For example the transparency changes from 7–33% to 60–85% by reducing the concentration from 40 g dm^{-3} to 20 g dm^{-3} . The X-ray diffraction detected the morphology, and the scanning electron microscopy investigated the layer quality (Figure 1).

Figure 1. Element map for Al and O atoms in cross section of gamma Al_2O_3 layer using SEM.



Functional and mechanical property tuning of superelastic NiTi filaments by electric pulse heat treatment

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Keywords: Shape memory alloy, nitinol, super-elasticity, property modification

Intermetallic shape memory alloy NiTi, due to its superelasticity and shape memory effect, has been a prominent material in scientific and engineering field. Microstructure and functional thermomechanical properties of the superelastic NiTi alloy can be deliberately modified by cold working and annealing.

In this work, cold-worked NiTi filaments were subjected to non-conventional electric pulse heat treatment with varied annealing time (5–25 ms) and fixed power density 160 W/mm³. Results of tensile tests up to the failure on annealed wires reveal that varied annealing time results in different functional properties, such as decrease in transformation stresses, increase in transformation strain and cyclic stability of superelastic stress-strain response. However, the annealing affects also the yield stress for plasticity, strength and ductility of the annealed wire: longer annealing time (>15 ms) makes the NiTi filaments to attain large ductility ~55 % after a strain hardening, while in case of shorter annealing time (< 14 ms), the wire fractures at strains of ~13% in the absence of strain hardening. The yield stress for plasticity decreases from 1800MPa to 400 MPa with increasing annealing time from 4 up to 25ms. It is concluded that the plastic deformability, hardening, strength and ductility plays essential role in functional property setting of NiTi filaments.

FeNiCoAlTaB single crystals and their superelastic properties

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Keywords: superelasticity, phase transformations, single crystals, orientation

Fe-based shape memory alloys (Fe-28Ni-17Co-11.5Al-2.5Ta-0.05B abbreviated NCATB) exhibit a large superelastic strain. However, the superelastic effect is strongly dependent on the precipitation hardening which is required to suppress the plastic deformation of the parent austenitic phase. The fcc γ' phase is the main strengthening component of these alloys. In order to obtain optimal mechanical properties the effect of heat treatment on the precipitation hardening in multicomponent single crystalline materials is studied. Heat treatment with variable annealing time (0.5h, 1h, 5h, 10h, 24h) at 973 K was performed to single crystals with $\langle 100 \rangle$ and $\langle 111 \rangle$ orientations. Employing synchrotron diffraction three different intermetallic phases such as NiAl, Ni₃Al and NiAl₃ are analyzed. Subsequently, single crystals with $\langle 100 \rangle$, $\langle 110 \rangle$, $\langle 111 \rangle$ and $\langle 112 \rangle$ orientations were compressed at different temperatures (77, 123, and 295 K) to provide an insight into the mechanism of superelasticity observed in these alloys. Plastic and elasto-plastic response, has been observed depending on the orientation and deformation temperature. The global and local orientation measurements were determined by diffraction of high-energy synchrotron radiation and electron backscatter diffraction. The results are discussed with respect to crystallographic orientation, deformation mode, precipitations and phase transformation.

Acknowledgement

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Interphase formation of cured epoxy resin near boehmite surface

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Inorganic nanoparticles are commonly used as reinforcement agents for polymer composites. Improving the performance of the material with desirable properties requires understanding of the interaction between polymer chains and nanoparticles and the properties of the

interphase. Boehmite, a mineral of aluminum with basic unit of $\text{AlO}(\text{OH})$, is a promising nanofiller which leads to enhanced performance of epoxy composites. Recently, we reported that in epoxy/ boehmite nanocomposites specially with high filler concentrations, boehmite causes changes in matrix properties and influences the curing process of the resin mixture. This brings up the importance of interphase (IP) properties. The nature of IP formation in thermosets can be complicated. There is a so called 'chemical IP' arises from concentration gradients of the hardener which needs to be distinguished from 'mechanical IP' which is the result of modulus gradients of polymer in the vicinity of inorganic surface. Due to resolution limitations of conventional nanoprobng approaches, characterization of the interphase between individual boehmite nanoparticles and matrix is a challenge. Therefore, in this study we simplify the three-dimensional nanocomposite system to a two-dimensional horizontally layered sample with a large and easy to access interphase area to be investigated by advanced AFM-based methods. For this purpose, $1\text{ }\mu\text{m}$ coatings of hydrothermally synthesized boehmite are prepared as the substrate on which the epoxy is later molded and cured. AFM force-distance curve (FDC) and amplitude dependent force spectroscopy (ADFS) are two main approaches which are used to investigate the mechanical IP. Nano-IR and scanning kelvin probe microscopy (SKPM) are used to investigate the chemical IP. The results show a large chemical IP area (approx. $1\text{ }\mu\text{m}$) near boehmite surface. Mechanical IP with the width of approx. 100 nm was observed. This region shows a considerable low elastic modulus compared to the bulk epoxy. Nano-IR approach shows high concentrations of anhydride hardener in boehmite region. Based on these observations we propose a mechanism in which boehmite contributes in the curing process and causes long-range alterations in the chemistry of epoxy network.

Black phosphorus oxidation: Problem or opportunity

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Keywords: Surface oxide, phosphorene, passivation

Black phosphorus is a novel material with interesting properties rediscovered recently, however, among 2D materials it suffers of prompt oxidation. According to theoretical works, oxidation induces drastic modifications in the electronic structure of the pristine material. Different types of oxides have been predicted but their identification at the experimental level still represents a challenge. We have evaluated the oxidation of black phosphorus by using thermogravimetric techniques and conventional Raman spectroscopy in the bulk and exfoliated systems, respectively. Our results were explained based on the state of the art of theoretical studies and agree with experimental works presented previously but using other techniques. The better understanding of black phosphorus oxidation opens the possibility to enhanced stability by transparent passivation with native oxide layers and the electronic modifications on the surface layers can improve the feasibility of certain applications.

Preparation and synthesis of hydroxyapatite bio-ceramic from Hungarian bio-waste by thermal heat treatment

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Keywords: Hydroxyapatite, bovine bone, calcination process, calcium phosphate

Hydroxyapatite [HAP, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$] is a compound from calcium phosphate family. This material is one of the most common biomaterials currently used in bone tissue engineering application, tooth implants and maxillofacial surgery due to their crystallographic and chemical similarity to human carbonated apatite. HAp is known to be highly biocompatibility, bioactive, osteoconductive, non-toxic, non-inflammatory and non-immunogenic agent.

This study aims to synthesize and characterize Hydroxyapatite (HAp) bio-ceramic powder from Hungarian Bio-Waste. Calcination treatment will apply to produce the hydroxyapatite powders from bovine bone as an eco-friendly and inexpensive source. X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Raman spectroscopy, Field Emission Scanning Electron Microscopy (FESEM) with Energy Dispersive X-Ray Spectrometry (EDS) and Atomic Force Microscopy (AFM) will carry out to characterize the synthesized powder.

Investigation of joining technologies of diamond drilling segments

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Keywords: joining technologies, welding, soldering, diamond segment, material testing

Due to the rapid development of the building industry, there is an increasing demand for tools for improvised concrete machining on site. The typical areas of application are chiselling, drilling, channel preparation, surface roughening and wall breaking. The most commonly used machining forms are the openings, cavities or holes in the structural elements of buildings. Such machining can be performed effectively with diamond edge tools.

Segments containing diamond particles are attached to replaceable inserts or steel tool bodies for the sake of cost-effectiveness. The used joining technology should meet both environmental and technical requirements. The joining zone must bear high mechanical and significant thermal loads during usage. In the event of improper joint, the segments may detach from the base and fly away, causing injury.

Nowadays, many methods of welding or soldering technics are used to attach diamond segments. The development of the segments composition and geometry requires the develop-

ment of joining technologies, as well. For this reason, the possible technologies of the segment fixing have been investigated, e.g. laser beam, capacitor welding and flame soldering. In addition to these joining technologies, other segment fixing methods have emerged as well, such as resistance welding, friction welding and induction soldering.

The micro structure of the joints has been examined by optical and scanning electron microscopy, and also chemical element maps have been recorded. Joints have been subjected to fracture and to hardness testing. The mechanical properties and composition changes of the joints with the different joining technologies have been evaluated and compared.

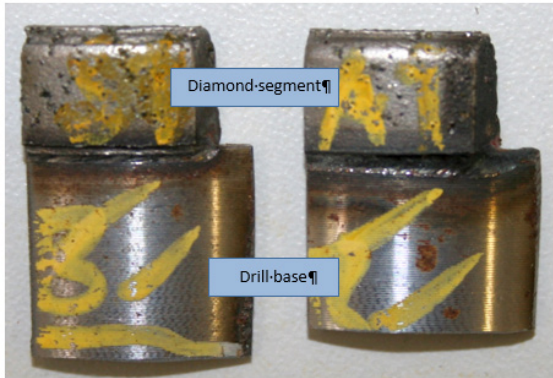


Figure 1 Laser welded segments

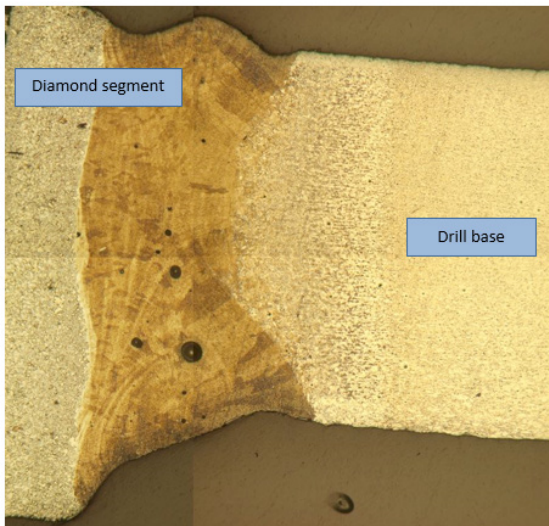


Figure 2 Optical microscopic image of a laser welded joint

Acknowledgments

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Microstructure and mechanical properties of 7XXX series aluminum alloys obtained by semi-continuous casting

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Rising client expectations in relation to the quality of products require developing new technologies that improve the quality of casting. Aluminum, in spite of it being perceived as a new material, holds the key position in the industry. Its role in the development of advanced technologies continues to grow, according to the latest publications of reports from the manufacturing and sales. Aluminum occupies a key position among non-ferrous metals – ahead of copper – both in the production area, as well as in importance to the global economy.

The advantage of Al-Zn-Mg-Cu alloys are undoubtedly good strength, light-weight and resistance to corrosion. In additions, the strength of 7xxx series aluminum alloys increases with an increased concentration of Zn. They are characterized by perfect energy absorption and good plasticity. These qualities are needed in this application area and provide a good solution. Aluminum alloys of the 7010 series are mainly used in the aviation industry due to their fatigue resistance and resistance to corrosion. This European alloy has properties similar to its American counterpart of the 7050 series and is mainly used in the aviation industry as well. Usage of this material in the production of highly durable parts offers a balance between mechanical properties and resistance to stress corrosion. Despite this, it is not suitable for application at elevated temperatures. Most common applications of 7010 alloy are durable aviation parts, closed die forgings for the aviation industry and large forged bars for the aviation industry.

Last decade brought great progress in the production of aluminum and the use of new techniques of its casting, forming, welding and modification of its surface. This development was intended to improve the structural integrity of aluminum alloys. They are used in aviation industry, aerospace, automotive industry, electrical engineering, in parts of electrical machinery and devices, in the construction sector, as well as in the fields of metallurgy and many others. The mentioned alloys are often used for aesthetic purposes as well. Aluminum to a large extent owes its attractiveness to favorable casting properties, (including good fluidity, high impermeability, and low casting shrinkage), the possibility of using a combination of alloying elements, improvements in the refining processes and the possibility of modification of its alloys.

Presented study was conducted for two selected 7xxx series aluminum alloys according to PN-EN 573-3:2010 – Polish version. The analysis of ingots was carried out on 7003, 7003S and 7010, 7010K alloys with a similar ratio of zirconium content. Symbols S and K are our internal modifications, still compatible with the standard. The ingots have been obtained by semi-continuous casting. Aluminum alloys of this series, with improved properties, are intended for plastic-processing. The grains are being formed in a particular way during crystallization. In the middle of the ingot-more far away from the crystallizer- grain are bigger.

Semi-continuous casting together with homogenization enables production of ingots with uniform cross sections as can be seen in the paper.

The aim of this publication is to show how the microstructure of ingots is being formed during semi-continuous casting and to present the results of strength tests, and correlation between basic properties of 7xxx series aluminum alloys, i.e. relative elongation E, yield strength YS and ultimate tensile strength UTS. The quality of Al-Zn-Mg-Cu ingot is strongly related to the functional properties of the final products obtained during plastic working. A key aspect of the casting process is the speed of casting process i.e. velocity of supplying liquid metal to chill mould. This stage is also being influenced by cooling and lubrication. That is why the previous work is an important element on the way of further development of the project related to finding the Quality Index connecting these pavements (macro- and micro structural, strength and ductile properties). Published results constitute the part of PhD work related with Quality Index for aluminum alloys base on Al-Zn-Mg-Cu depending on the heat treatment parameters.

The chemical profiles of ingots have been determined using optical emission spectroscopy. Three tests have been carried out on cross section of each sample: near the edge, in the middle of the cross section and in the middle of its radius.

Chemical analysis in micro-areas with evaluation by scanning electron microscope SEM with EDS analyzer has been performed and the distribution of chemical elements in the microstructures have been also presented. XRD detector has been used to show specific phases in the alloys. Chemical analysis of the microstructure for 7003S alloy has been confirmed by the EDS analysis.

NiSn bimetallic nanoparticles as stable electrocatalysts for methanol oxidation reaction

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Keywords: colloidal synthesis, bimetallic nanoparticles, methanol oxidation reaction, electrocatalysis

Nickel is an excellent alternative catalyst to high cost Pt and Pt-group metals as anode material in direct methanol fuel cells. However, nickel presents a relatively low stability under operation conditions, even in alkaline media. In this work, a synthetic route to produce bimetallic NiSn nanoparticles (NPs) with tuned composition is presented. Through co-reduction of the two metals in the presence of appropriate surfactants, 3–5 nm NiSn NPs with tuned Ni/

Sn ratios were produced. Such NPs were subsequently supported on carbon black and tested for methanol electro-oxidation in alkaline media. Among the different stoichiometries tested, the most Ni-rich alloy exhibited the highest electrocatalytic activity, with mass current density of 820 mA mg⁻¹ at 0.70 V (vs. Hg/HgO). While this activity was comparable to that of pure nickel NPs, NiSn alloys showed highly improved stabilities over periods of 10000 s at 0.70 V. We hypothesize this experimental fact to be associated to the collaborative oxidation of the byproducts of methanol which poison the Ni surface or to the prevention of the tight adsorption of these species on the Ni surface by modifying its surface chemistry or electronic density of states.

Magnetite nanoparticle synthesis and study based on stabilization techniques using different biocompatible coatings

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Keywords: Magnetite nanoparticles, stabilization techniques, washing, gelatine, sucrose

Magnetite nanoparticles with super paramagnetic properties are widely used in many field of science especially for biomedical applications. They could be used for targeted therapies, drug delivery and for precise diagnostic imaging. In view with the application of magnetite particles in biomedicine it is highly important that they are biologically inert and stable. The stability of the magnetite particles using different kinds of polymers and saccharides was studied. However, the use of gelatine as a stabilizer is gaining attention due to its biocompatibility and versatile nature in case of drug carriers. The aim of the study was to stabilize the magnetite suspension prepared using co-precipitation method by both in-situ and ex-situ method. Various concentration of gelatine (0.15 to 2.6 mg/L) was used to stabilize the magnetite. The particles characteristics were also compared based on the washing techniques (magnetic separation and centrifuging) used during magnetite preparation. Saccharides are best-known for coating magnetite and studied to have influence on morphology of the magnetite. The properties of magnetite stabilized ex-situ with given sucrose concentration by varying the gelatine concentration was investigated. Zeta potential analysis showed ex-situ stabilized samples had good stability especially at higher concentrations (1.14 & 1.3 mg/L). The zeta potential value of sucrose and gelatine coating was stable and in the range of 35-45 mV. Visually the ex-situ suspensions were stable when compared to in-situ stabilized samples. The magnetite structure in the samples were confirmed by XRD analysis and the FTIR revealed the peaks of magnetite formation and peptide bonds in case of gelatine coating. SEM analysis showed different structures in the samples based on the added amount of gelatine. With increasing concentration there were formation of sticks. However, the sucrose

with gelatine samples didn't show stick formation at any concentration. The zeta potential of the magnetically separated particles was much stable than centrifuged samples. The results suggest that higher concentration of gelatine (more than 1.3mg/L) is needed for a stable suspension. In addition, we conclude ex-situ stabilization and magnetic washing is better way to synthesize stabilized magnetite suspensions.

Acknowledgments

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Fe-C coatings as an environmental friendly alternative to hard chrome

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Keywords: Electrodeposition, Nanocrystalline, X-ray diffraction, electron microscopy

Strong concerns about the harmful effects of selected chemicals on humans and eco-systems have resulted in EU's REACH regulation to phase out, for example, the use of chromic acid. This has huge consequences for the traditional hard chrome plating as the predominant current process to produce hard and wear resistant coatings for the aerospace, automotive, drilling and military industries.

Electrochemically deposited iron-carbon coatings are a promising alternative to hard chrome plating, as they allow environmental friendly deposition and provide excellent mechanical properties. The coating is seen as environmental friendly in the sense of using none toxic chemicals, working at low temperatures and having a high current efficiency. Proof-of-concept has been achieved on lab-scale for the technical solutions developed for handling the technical difficulties with oxidation in air and pH stability of the electrolyte. Fe-C coatings have successfully been deposited with different thicknesses ranging from few up to hundreds of micrometers. The coatings are nanocrystalline and provide, independent of the substrate material, high microhardness of around 750 HV. The Fe-C coating was subjected to scratch testing and compared with different diamond like carbon coatings and a hard chrome coating, showing a high adhesion of the Fe-C coating on tool steel AISI D2 and good scratch resistance.

The electroplated Fe-C coatings contain about 0.88 wt% carbon, which is homogenously distributed in the coating together with some oxygen and hydrogen as byproducts from the deposition process. Phase identification by means of conventional X-ray diffraction and synchrotron diffraction revealed the presence of bcc-iron, but no carbides or another carbon-containing phase have been identified in the as-deposited state. Neither dedicated electron microscopy investigations could reveal the nature of carbon in these coatings. Results of

complementary methods of materials characterization are discussed to understand the role and type of incorporated carbon, which is presumed to influence the long-term performance of the Fe-C coatings.

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Semi-insulating FeGaN grown using e-Beam PVD: structural and electrical characterization

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Keywords: Transition metal nitrides, III-V semiconductor, semi-insulating GaN, electron microscopy

Over the last few decades, wurtzite III-V nitride materials have received great attention as possible alternative materials to silicon in both optical and electronic applications. Generally, nitride devices are based on the generation of a 2D quantum well at the interface between GaN and a ternary alloy, such as AlGaIn or InGaIn, grown on the top of it. Because of the 2D nature of the quantum well, in nitride devices electrons are spatially confined and fundamental properties, such as electron mobility and carrier concentrations, are higher if compared to similar silicon-based devices.

However, there is still room for improvement: for instance, one of the biggest limitations of nitride-based devices is the lack of an inexpensive high-quality native substrate. Therefore, other foreign substrates, such as silicon or sapphire, are generally used for the growth of a thick GaN buffer layer. However, the lattice mismatch between GaN and these substrates lead to high concentrations of defects and residual carriers. In particular, these residual carriers in the thick GaN layer are responsible for the presence of current leakage that eventually leads to current collapse in the devices, reducing the maximum voltage at which the device can operate [1]. In order to reduce this leakage, one possible approach is to dope the GaN layer with impurities, such as Fe, C or Zn, that can act as acceptors. The presence of these acceptors drastically lowers the concentrations of residual carriers and increases the resistivity of the GaN layer.

Among them, FeGaN is of particular interest as it shows not only semi-insulating properties, but also high crystal quality and ferromagnetic behaviour [2]. However, to successfully employ this material in optical and electronic devices, it is necessary to fully understand both its structural and electrical properties, as well as achieve high-purity level in the thin films. For this reason, in this work e-beam physical vapour deposition (e-beam PVD) is used as alternative growth technique. In this new growth technique, the evaporation of the target is obtained by bombarding its surface using a virtually-pointless electron beam. Therefore, because of the focused and pointless nature of the beam, enhanced purity levels in the thin film are expected.

In addition, the structural and electrical properties of FeGaN thin films grown on sapphire are fully characterized. HR-TEM, STEM, XRD are used to explore the change in the microstructure of all the thin films for different Fe content. SIMS is employed to calculate both the Fe content and the level of impurities in each film. Finally, the electron resistivity and band gaps of these thin film are studied.

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Superhard multilayer CrN/MoN protective coatings as a solution for critical raw materials under extreme conditions

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Keywords: multilayers, microstructure, phase composition, hardness, H/E ratio, friction, wear

The needs of business and industry in materials used under extreme conditions of temperature, loading, friction, corrosion and wear are growing every year. Sometimes it leads to the design of new materials and tools, but more often just to the cheaper and easier increase of raw materials extraction. It decreases the volume of available world natural resources, the access to raw materials becomes more difficult. Together with low recycling level it harms nature and ecosystem.

The authors of the presented research are convinced, that one of the key solutions to overcome the problem, to substitute critical raw materials or significantly reduce their using in industry, is the protective coatings, which will meet a new wave of high attention in nearest years and decades.

Nitride films based on transition and refractory metals exhibit high hardness, high melting temperatures, good chemical and physical stability, which allow to use such coatings as protective ones. Films with micro- or even nanoscale thickness deposited on the surface of machine parts or tools may successfully extend their service life in many times. When coating is destroyed, new layers can be easily deposited on the surface and tools can be in service for a new period of time. In this way, protective coatings provide easy and fast reuse and recycling of products and materials.

In the presented research the authors consider new superhard CrN/MoN coatings with multilayer architecture to substitute already existed analogous with lower level of protective properties and to find new applications in industry.

The synergy of individual features of chromium and molybdenum nitrides was multiplied by control of the microstructure and grain size, which provides increase of the hardness due to the Hall-Petch strengthening and by multilayer design of the coatings, which increase wear resistance and protection via reduction of cracks propagation towards the substrate under the load by the deflection of cracks on the interlayer interfaces.

The highest hardness achieved in the research was 42 GPa and ratio H/E was $\Lambda = 20$ nm for the films deposited at $pN = 0.4$ Pa and bias voltage -20 V. Ratio H/E is a very important parameter for protective coatings evaluation and must be equal or higher than 0.1 for films to be characterised by high resistance to the wear and load. Thereby, CrN/MoN coatings with such parameters are highly recommended for industrial applications as protective ones [1].

It should be noted, that deposition techniques used in the research (magnetron sputtering and vacuum-arc evaporation of cathodes) are eco-friendly, don't pollute the environment and don't require high amount of materials. Moreover, they don't use or produce harmful hexavalent chromium (chromium 6) or chromium trioxide CrO_3 which are carcinogenic (and nowadays become to be prohibited by European regulations) but involved in some other technologies of coatings fabrication as electrochemical process.

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Improved Polymers with embedded nanoparticles for lightweight construction

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The enhancement of mechanical properties of various thermoplastic and thermosetting polymers, such as scratch resistance and hardness, are strongly desired for lightweight constructions. The embedding of inorganic nanoparticles into a polymer resin is widely utilized and shows promising results. In this way, not only the nanoparticles themselves but the specific surface modification of the used particles can significantly influence a number of properties of the intermediate formulation that are relevant to processing, such as viscosity or curing behavior.

One crucial factor for the mechanical properties of nanocomposites is the particle-matrix interface, which is strongly influenced by this surface chemistry: It determines the colloidal stability and therefore, the particle distribution in the matrix. An adjustment of the surface chemistry can be used to optimize the interface with a specific matrix and thus enhance the mechanical properties of the composite. However, since there are several possible mechanisms of mechanical reinforcement in nanocomposites that influence the structure-property relations and a multitude of materials combinations can be utilized for the nanoparticles and

the matrix, specially tailored organic ligands are often required that bind to the nanoparticle surface and ensure an optimized interface. On the one hand, these ligands need to possess functional groups which covalently attach to the nanoparticle surface to ensure permanent binding. On the other hand, they must show good compatibility or even strong chemical interaction with the polymeric matrix. Such ligands are usually not commercially available or costly and therefore difficult to investigate.

In this contribution, we present a covalent attachment of a standard aminosilane to the surface of commercial boehmite nanoparticles. Subsequently, the surface chemistry can precisely be adjusted using an amide-coupling strategy by linkage with an appropriate carboxylic acid. We thoroughly investigated and optimized the reaction conditions since this facile and versatile strategy enables the covalent modification of nanoparticles with a broad variety of terminal moieties even for product quantities of 100 grams. The modified particles were incorporated into a polymer resin that was subsequently cured. Standard test pieces were characterized to determine the mechanical properties. Thereby, we demonstrate that a particle surface modification can lead to a significant enhancement of the mechanical properties of the composite even at low filler content.

Micropollutants treatment based on textiles functionalized with polymers network

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Keywords: Micropollutants, PFCs, polyvinylamine, textile filter

In recent years, the occurrence of micropollutants in the environment become a subject of public concern, due to the potential negative effects on aquatic ecosystems and human health. Pesticides, pharmaceuticals and perfluorinated compounds (PFCs), are widely distributed and persisted in the environment due to their massive used and highly stability. PFCs were detected in surface-, ground-, and even drinking water in ranges from ng/L to several µg/L.

PFCs are not removed by drinking water treatment processes. Activated carbon is effective for long chain PFC, but not for problematic shorter chain ones. Textile based adsorbents has been successfully used for the adsorption of chromium (VI)² and noble metals³. Recently, researches showed cheap and sustainable insoluble β-cyclodextrin based polymers are capable of removing micropollutants e.g. pharmaceuticals and PFCs from water by adsorption⁴. The

disadvantage of β -cyclodextrin based polymers is a low surface areas and poor removal performance compared to conventional activated carbons.

In this work, micropollutants absorbing textile filter material was developed based on coated polyester fibers with a grafted β -cyclodextrin modified polyvinylamine, polyvinylguanidine or others. The surface modification of the textile material is possible with common textile finishing methods, yielding a durable, high-performing and even cheap adsorbent for pharmaceuticals and PFCs from water.

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Dissolution tests of a selective soldering tool material combination with enhanced lifetime in a calm SAC309 solder melt

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Nowadays, soldering has an increasing role in the electronics industry. The largest amount of PCBs are produced using this technology. In selective wave soldering, the most critical point is the soldering tool, since during soldering, the aggressive lead-free Sn-based solder melt dissolves the Fe atoms of the tool. This process causes surface damage, pitting on the surface of the soldering tools, thus, decrease the wetting and then ceases the stable solder wave required for precise soldering. This failure process takes place quite rapidly (maximal operation of a tool is 1 month) and demands the replacement and adjustment of the tool. This results a lack in production and increases costs. Preventing the failure process is therefore a major challenge and has a big importance for the soldering industry. In this article, material combinations with proper wetting with SAC309 solder alloy have been developed using various steel types. To examine their dissolution resistance, a dissolution simulator was created,

in which the samples were submerged into calm SAC309 solder melt for 10, 20 and 40-days. The microstructure of the tested samples was investigated with a scanning electron microscope (SEM), and EDS (energy dispersive spectrometry) line scans to search for traces of Fe dissolution or any other degradation processes.

Development of melt-spinnable PES/PA blends for the substitution of PA filaments in textile applications

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Keywords: polymerblend

Polyester (PES) is the most widespread synthetic chemical fibre in the textile market, accounting for 82% of all filament yarns with a production volume of 35,2 million tons in 2015¹. PES is widely used due to its good thermal and mechanical properties, excellent dyeing behaviour and the favourable raw material price (0.9–1.3 €/kg). Polyamide (PA) accounts for 11 % of the market share with a production volume of 4,7 million tons in 2015¹. PA has excellent abrasion resistance and lower dyeing temperatures compared to PES. PA is thus more resistant to wear and tear. However, PA with an average cost of 2.5–3 €/kg is round about 1.5 €/kg more expensive compared to PES. The aim is to develop a polymer blend from PES and PA to combine the properties of both polymers in one filament yarn for textile applications like ropes, carpet yarn, belts and sportswear.

The blends of PET/PA blends are produced via melt mixing using a co-rotating twin screw extruder. The addition of compatibility agents (like PES/PA-copolymers, maleic anhydride or functionalized epoxy resins) is avoided for economic reasons. Compatibility of the PES/PA blend is achieved by in-situ copolymer formation during solid-state post-condensation (SSP). SSP is normally used to adjust the intrinsic viscosity of PES prior to melt spinning of high tenacity PES yarn. The resulting PES/PA blend is spun to mono- and multifilament yarns. The resulting blend and yarn properties are characterized. Scanning electron microscopy is used to characterize blend morphology, and intrinsic viscosity measurement is used to evaluate the compatibility. Crystallinity and crystalline structure is characterized using differential scanning calorimetry and wide-angle X-ray diffraction. Tensile tests are used to characterize the mechanical properties of the yarn. Finally a knitted fabric is produced and dyeing tests are carried out to characterize the dyeing behaviour of the blended textile. An overview about the development process for PES/PA textiles is shown in Figure 1.

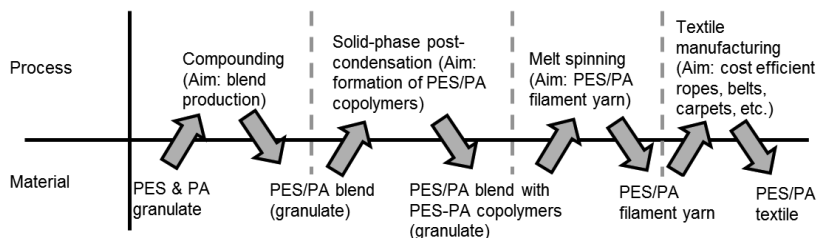


Figure 1: Overview of process level and material level for the production of PES/PA textiles

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Optical properties of ALD deposited ZnO as a functional sensing layer based on self-assembled mesoporous membranes

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Keywords: zincoxide, nanostructures, photoluminescence, optical spectrometry, AAO

Large scale nanopatterns can be generated by using anodic aluminum oxide (AAO) masks which are porous alumina membranes fabricated in an electrochemical dissolution of aluminum in an acidic electrolyte. AAO is utilized as a self-assembled mesoporous template for producing an ordered nano-array of zinc oxide (ZnO) via atomic layer deposition (ALD). Its structure is well controllable and the correlation with optical properties can be closely studied.

The characterization of structural properties by scanning electron microscopy (SEM) and atomic force microscopy (AFM), as well as X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDX) and optical spectrometry, reveals the dependence of template based ZnO growth and microstructure on the process parameters during sample synthesis. The results show that the emission wavelength of nanostructured ZnO is tunable. The applicability of AAO embedded ZnO structures as a functional sensing layer is evaluated and discussed.

Growth and study of NiO wires and ordered cavities showing optical resonant modes

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Keywords: NiO, microstructures, luminescence, optical resonance

Nickel oxide (NiO) is a wide band gap p-type semiconductor, which presents excellent electrical, optical and magnetic properties, as well as excellent chemical and thermal stability. This material has recently demonstrated potential applicability in electrochemical capacitors, alkaline batteries, smart windows and gas sensing, among others. NiO is usually synthesized in form of nanoparticles, ceramic, or thin films, and less has been done in the fabrication of micro- and nanostructures with variable morphology, so far.

In this work, a vapor-solid method, using Ni metallic powders as precursor, has been employed to obtain different NiO micro- and nanostructures. Thermal treatments were carried out at temperatures ranging from 800 to 1500 °C during 10 hours under a controlled argon flow. Treatments at temperatures of 800 and 900 °C lead to the growth of rods some microns length with a low aspect ratio. Surface reconstruction and texturing are achieved by increasing the temperature of the treatment. Using temperatures between 1000 °C and 1400 °C large microcrystals are grown on the surface of the treated pellet, most of them exhibiting surfaces with ordered hollow cavities with square sections of hundreds of nm (Fig. 1a). Besides, some other regions exhibit a layered structure formed by piled terraces tens of nm high. In the sample grown at 1400 °C, NiO wires with lengths of several microns are also obtained mainly at the edge of the pellet (Fig. 1b). Finally increasing the temperature up to 1500 °C leads to the formation of terraced structures without wires or ordered cavities.

The characterization of the microstructures was performed by X-ray diffraction (XRD), confirming that all the analyzed samples can be indexed with the NiO rock-salt structure. Scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS) and Raman spectroscopy have been employed for the morphological, compositional and structural study. Cathodoluminescence (CL) and photoluminescence (PL) measurements indicate the presence of emissions at 1.7 eV, 2.4 eV, 3.1 eV and a wide emission centered at 4.4 eV, the origin of which is under discussion. Moreover, optical resonances have been observed in some of the PL spectra, as shown in Figure 1c, which confirms their behavior as resonant optical cavities.

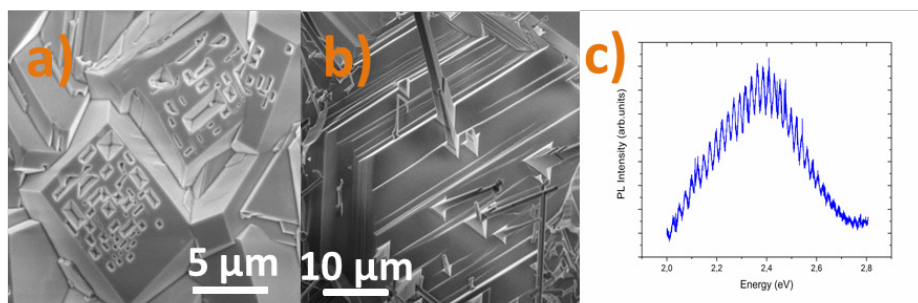


Figure 1: (a-b) SEM images of different NiO microstructures and (c) PL spectrum showing optical resonances

Superelastic fatigue of NiTi filaments and coupling between plasticity and martensitic transformation

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Near-equiatomic intermetallic NiTi alloy, well-known under the commercial name Nitinol, shows superelastic and shape memory properties, which is utilized in a wide range of branches such as medicine, aerospace and automotive. Theoretically, superelastic loading should be feasible in a range of austenite finish and martensite desist temperatures. However, coupling of martensitic transformation with plastic deformation occurs and becomes more pronounced at elevated temperatures, where transformation stresses increase even above 1000 MPa. In this work, a nonconventional method of pulse heating by electric current was employed to recover cold-worked NiTi filaments (100 μm in diameter) and set the initial microstructure (approx. 100 nm grain size without Ni-rich precipitates). Characterisation of stress-strain behaviour in a wide temperature range (−90–200 °C) and fatigue tests of the filaments were carried out to determine a superelastic fatigue life and stability of functional properties such as transformation stresses and transformation strain. Transmission electron microscopy has also been performed to find out changes in the microstructure after superelastic loading. Considerable increase of dislocation density at temperatures above 80 °C correlates with instability of superelastic cycles and increase in permanent strain as transformation stresses rise. Accumulation of permanent strain in the first superelastic cycles can be used as a criterion for prediction of the fully superelastic fatigue life of the tested filaments. The experiments also shows that the reverse transformation from martensite to austenite causes more damage than the forward one, even though reverse transformation stress is lower.

Development of an austenitic/martensitic gradient steel by additive manufacturing

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Keywords: Direct Metal Deposit, additive manufacturing, austenitic steel, martensitic steel, gradient material

These last years, additive manufacturing as gain interest to build, layer-by-layer, component with complex shapes in many applications. With the Direct Metal Deposit (DMD) process, a nozzle delivers the powder directly to the molten pool, generated beneath the laser beam. An advantage of this process is the possibility to tune the material's composition, using different powder feeder, through the powder flow. Thus, it became possible to build composition gradient parts. Gradient components are studied for industrial applications such as functional materials ([1], [2]) or as a way to solve welding problems ([3], [4]). In this study, we focused on a gradient from a 316L austenitic stainless steel to a 9Cr-1Mo martensitic steel. These steels are widely used for nuclear applications, but due to their chemical composition differences, welding them is uneasy.

Three different manufacturing processes are studied, Spark Plasma Sintering, High Iso-static Pressure and Direct Metal Deposit. Sample's microstructure is analyzed using Scanning Electron Microscope (SEM) and Electron BackScatter diffraction (EBSD) and correlated to thermokinetics calculations. The relationships between the mechanical behavior, the microstructures and the processes are discussed.

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Investigations on the microstructure and corrosion behavior of as-cast Mg-Si alloys

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Keywords: magnesium alloys, microstructure, corrosion

Magnesium alloys specially designed for biodegradable implant applications have attracted great attentions in recent years. From the perspective of element biosafety and dietetics, the ideal alloying elements suitable for biodegradable magnesium alloys should be those essential to or naturally presented in human body. Silicon is an essential trace element for bone development. It was reported that Si only located in the active areas of young bone and is involved in the early stage of bone calcification [1]. In this study, the as-cast Mg-Si alloys with different Si contents were prepared through a direct chill casting process in a permanent mould [2]. The microstructure and XRD patterns for the binary alloy specimens are shown in Fig. 1. The results showed that the as-cast Mg-Si alloys consisted of dendrites of α -Mg matrix (bright white dendrites) and eutectic phases (α -Mg + Mg_2Si , network shaped) at the interdendritic regions. The intensity of Mg_2Si phase diffraction peak increases with the increase of Si content. The corrosion properties are also evaluated using mass loss, hydrogen evolution and electrochemical tests. The aim of the present work is to develop and characterize new magnesium-based biomedical implant materials with aluminum-free.

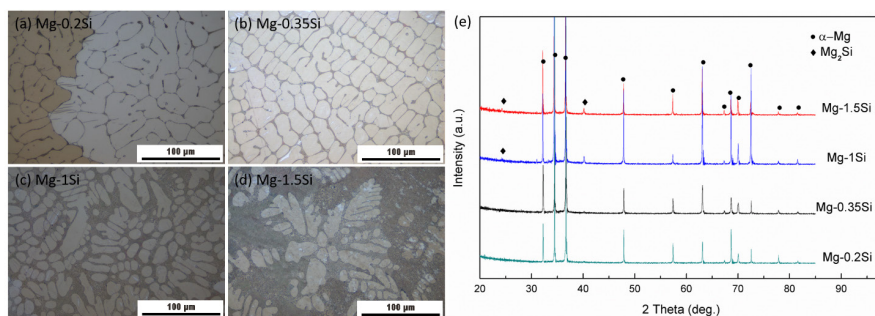


Figure 1. (a)-(d) Microstructures of as-cast Mg-Si alloys; (e) XRD patterns

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Poster

Fiber drawings from viscous Al-containing systems

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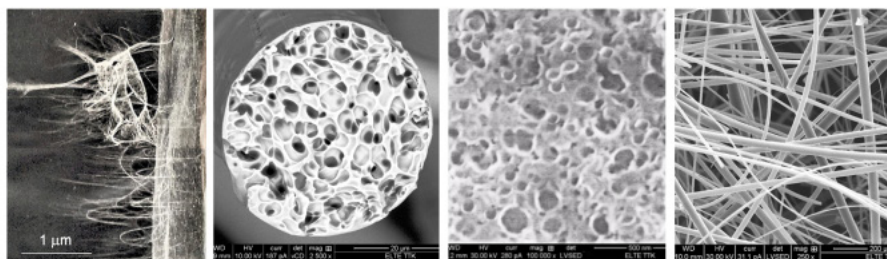
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Two types of aluminum oxide fibers were aimed to produce by sol-gel technique; nanofibers by electrospinning (\varnothing : 10–50 nm, Fig. 1) and porous fibers by centrifugal solvent spinning (Fig. 2, 3). The nanofibers proved to be very fragile, the fibers drawn by dry spinning technique are too thick (\varnothing : $\geq 200 \mu\text{m}$) for the drying. The thick fibers come to pieces during the heat treatment due to the gas escapes. Regarding the drying, the ideal diameter of fibers is between 1 and 50 μm . The diameter of fibers drawn by centrifugal solvent spinning technique varies from 2 to 20 μm , and the average diameter is 10 μm . The pore sizes are changed between 5 nm and 2 μm depending on the drawing and drying parameters. The porosity is altered from 55% to 80%. The fibers can be applied up to 1600 °C, the fibers are not be degraded or crumbled, however both the bond systems and the crystalline structure are modified. The porous fibers with about 10 μm diameter can be well applied as an insulating materials.

A new method has been developed for the procedure of the alumina fibers. The new method has a low energy consumption. The reaction temperature is 80 °C and the heat treatment is carried out at 500–600 °C. Only two materials are needed for the synthesis; an inorganic Al salt and an organic solvent. Basic or chelating agents are not necessary to apply for the hydrolysis and condensation reactions. High Al(III) concentration ($> 5 \text{ w/w}\%$) is necessary for the condensation. The viscosity required for the drawing can be reliably controlled by the solvent content; the Al(III) concentration of about 10 w/w% is favourable.

The characterization of fibers is provided by scanning electron microscope (SEM) and wide angle X-ray scattering (WAXS). SEM images represent the size and the porous structure of fibers. (See the SEM images!). WAXS measurements identify the amorphous character of the aluminum oxide-hydroxide fibers up to 400–500 °C and the poor crystallinity of mixed Al_2O_3 phases above 600 °C.

Figure 1. Scanning electron microscope images of fibers



Fibers by electrospinning (1); fibers by centrifugal solvent spinning technique (2–4)

Production of inorganic polymer foams with combined foaming process (GSP)

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Keywords: inorganic polymer, alkali activation, foaming, open porosity, compressive strength, X-ray diffraction, FT-IR spectroscopy, Scanning electron microscopy, CHNS analysis, photocatalytic degradation process

Alkali activated inorganic polymers (AAIP) are not only future but today's structural materials, which are intensively investigated research area. Besides that AAIPs can be used to replace cement-based traditional binders due to their high strength, these materials can also easily be well foamed. Therefore the number of research about AAIP foaming has increased steadily in recent years. These foams are eco-friendly porous materials, which have favourable production conditions, and promising properties (low shrinkage after foaming, mechanical and chemical stability, high temperature resistance, etc.). There are several methods producing foams (e.g. mechanical frothing, H_2O_2 decomposition, freeze-casting techniques) but foams with sufficiently high open porosity (~ 70 vol %) can be produced by gelcasting/saponification/peroxide decomposition (GSP) combined method. However, the low compressive strength (~ 0.45 MPa) of the foams limits their application potentials.

My aim was to optimize the relationship between the compressive strength and the open porosity of AAIP foams, which allows the use of these materials as a catalyst support for photocatalytic sewage treatment processes.

In the study metakaolinite-based AAIP foams were produced by GSP combined method. As an activating component the mixture of NaOH and water glass were used, and different vegetable oils, sodium oleate and hydrogen peroxide were added in order for saponification reaction and foaming. The combined foaming method allowed the production of AAIP foams with designed open porosity. Based on the relevant physical properties of the samples (compressive strength, open porosity, thermal conductivity) the optimal composition was chosen: sunflower oil and 4,5 vol % H_2O_2 solution (N-4,5). Furthermore the relationship between compressive strength and structure of the AAIP foams was investigated, using FT-IR for polymer structure examination, XRD for phase composition determination (?) and SEM for morphological studies. The organic matter content of the samples is a major problem, which can be reduced by the reduction of the oil content and by calcining the specimen with appropriate firing parameters. The organic matter content of the test specimens was monitored by CHNS analysis. The produced AAIP foams can be used as a catalyst support in photocatalytic degradation processes.

Acknowledgments

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Effect of structure on corrosion resistance of Mg-Zn-Ca alloy

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Keywords: magnesium alloy, biodegradation, corrosion, immersion test, SBF, amorphous structure

The structure of materials is extremely interesting and broad topic. This is due to the fact that it is possible to link it to the properties of the materials. By knowing the exact impact of a particular microstructure on the properties of the alloy, it is possible to better understand the processes occurring within the material and it enables the perfect design of materials with desired properties. Metallic materials typically have a crystalline structure. It is described as a long-distance order, manifested by geometrically ordered arrangement of the atoms resulting in lattice structure. But this is not the only structure present in metal alloys. They may also exist in an amorphous form. Amorphous state is characterized by short-range ordering but lacking long range order within the lattice. Properties of metallic materials mainly depend on the microstructure. That is why it so important whether it will be fully crystalline, mixed or completely amorphous. Properties of amorphous materials in fact, may be much different than the properties of their counterparts with crystalline or mixed structure [1–2]. In recent years, more and more interest is placed on Mg-Zn-Ca alloys [1–3]. This is due to their physical and mechanical properties, which may include: a low density, high strength and high ductility [4–5]. High popularity of these alloys is also affected by their biological properties such as biocompatibility and bioresorbability [1]. In addition, these materials are characterized by a relatively low price. Taking into account the abovementioned, in this research, an attempt will be made to find the relationship between the structure of the analyzed $\text{Mg}_{67}\text{Zn}_{29}\text{Ca}_4$ alloy and their corrosion resistance under physiological conditions. $\text{Mg}_{67}\text{Zn}_{29}\text{Ca}_4$ alloy were prepared using high purity elements (99,99%) in a vacuum atmosphere. Rods with 3 mm diameter were obtained using cooper mould casting method. Samples were cut to 3 mm long rods and than annealed in fixed temperature with different times. The parameters of the annealing process are important due to fact they allow to control the structure of the materials. After annealing the x-ray diffraction (XRD) was carried out. Diffraction techniques were used to determine whether the alloy has a crystalline, mixed or fully amorphous structure. The corrosion rate under physiological condition was tested by the immersion test. The effect of material's structure on corrosion resistance was determined. According to experimental results, the corrosion resistance of the rapid solidified $\text{Mg}_{67}\text{Zn}_{29}\text{Ca}_4$ samples changes with structure.

Acknowledgments

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Investigation of the effect of undercoat on thermal spray deposits elaborate on aluminum substrate

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Keywords: Electroless deposition, Ni-Zn-P alloys, corrosion, aluminum, thermal spray

The natural alumina oxide layer is a barrier layer to all coatings. Ni-P et Ni-Zn-P alloys coatings were prepared onto aluminum substrates with different concentrations of zinc ions using electroless plating method with sodium hypophosphite as reducer. The deposits were used as undercoat for thermal spray. Different techniques such as scanning electron microscopy (SEM), energy dispersive X-ray (EDX) and X-ray diffraction (XRD) were employed to characterize the morphology, composition and the structural properties of the resulting films. The effect of the undercoat layer was evaluated. The corrosion properties of the prepared coatings were tested in 3% NaCl media, by means of current-potential curves, potential transients. The result shows that the undercoat layer improves adhesion of the thermal spray coating. A significantly, reduces of corrosion rate.

Investigation of the effect of milling conditions on the morphological and structural properties of hot-isostatic pressed h-BN/Si₃N₄ ceramic composites

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Silicon nitride (Si₃N₄) ceramics containing 1 and 5 wt% of hexagonal boron nitride (h-BN) were prepared by attrition milling and hot-isostatic pressing. Thorough morphological characterizations have been carried out to reveal the influence of the milling parameters on the size of the h-BN additives. The results confirmed significant decrease in h-BN particle size by increasing milling time. The transmission electron microscopy (TEM) measurements revealed that the h-BN particles were incorporated into the ceramic matrix. The results showed that the increase of the h-BN content decreased significantly the hardness of materials. Moreover, the hardness values were higher when the size of h-BN was higher. The same tendency was observed in the case of Young's modulus.

Influence of surface treatment on corrosion and other electrochemical properties of titanium

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Keywords: titanium, colouring, corrosion properties, polarization, biocompatibility

Titanium is widely used across many medicinal fields like implantology or surgery. Electrochemical colouring of titanium tools or implants is one of the common ways how to differentiate different sizes or types of each application. Titanium grade 4 plates 50 x 20 x 0.1mm were tested to obtain their electrochemical and other technological properties which can affect especially their biocompatibility if contacted to living organism.

There were many papers or research studies published regarding the colouring process, but this one tries to fill up “research gap” and describe influence of this colouring process on electrochemical surface parameters. Part of surface of each sample was anodized in fluorine ions containing solution using 60 V potential for 5 minutes. This process is also used in implantology to increase free surface of implants. After that colouring process was done using potential of 15, 30, 45, 60 and 75 Volts for 5s. Solution of 1 wt. % citric acid in demineralized water was used for electrochemical colouring of each sample.

Sessile drop method was used to obtain contact angle of demineralized water on free surface and there was significant difference of contact angle observed for anodized + coloured surface against only coloured surface. Electrochemical impedance spectroscopy was used

for determination of some surface layers parameters like capacitance or resistance. Potentiodynamic polarization was used for corrosion testing of this samples and corrosion potential, polarization resistance or corrosion rate of each sample was found using Taffel or Stern method. There was found that anodization process before colouring decreases significantly corrosion potential. There was also found that higher potential used for colouring results in higher polarization resistance but also decreases corrosion potential. Main results of polarization tests for only coloured samples tested in aerated physiological solution are shown in followed table. According to these results anodization process before colouring can be recommended as it increases corrosion rate only insignificantly but ensure that resulting surface is highly porous and biocompatible for living cells. If anodization is not applied, colouring at 75V results into lowest corrosion rate under 1nm/year and the most noble corrosion potential.

Table

Sample	Colouring voltage (V)	E_{corr} vs. SCE (mV)	Corr. rate (nm/year)	Polar.resistance (kOhm*cm ²)
1	15	-336	2.1	63
2	30	-278	1.1	79
3	45	-256	1.5	85
4	60	-221	1	82
5	75	-194	0.7	126

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Biodegradable polymeric blends based on thermoplastic starch and poly (butylene adipate-co-terephthalate)

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Keywords: biodegradable, thermoplastic starch, polymer blend, plasticizer

The goal of the experiment was to demonstrate the influence of composition of polymeric blends consisting of thermoplastic starch (TPS) and poly(butylene adipate-co-terephthalate) (PBAT). We investigated tensile strength and elongation at break as the two most important mechanical parameters. We prepared samples with two different proportions of the individual components of the mixture (PBAT/TPS = 90/10; 60/40), using two types of plasticizer (glycerol, urea) to prepare TPS.

TPS was prepared from mixture of natural cornstarch, water and glycerol or urea. Thermoplastic starch matter was prepared in four consecutive steps. Components were mixed at increased temperature and then dried, kneaded in Brabender at 130 °C and compression-molded. Two ways to blend preparation were tested and we examined the influence of the preparation process on the resulting material properties. In the first case, we prepared the plasticized starch by described procedure in the first step and subsequently mixed it with the polymer. In order to simplify the preparatory process, we used the second alternative, i.e. we added the dried starch mixture into the Brabender chamber in one step with the PBAT.

Compared to PBAT without starch, the values of tensile strength and elongation at break of starch-containing PBAT were lower and did not differ significantly regardless the amount of TPS present. The tensile strength values remain almost the same, except for the both samples containing 40 wt% of TPS plasticized by glycerol.

The values of elongation at break also drop down with addition of TPS and the decrease was more pronounced in the case of all samples containing 40 wt% of TPS. The decrease of tensile strength and simultaneous decrease of elongation at break can be justified by immiscibility on molecular level.

When comparing the two procedures of blends preparation the two-step mixing did not provide a significant improvement in mechanical properties.

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Hardness improvement and corrosion resistance examination of nitrided Ti-6Al-4V ELI biomedical material

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Keywords: nitriding, titanium alloy, Ti-6Al-4V, corrosion, surface modification

Titanium and titanium alloys are widely used in biomedical devices and components. Ti-6Al-4V ELI is mostly used for orthopaedic and spinal implants. This alloy has good biocompatibility, formability, machinability and corrosion resistance. However, its hardness and wear resistance can be improved. Therefore, surface modification is necessary and there are numerous surface engineering methods to enhance its tribological performance [1].

Nitriding is a thermochemical surface treatment which can improve the hardness and wear resistance of the surface. Nitrogen diffusion hardening creates $\text{TiN} + \text{Ti}_2\text{N} + \alpha\text{Ti(N)}$ in the top layer of the material and leads to an increased surface roughness [2], [3]. The cell viability after 5 days is also better on the nitrided titanium alloy, so the biocompatibility can be increased with the treatment [4]. However, the nitriding parameters for titanium are not well defined, and direct current plasma nitriding (DCPN) can cause irregularity on the surface.

In this experiment Ti-6Al-4V ELI disks were treated by various plasma nitriding methods (conventional plasma nitriding – DCPN and active screen plasma nitriding – ASPN) as well as gas-nitriding. Surface hardness was measured before and after nitriding. The treated samples were cut in half for cross section examination with scanning electron microscope. The corrosion rate of the material is determined by potentiodynamic measurements with a potentiostat. The final aim of the research project is to improve the properties of spinal screws and other implants with surface treatment for reaching better wear and corrosion resistance.

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The Formation of Tb doped TiO₂ thin films using thermal oxidation method

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Keywords: Tb-TiO₂ film; thermal oxidation; UV induced photocatalysis; co-sputtering, microstructure

In this study, Tb doped TiO₂ (Tb-TiO₂) thin films were deposited by RF and DC magnetron co-sputtering, which were formed using thermal oxidation. The effects of the constitution and annealing temperature on the characteristics of TiO₂ thin films were investigated. The crystalline structures, morphological features and photocatalytic activity of TiO₂ films were systematically studied by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM) and ultraviolet spectrophotometer, respectively. Furthermore, photocatalytic activity of Tb-TiO₂ thin film was evaluated using degradation methylene blue aqueous solution. The Tb-TiO₂ film thickness was about 200 nm after annealing at 550 °C. The transmittance and hydrophobic of film can be significantly improved after annealing. There are only rutile phase in TiO₂ film with terbium addition. The degradation rate of methylene blue was 46.6% under UV-C light irradiation for 3 hours.

Polyester fabrics modified with zwitterionic polymer brushes to reduce biofilm formation

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Keywords: Anti-Biofouling, lubricant-infused surfaces, Zwitterions, polymer brushes, polyester fabric

Biofouling is a problem affecting the efficacy of a broad range of systems and applications. These include implants, biosensors or textiles used in the medical sector, where the contamination by bacteria and microorganisms is a major cause of infections.¹

In recent years, lubricant-infused surfaces have been found to reduce lateral adhesion of all types of liquids and to induce bacteria and microorganisms to slide off.²

A major drawback concerning these surfaces is that liquid lubricants are easily removed by shear stress or evaporation. Several strategies have been adopted to limit the depletion of the lubricant, yet further efforts are needed to improve the performances of slippery surfaces. A long term stability might be achieved using highly hydrated polymer brushes to replace the lubricating layer. PEG or zwitterionic polymers proved to be highly effective for this purpose, since several coatings that prevent the attachment of proteins and bacteria have already been synthesized.^{3,4}

In this work, we aim at functionalise a polyester fabric with zwitterionic brushes. The high motility of the polymer chains and the strong hydration effect associated with the charges make these materials promising candidates for the fabrication of lubricant-infused surfaces.

Acknowledgments

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Formation and analysis of interphase between carbon fibres and carbide forming elements in Mg based composites

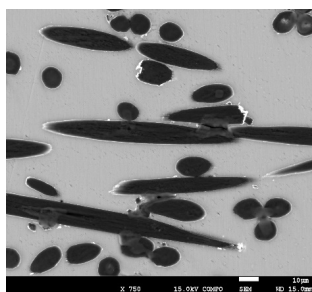
Štefan Nagy^{1}, Pavol Štefánik¹, Stanislav Kudela Jr.¹, Martin Nosko¹, Ľubomír Orovčík¹, Andrej Opálek¹, František Šimančík¹, Karol Iždinský¹*

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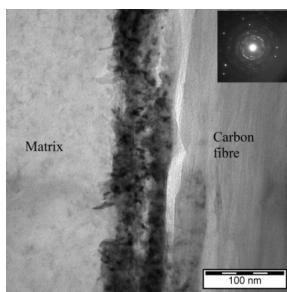
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Keywords: metal matrix composite, magnesium, carbon fibre, interface

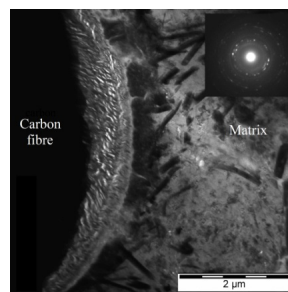
Metal matrix composites (MMC) based on magnesium matrix reinforced with carbon fibres are promising candidates for lightweight structures where appropriate interface formation between matrix and reinforcement is essential to assure required properties of the MMC. In this paper characterization of the microstructure and interface between carbon fibres composite in combination of Mg based matrix is discussed. MMC was prepared through gas pressure infiltration of pitch based short carbon fibres by liquid magnesium alloyed with different carbon forming elements (Zr, Y) to create interface reaction between the fibres and matrix. The microstructure and the interlayer were investigated by scanning electron microscopy, EDS analysis and high resolution scanning transmission electron microscopy. To support identification of the interfacial phases, X-ray diffraction (XRD) was conducted. Reaction layer on the interface between carbon fibre and matrix consist mostly of thin interaction layer based on yttrium carbides, ZrC and MgO.



SEM micrograph of the microstructure
C-fibres/MgZr



TEM micrograph of the interface
C-fibres/MgZr



TEM micrograph of the interface
C-fibres/MgY

Acknowledgments

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Cu-ZrB₂ composites prepared by gass pressure infiltration

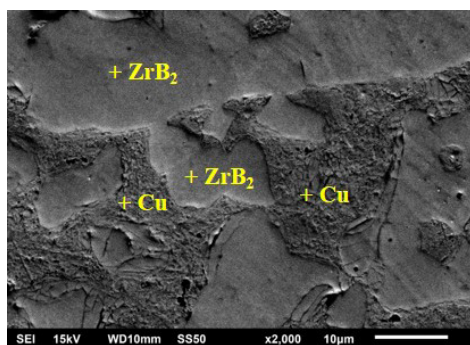
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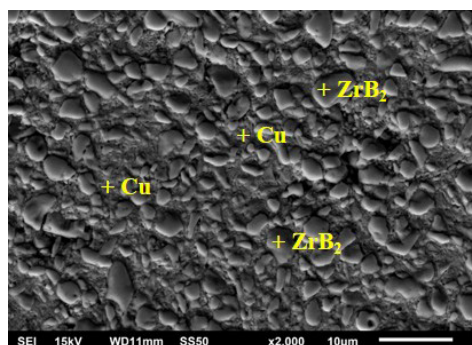
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Keywords: Cu-ZrB₂ composite materials, Pressure infiltration, Thermal treatment

Composite materials based on Cu matrix have wide applications range thanks to extraordinary features (high electrical/thermal conductivity and high mechanical strength). In this work is described preparation of Cu-ZrB₂ composites by pressure gas infiltration technology. In contrast to most ceramic materials, ZrB₂ is electrically and thermally conductive. In both prepared types, ZrB₂ porous preform with porosity of 60% as well as ZrB₂ powder with particle size < 5 μm (D50 = 2.81 μm) were infiltrated with molten Cu. Microstructure and samples homogeneity after infiltration were observed by using of SEM-EDS microscopy and 3-D computed tomography. Interface between Cu matrix and ZrB₂ was observed via TEM microscopy. Both types of Cu/ZrB₂ composites were thermocycled up to 800 °C with heating and cooling rate of 3 °C/min in argon atmosphere.



SEM microstructure of ZrB₂ porous preform infiltrated with Cu



SEM microstructure of ZrB₂ powder infiltrated with Cu

Acknowledgments

This work was supported by Slovak Foundation VEGA Grant 2/0172/16 and by Grant APVV-14-0936. Some results were acquired using infrastructure built during the projects ITMS 26240120006/ ITMS 26240120020.

Growth of boron-doped carbon nanowalls on quartz glass

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Keywords: carbon nanowall, CVD, transparent

The optically transparent and electrically conductive material constitute as an important component in a number of electronic devices nowadays.

In this study, we have investigated the growth of boron-doped carbon nanowalls (B-CNW), under different deposition time, on optical grade fused silica substrates as alternatives to indium-based transparent conductive electrode materials.

B-CNW were fabricated by a microwave plasma-enhanced chemical vapor deposition (MWPECVD). Fused silica substrates were initially pretreated for 20 minutes in the MWPECVD reactor, with a flow of H₂, at 500°C with 1000W of microwave power to modify the surface. Next, substrates were seeded by spin-coating with nanodiamond slurry. Finally, B-CNW films were grown on fused silica glass, optical grade, using a gas mixture of H₂, CH₄, N₂, B₂H₆ with a total flow of 328 sccm, at a process pressure of 50 Torr. Microwave power was set to 1300 W and the substrate holder was heated up to 700°C by an induction heater and controlled by a thermocouple.

Electrical conductivity was determined at room temperature by four-point probe, optical bandgap was estimated by Tauc plot utilizing light absorbance was measured by UV-vis spectra.

Process duration affect strongly the thickness of the nanowall films. Longer times of growth lead to a thicker and higher conductivity films, jointly with well-developed nanowall morphology. Simultaneously, the optical transmittance was decreased. Light absorbance in the 350–700 nm range shows values from 0.29 to 0.97, while calculated optical bandgap decreases, from 3.8eV to 3.4eV, for nanowall growth respectively for 7.5 mins and 30 mins.

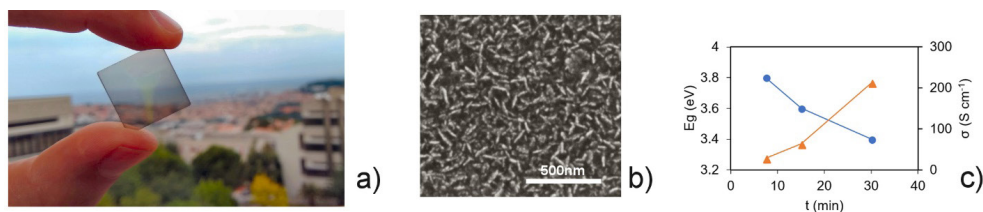


Figure 1. B-CNW growth on fused quartz. a) picture and b) SEM image of the specimen growth for 7.5 min. c) electrical conductivity and optical bandgap of the specimen growth at different temperatures.

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Are nanoparticles a solution to stress whitening in pre-painted metal systems?

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Polyester resins are used as a binder in a variety of industrial coil coatings for pre-painted metal systems. A specific aspect of coatings for pre-paint systems is their increased need to be flexible, whilst still maintaining high toughness and durability, as bending and shaping of the steel substrate occurs after the coating has been applied¹. One defect which can occur during forming is stress whitening, this appears as whitening along an area where high strain has occurred^{2–4}. Current research suggests stress whitening is the macroscopic visual appearance of scattered light from nanometre sized cracks and crazes in the coating^{2–8}.

Nanoparticles are a potential solution to this problem for a number of reasons. Nanoparticles included in coatings previously have been used to prolong strength by spreading crack propagation throughout the material in a dendritic or branching pattern of smaller cracks, as opposed to a single large crack⁹. The lower limit for cracks causing stress whitening appears to be around 100nm. Therefore if the nanoparticles can limit crack size to under 100nm then stress whitening should not be visible. Nanoparticles generally also improve the properties of the overall material by imbuing with their own properties^{3,10,11}. Because of this, super-elastic nanoparticles were one of the varieties used.

Both incorporation in the resin binder as part of the resin synthesis stage and incorporation into the coating system were considered. Several variants of nanoparticle were also investigated, and the resulting coatings were all compared to determine which, if any, had prevented stress whitening. The resins and coatings underwent various forms of analysis, including scanning electron microscopy and X-ray spectroscopy, to allow further evaluation of the results.

Acknowledgments

The author would like to thank David Gethin and Cris Arnold, of Swansea University; Chris Lowe, Susan Willis and Andrew Higham of Becker Industrial Coatings. Thank you to the EPSRC and the European Social Fund through the Welsh Government for funding the COATED project, which made this research possible. Finally we would like to acknowledge the assistance provided by Swansea University College of Engineering Advanced Imaging of Materials (AIM) Facility, which was funded in part by the EPSRC (EP/M028267/1), the European Regional Development Fund through the Welsh Government (80708) and the Ser Solar project *via* the Welsh Government, with particular thanks to Peter Davies and Richard Johnston at AIM.

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New electro-optic glasses containing metal nanoparticles for light modulators

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For the construction of fast electro-optic modulators functioning in VIS and NIR part of spectra, the most important component is an optical material with a sufficiently high electro-optic coefficient. As suitable and cost effective, were developed new glasses with Ag⁰ nanoparticles based on a PbO-Bi₂O₃-Ga₂O₃ system. The glasses were melted in PtRh crucibles in an electric furnace at 1000°C for 30 min, mixed twice after 15 min and cooled down in an annealing furnace at 350°C. For the formation of Ag₀ nanoparticles the Ag layers were coated on the glass surface with magnetron sputtering. The clustering of the electro-optically active Ag⁰ nanoparticles was achieved in the prepared glasses by interdiffusion, reduction, nucleation and growth during heat treatment. The optical properties were measured with M-line spectroscopy, UV-VIS and UV-VIS-NIR spectroscopy and the size of nanoparticles was measured with TEM and evaluated with image analysis. The electro-optic properties were measured in an optical system comprising a laser source, operated in continuous regime, a polariser, the glass sample, a quarter wave plate, an analyser, a diaphragm and a detector. On the glass sample, a high voltage from 5 to 30 kV was applied that influenced the refractive index and thus then transmittance of the system. From the angle of rotation of the analyser, the electro-optical coefficient was calculated. Because of the achieved electro-optic coefficient are able the electro-optic modulators to operate as light intensity modulators or phase modulators.

The effects of processing parameters on the properties of polypropylene-based thermoplastic dynamic vulcanizates

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Keywords: thermoplastic dynamic vulcanizates, TDV, elastomer, polypropylene

Elastomers can sustain 100% and more strain without failure and after the load stops, they reversibly regain their original form. This high flexibility can be attributed to their chemical or physical cross-linked structure. Because of this structure, elastomers have numerous positive properties, but it is difficult to recycle them. Elastomers cannot be recycled by remelting like thermoplastic polymers. But there are some solutions to this problem. For example, devulcanization, pyrolysis or the use of thermoplastic elastomers (TPEs) in some fields, where substitution is possible.

The first TPEs appeared in the middle of the 20th century. These materials belong to a class of material that have the combined physical properties of thermoplastics and elastomers. TPEs have a physically cross-linked structure. They show properties typical of rubbery materials but can be processed like thermoplastics.

Thermoplastic dynamic vulcanizates (TDVs) belong to a novel family of thermoplastic elastomers. They are blends composed of thermoplastic polymers and elastomers in which the fully or partially cured elastomer component is finely dispersed (mean particle size: 1-2 μm) in the thermoplastic matrix. The matrix is mostly polypropylene (PP) or high-density polyethylene (HDPE), whereas the elastomer component is mostly either saturated (EPM) or nonsaturated (EPDM). TDVs can be produced with high productivity because compounding and vulcanization take place at the same time and they are easily processed with internal mixers or by extrusion. Furthermore, TDVs give better material utilization because scrap and rejects can be recycled (for example ground tire rubber (GTR)). As the composition of blends can be varied, materials with a wide range of properties can be obtained.

In this study PP-based TDVs were produced with a co-rotating twin-screw extruder. Different processing parameters of TDVs were examined (temperature profile, different screw configurations, and different places of feeding). The elastomeric component was 70/30 styrene-butadiene rubber/natural rubber. Screw speed was 180 1/min. The specimens were produced by injection moulding. Tensile and Shore A hardness tests were performed on the specimens and curing curves were recorded, as well. Because of the high screw speed, the duration of stay was not enough to reach 90% of curing, therefore curing was finished during the injection moulding of the specimens. The SEM pictures showed that the particle size of the elastomeric component was 1-2 μm , thanks to the high screw speed. The results showed that matching feeding and screw configuration with the right extruder temperature profile can increase the mechanical properties of TDVs.

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Fabrication of graphite-reinforced Cu matrix composites

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Metal matrix composites (MMCs) consist of the heterogeneous combination of two or more constituents. [1] The metal forms the matrix within which the other constituents are dispersed as fillers. Although the individual constituents maintain their distinct physical and chemical natures, the dispersion of materials to various extents and at different length scales can result in enhanced properties and performances compared to those exhibited separately by the constituents [2, 3]. Therefore, MMCs represent an important case study for meeting the demand of replacing critical raw materials in future technology.

While showing promise for advanced technological applications in several fields, MMCs also raise fundamental challenges for the current understanding of structure-property relationships. In this respect, there is still considerable room for experimental and theoretical investigation [4].

Based on above-mentioned considerations, the present work addresses the fabrication of MMCs by mechanical processing. In particular, Cu-graphite powder mixtures have been subjected to mechanical processing by ball milling under inert atmosphere to induce mutual dispersion of the constituents [5]. Then, powder has been consolidated by spark plasma sintering, and the obtained pellets subjected to nanoindentation to investigate the variation of mechanical properties with milling time and graphite content.

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Improved photo-catalytic performance of CeO₂-TiO₂ nanocomposite against degradation of crystal violet dye

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The study discusses the potential of CeO₂-TiO₂ nanocomposite for the photo-catalytic degradation of crystal violet dye under ambient air conditions. The nanocomposite were synthesized using simple hydrothermal route without the assistance of any template molecule. The formed nanostructures were determined to possess spherical shape with rough surface morphology and average size in range of 50–120 nm. The as-synthesized nanocomposite were further investigated for their heterogeneous photo-catalytic potential against the oxidative degradation of crystal violet dye taken as model pollutant. The photo-catalytic performance of the as-synthesized material was evaluated both under ultra-violet and visible light where the best photo-catalytic performance was achieved under visible light with complete degradation (100%) achieved within 60 min of irradiation time. The kinetics of the photo-catalytic process were also considered and the reaction rate constant for CeO₂-TiO₂ nanocomposite were determined to be 0.0125 and 0.0662 min⁻¹ for ultra-violet and visible region respectively.

Microstructure and texture control of 316L stainless steel parts

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Keywords: Selective laser melting, keyhole, texture, microstructure, porosity, vapor plume

Selective Laser Melting (SLM) is a powder bed additive manufacturing process where parts are made layer-by-layer from a 3D file (STL). Powder layers are melted on top of each other with the laser, following a 2D pattern extracted from the discretization of a 3D volume. The complexity of a part is not a barrier in SLM, and thus the process opens new doors to design and elaborate complex and intricate shapes [1].

With the development of this innovative manufacturing process, new challenges have emerged, such as the microstructure and texture control of SLM parts, which need to be controlled precisely in order to obtain good, predictable and reproducible mechanical performances.

Power, scanning speed and hatch are usually the main variables used for parametric studies in SLM parts in the literature. However, some additional and often overlooked parameters, such as the laser scanning direction and strategy, can strongly influence the sustainability and reproducibility of the microstructure of parts fabricated with SLM [2]. This study focuses on identifying the impact of some secondary parameters on the melt pool morphology, and the resulting grain morphology and crystallography in 316L stainless steel SLM parts.

It has been observed that epitaxial grain growth can occur preferentially in the SLM process, and can lead to strongly textured materials with columnar grains aligned along the building direction [2]. By modulating the melt pool shape and overlap between subsequent melt pools, it is possible to design the material texture. A high overlap of the melt pools leads to strong epitaxial growth with highly textured parts. On the contrary, a low overlap results in smaller grains with a more random orientation, linked to a homogeneous grain nucleation following the thermal gradients. Additionally, the texture orientation itself can be modulated. By changing the laser scanning strategy, it is possible to select specific grain orientations that can grow epitaxially, according to the pattern in which the melt pools overlap between each layer.

Acknowledgments

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In-situ thermal cycling and fatigue of high temperature shape memory alloys

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Keywords: High temperature shape memory alloys, thermal cycling, corrosion, fatigue

Shape Memory Alloys (SMA) are known for two key properties; shape memory and pseudo-elasticity. Their elastic limit exceeds typical values and this extend depends on the alloy, e.g. 8% for the NiTi25Hf. Aerospace and automotive industries are among the potential users of high temperature NiTiHf-based shape memory alloys (HTSMAs). Large actuation capability, broad range of transformation temperatures and stable responses are some of the requirements in most of these applications. Understanding the failure mechanisms and fatigue of NiTiHf-based HTSMAs is a challenging, still unexplored task. This on-going work aims to provide the effect of thermal cycling on the fatigue behavior of HTSMAs. The samples were prepared by wire-EDM in a “C-ring” shape. A novel in-situ thermal-cycling and fatigue device, able to produce cyclic thermal and mechanical loadings was developed and employed in these investigations. A full fractographic analysis of the failed samples was facilitated by optical and electron microscopy. The first results show that due to the intrinsic recoverable deformability of the surface-originating martensitic transformation, a complicated failure mechanism is revealed.

Structural, morphological and optical properties of yttrium modified ZnO thin films

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Keywords: Y doped ZnO Thin films, Spray pyrolysis route, optical properties.

Pure ZnO and yttrium doped Zinc oxide ($\text{Zn}_{1-x}\text{Y}_x\text{O}$, $x = 0, 0.02, 0.05, 0.07$) thin film has been synthesized successfully via a spray pyrolysis processing on glass substrate at 450 °C from the source materials of zinc chloride and yttrium acetate. Doping concentration was modified from 0, 2, 5 and to 7% by adding yttrium acetate and the solution was sprayed for 6 min. The yttrium doping concentration impact on the structural, surface morphology, compositional and optical properties of $\text{Zn}_{1-x}\text{Y}_x\text{O}$ thin films was investigated in detail. The as-prepared films exhibit well-defined hexagonal würtzite structure and grew along [002]. X-ray diffraction technique was carried out to investigate the structure and crystalline nature of synthesized thin film. The crystallite size ranged in nanometer range and seems to be only lightly affected. The optical band gap, E_g , has been examined for the films. A shift from $E_g = 3.12$ eV to 3.21 eV has been noticed for synthesized films. Field emission scanning electron microscope micrographs showed that the prepared ZnO and $\text{Zn}_{1-x}\text{Y}_x\text{O}$ films acquired a dominance of hexagonal-like grains with some extra minority of rode-like and platelets, the morphology was influenced by Y incorporation. The optical features of undoped and $\text{Zn}_{1-x}\text{Y}_x\text{O}$ films were studied by UV-Visible measurements. The average transmittance values of $\text{Zn}_{1-x}\text{Y}_x\text{O}$ films were more than 82% in the visible domain.

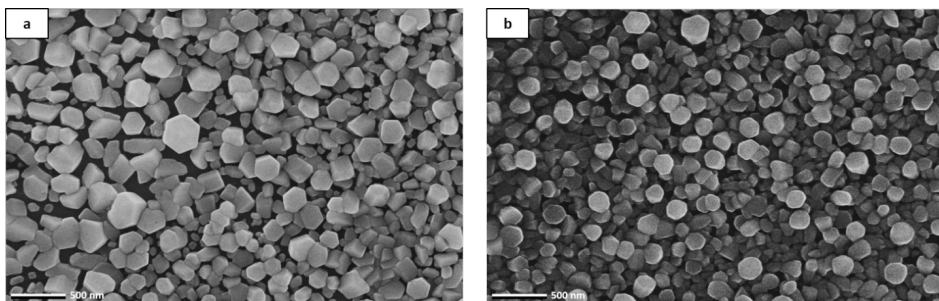


Figure 1 SEM images of ZnO:Y thin films with various Y doping: (ZnO:Y 2%, b ZnO:Y 5%)

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Epitaxial growth of GaSb based devices onto Silicon wafers

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Keywords: Mid-infrared, GaSb, Molecular beam epitaxy, Si wafer, APDs

III-V semiconductor materials are a family of materials developed from the group III and V of periodic table. The binary semiconductors such as InAs, AlSb, GaSb and their respective alloys cover a broad range of band alignments and energy gaps, that can be used to produce a wide range of high performance optoelectronic devices operating in the technologically important mid-infrared (MIR) spectral range. As a result, such materials are the basis of almost all the thin film light structures, such as solid-state lasers, light emitting diodes, etc. These structures are usually grown on wafers such as GaSb and InAs, which have high cost, a non-optimized surface oxide layer, poor thermal conductivity and are only available in small sizes. The integration of III-V semiconductors on Si wafers would be most desirable to enable cost effective manufacture and open new applications in lab-on chip MIR photonic integrated circuits¹. However, their direct epitaxial growth onto Si wafers is a difficult task due to the large differences between the lattice constants (13%) and the thermal expansion coefficients which can lead to the formation of threading dislocations and cracks. Furthermore, the polar/nonpolar character of the III-V/Si interface, due to the single atomic surface steps, may result to the formation of planar defects known as antiphase boundaries domains (APDs)² which should be avoided.

In this work, we report on the molecular beam epitaxial (MBE) growth of high quality APD-free GaSb onto Si using a novel buffer layer design and the resultant performance of Sb-based multilayer structures. The samples were grown in a VeecoGENxplor MBE system on (001) silicon substrates with a 4° miscut in order to eliminate the formation of APDs. A thin ~ 5nm AlSb nucleation layer was grown prior to the GaSb, to relieve the large lattice mismatch via a periodic, self-ordered network of 90° edge dislocations which predominately propagate laterally rather than vertically into the overlying epilayer³. This is followed by a low growth temperature GaSb buffer layer which confines the dislocations within the first 700 nm.

Figure 1 shows the high resolution X-Ray diffraction (XRD) pattern measured from the GaSb buffer samples. The FWHM of the GaSb peak is 200 arcsec and is fully relaxed, indicating the high crystalline quality. Figure 2 shows the periodic interfacial misfit arrays formed at the III-V/Si interface and confirms the formation of the 3D AlSb islands with an average height of 8.5 nm. An InAs_{0.88}Sb_{0.12} p-i-n light emitting diode (LED) was grown on top of the GaSb buffer and the wafers were processed into mesa etched devices with top-top metallic contacts, using standard photolithographic techniques, as shown in Figure 3 (a).

The temperature dependent electroluminescence emission spectra measured from one of the InAsSb LEDs is shown in Figure 3 (b), where a bright peak can be observed up to room temperature peaking at 4.5 μm .

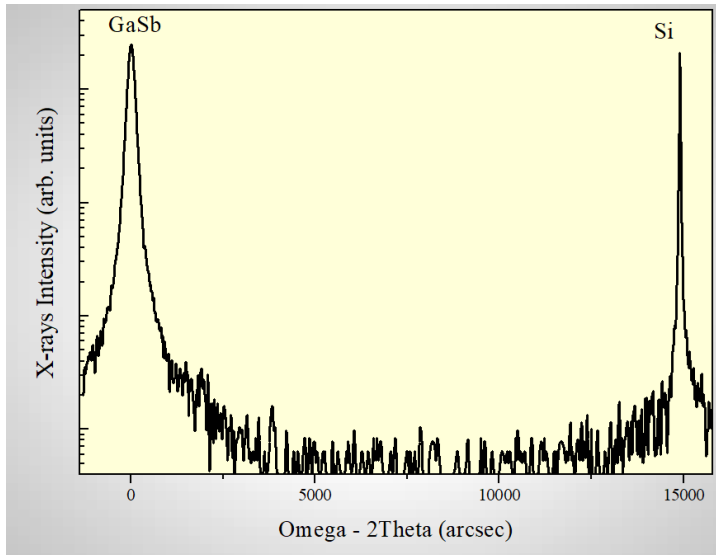


Figure1. X-rays rocking curve of the GaSb/Si buffer layer

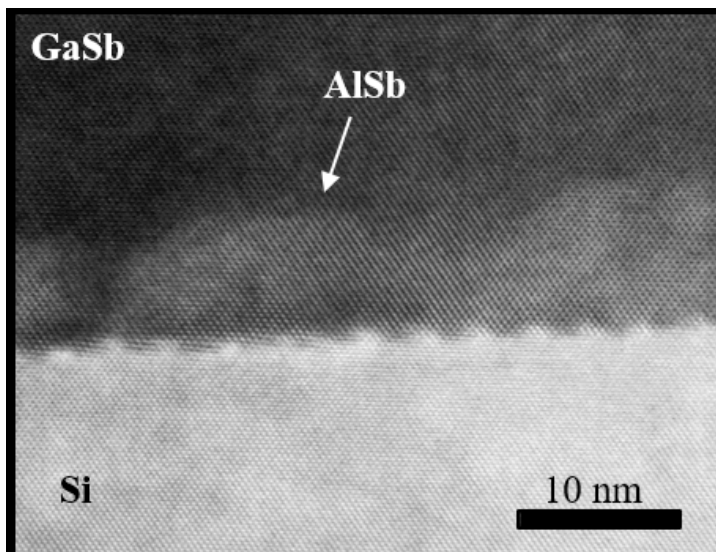
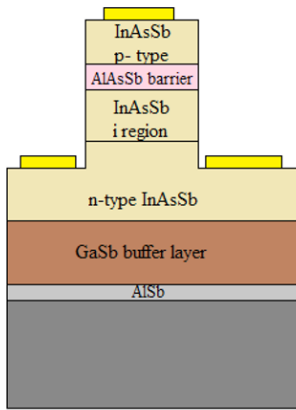
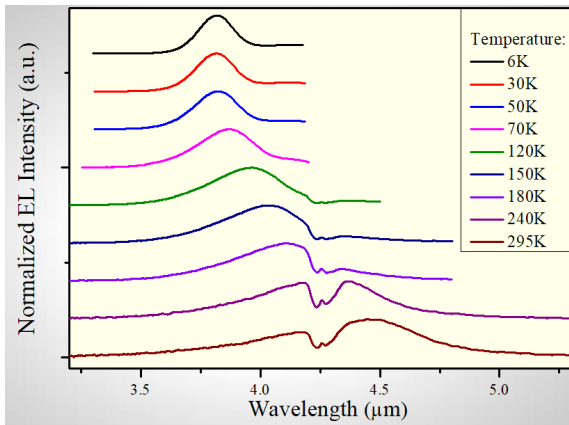


Figure 2. Cross section high resolution TEM image of the III-V/Si interface. AlSb islands can be clearly seen.



(a)



(b)

Figure 3. (a) Schematic of InAsSb light emitting diode grown on Si with the top-top contacts configuration, (b) The electroluminescence emission spectra of an InAsSb p-i-n measured at various temperatures.

Acknowledgments

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Grain boundary engineering parameters for ultrafine grained microstructures: Proof of principles by a systematic composition variation in the Cu-Ni system

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Keywords: Grain boundary engineering, EBSD, high-pressure torsion, stacking fault energy, severe plastic deformation

In the talk, the main principles of grain boundary engineering for ultrafine grained materials are examined via a systematic variation of the stacking fault energy, solid solution effects and the homologous deformation/annealing temperatures choosing Cu-Ni as a model case. Cu and Ni are completely miscible and the stacking fault energy varies strongly with the alloy composition. Ultrafine grained microstructures are produced by high-pressure torsion and their evolution upon annealing is investigated. The thermal stability and the saturation grain sizes after severe plastic deformation are controlled by solid solution effects. The fraction of deformation twins varies in accordance with the stacking fault energy. The fraction of $\Sigma 3$ grain boundaries after annealing is related to the grain size and influenced by solid solution effects, whereas the fraction of $\Sigma 9$ grain boundaries is found to depend on the stacking fault energy. The results indicate that grain boundary engineering results in an optimum structure, i.e. a high length fraction of $\Sigma 3$ and $\Sigma 9$ grain boundaries for grain sizes below 1000 nm, in alloys close to the equiatomic composition, showing a comparably low stacking fault energy and a high melting temperature. This high fraction of $\Sigma 3n$ grain boundaries (including their conjunctions) proved itself most effective for microstructure stabilization in virtually non-segregating systems. Thus, for Cu₅₀Ni₅₀ and Cu₆₅Ni₃₅, a narrow grain size distribution of small grain size, a high hardness and a high fraction of special grain boundaries could be adjusted.

Structural and inhibitor characteristic of $\text{Bi}_2\text{O}_3\text{-P}_2\text{O}_5\text{-Nb}_2\text{O}_5$ phosphates glasses

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Glasses based on P_2O_5 have several advantages over conventional silicate and borate glasses due to their several unique physical properties, such as lower glass transition and melting temperatures, higher thermal expansion coefficient, high UV transmission and electrical conductivity [1–5]. However, phosphate glasses typically have a relatively poor chemical durability, which often limits their applications. By addition of heavy metal oxides like Bismuth oxide Bi_2O_3 or Nb_2O_5 to the P_2O_5 network, the physical properties could be improved. These oxides lead to the formation of Bi-O-P and Nb-O-P bonds which replace the easily hydrolysable P-O-P bonds and improve dramatically the chemical durability of the modified phosphate glasses [6-7].

Inhibition of mild steel corrosion in 1M HCl solution by phosphate glasses having composition $10\text{Bi}_2\text{O}_3\text{-(90-x)P}_2\text{O}_5\text{-xNb}_2\text{O}_5$ with $x = 2,5\text{--}15$ mol%, has been investigated using electrochemical polarization and scanning electron microscopy techniques. The structural characteristics were investigated by X-ray diffraction, thermal analysis, and Raman studies. Various corrosion parameters such as Tafel slopes, Nyquist plots, corrosion current density, and charge transfer resistance has been calculated to understand the inhibition mechanism. The results showed that these glasses act as a corrosion inhibitor in acidic medium and their inhibition efficiencies depend to the Nb_2O_5 content in the glass. In addition it is observed that the metaphosphate chains provided a better protection pyrophosphate $\text{P}_2\text{O}_7^{4-}$ and orthophosphate $\text{P}_2\text{O}_4^{3-}$.

IR and Raman spectroscopy's indicate that when Nb_2O_5 is added to phosphate glasses, the structure chains is broken into pyrophosphate groups $(\text{P}_2\text{O}_7)^{4-}$ and the P-O-P bonds are depolymerized by the incorporation of distorted Nb(6) units through P-O- Nb bonds. These results agree with a closer structure and act in a manner that Nb_2O_5 enter the glassy matrix as a network former character. It is assumed that niobium to be present as corner-sharing $[\text{NbO}_{6/2}]^-$ octahedral units.

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On the role of alloying element interactions in the modification level assessment of foundry aluminium alloys via thermal analysis approach

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Keywords: Sr modification, thermal analysis, elements interaction

The morphology of the eutectic Si particles has a pivotal role in improving the mechanical properties of aluminium alloy cast components, particularly ductility. In this regard, significant enhancements can be achieved by adding certain modifying agents, which promote the transformation from a coarse and plate-like morphology into a fine and fibrous one. In particular, Sr has become the most widely used alloying element thanks to its good modification rate, long fading time, high recovery efficiency and ease of handling [1]. Thermal analysis is an effective control method employed in the foundry practice to evaluate the quality of the modification treatment prior to casting. Despite that, few studies have examined the role of alloying elements on the depression of the eutectic growth temperature, evaluated from the cooling curves [2, 3].

In the light of this, the present work aims to contribute the understanding of the effects of Mg and Ti on thermal parameters and microstructural evolution of Sr-treated Al-7Si alloys. Alloys with increasing Sr amounts (from 40 ppm to 140 ppm) have been considered to thoroughly investigate the role of element interactions on the modification efficiency. The analysis of the cooling curves suggests that Mg addition provides a remarkable decrease of the eutectic thermal parameters with respect to the Al-7Si alloy and Ti addition also seems to have a slightly similar effect on the eutectic characteristic temperatures. A quantitative microstructural evaluation of the Si particles enables to point out the poisoning effect of Mg on Sr, as shown by the loss of homogeneity of Si morphology. This combined approach suggests that, in the modification level evaluation, the effects of alloying elements on both cooling curves and microstructural features cannot be neglected.

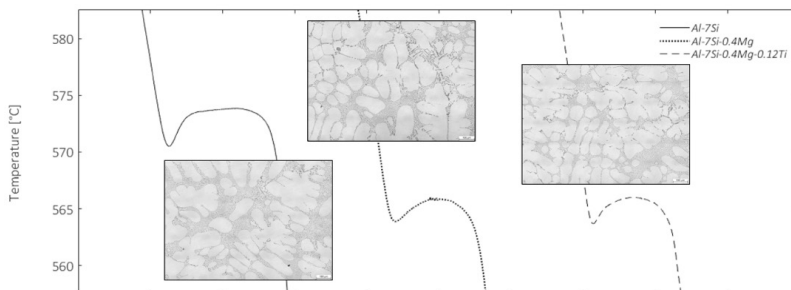


Figure 1. Comparison among Al-7Si, Al-7Si-0.4Mg and Al-7Si-0.4Mg-0.12Ti alloys, modified with 100 ppm of Sr: eutectic region of the cooling curve and related optical micrograph.

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Aspects of Selective Laser Melting technology considered in preparation of trabecular structures

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Keywords: 3D printing, SLM, titanium alloy, porous structures, bone implants

In modern regenerative medicine, biomedical engineers try to mimic natural structures to get closer to physiological properties of bones. When a missing bone is about to be replaced, porous structures are desired to stimulate a regeneration process of the adjacent bone tissue through an osteoconductive and osteoinductive activity. Pores should be well admissible to bone cells; open, interconnected and of an appropriate size. It is also important for the implant to match mechanical properties of bones. Otherwise, stress-shielding effect caused by the difference in bone and material stiffness can yield in thinning of adjacent bone, implant loosening and even need of reoperation. By introducing an appropriate level of porosity into bulk materials, their stiffness can approach that of bones.

However, preparation of such complex structures is not easy to control using traditional technologies such as replication, use of space fillers, CVD etc. In this field, the additive manufacturing (AM) technology has been a breakthrough as it enables to create net-shape objects while allowing to control their internal structure in each part of their volume. Even individual implants based on the results of computer tomography or nuclear magnetic resonance of a particular patient are possible.

In this conference contribution, we show possibilities of selective laser melting (SLM), one of the AM technologies, in the preparation of trabecular structures. SLM is based on localised melting and consolidation of powder material in a layerwise fashion by the use of a computer-controlled laser beam. Despite great advantages offered by SLM, there are specific process-inherent aspects that must be considered before the production of porous structures. First, we tested different orientations and thicknesses of struts to verify SLM capabilities. Using these findings, porous structures were designed and produced. The mechanical properties were measured and discussed with properties of bones.

Acknowledgments

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The development of plate and lath morphology in Ni_3Ge_3

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Keywords: rapid solidification, intermetallic compound, plate & lath microstructure, isolated hexagonal crystallites

The congruently melting, single phase intermetallic Ni_3Ge_3 has been subject to rapid solidification via drop-tube processing wherein powders, with diameters between 850–53 μm , are produced. At low cooling rates (850–150 μm diameter particles, 700–7700 K s^{-1}) the dominant solidification morphology, revealed after etching, is that of isolated plate and lath microstructure (Figure 1a) in an otherwise featureless matrix. At higher cooling rates (150 – 53 μm diameter particles, 7700–42000 K s^{-1}) the dominant solidification morphology is that of isolated hexagonal crystallites (Figure 1b), again imbedded within a featureless matrix. Selected area diffraction analysis in the TEM reveals the isolated hexagonal crystallites, are a disordered variant, whilst plate and lath microstructure are the partially ordered variant of $\epsilon\text{-Ni}_3\text{Ge}_3$. However, the featureless matrix of both microstructures are the fully ordered variant of the same compound. Microvicker hardness test results shows that isolated hexagonal crystallites has higher mechanical properties with respect to plate and lath structure.

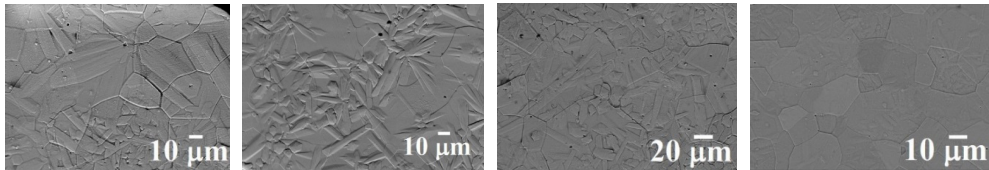


Figure 1 (a) : SEM micrograph of HF etched Ni_3Ge_3 drop-tube particle from the 850–150 μm size fraction showing numerous plate and lath structures in a more-or-less featureless matrix.

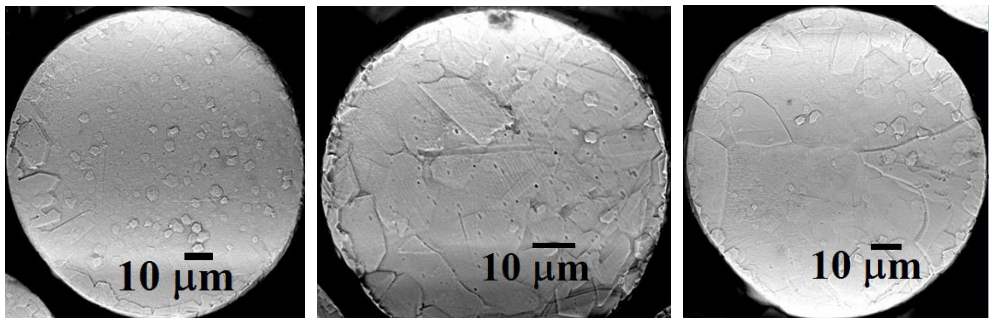


Figure 1 (b) : SEM micrograph of HF etched Ni_3Ge_3 drop-tube particle from the 150–53 μm size fraction showing numerous isolated hexagonal crystallites in a more-or-less featureless matrix.

A Method for the production of titanium-tantalum alloys via the electrolytic reduction of their respective oxides using the metalysis-FFC process

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Keywords: Tantalum, titanium alloys, powder metallurgy, FFC

Tantalum-Titanium alloys possess interesting properties such as good strength, good ductility, chemically inert and high temperature resistance; this makes them desirable for various high temperature applications (e.g. fusion reactors, solar concentrator towers, heat shields). Tantalum-Titanium alloys have historically been difficult to produce due to the large difference in melting point and poor interdiffusivity rates of the two elements; leading to inhomogeneity. However, using the Metalysis-FFC process we have been able to produce a range of homogeneous Ti-Ta alloys. The Metalysis-FFC process is an electrochemical process in which metal oxides can be reduced by acting as a cathode in an electrolytic cell with a graphite anode and molten calcium chloride electrolyte. We have proven that the process works when applied to mixed oxides to produce alloys. The reduction process is performed in the solid state and so negates the problems associated with traditional melting practices.

Preparation of TiAl15Si15 intermetallic alloy by mechanical alloying and spark plasma sintering

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Keywords: mechanical alloying, Spark Plasma Sintering, intermetallics

This work is devoted to the preparation of alloys based on intermetallic compounds in Ti-Al-Si system by powder metallurgy using mechanical alloying (MA) and Spark Plasma Sintering (SPS) method. The aim of this paper was to describe the formation of intermetallic phases during mechanical alloying of TiAl15Si15 (wt. %) and to consolidate the powder prepared by optimized conditions. Phase composition, microstructure and hardness of compacted alloy was determined. Four hours of mechanical alloying is sufficient time for preparation of the material composed only of intermetallic phases without pure elements. After consolidation, TiAl15Si15 alloy has homogeneous structure comprised of Ti₅Si₃ silicides in aluminide TiAl matrix. Hardness of material is very high.

Acknowledgments

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Structure and texture of magnesium plates and foils obtained by low-temperature severe plastic deformation

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Keywords: magnesium, severe plastic deformation, microstructure, mechanical properties

Magnesium and its alloys have the excellent specific properties. However, magnesium as a metal with hexagonal close packed crystal structure has a limited number of slip systems, and because of that, its ductility and formability are poor at room temperature. The using of severe plastic deformation (SPD) leads to significant structure refinement and basal texture weakening for improving mechanical properties.

The aim of the work was to study the texture, structure and mechanical properties of pure magnesium after SPD by lateral extrusion and following rolling at different temperatures.

Three cylindrical samples of 30 mm in diameter and 50 mm in length were deformed by the lateral extrusion method (LE) at room temperature. Samples had different grain orientations in relation to the load. Deformation degree was $\varepsilon \sim 3.9$. As result, 1-mm magnesium plates were obtained. The plates showed no visible traces of cracks, except a little crackling along the edges. After deformation by LE the basal texture (0001) was formed for all samples. The sharpness degree of the basal texture is determined by the initial sample orientations. It was found the significant structure refinement from 7 mm to 3 μm . Low fraction of twins were revealed in plates by EBSD-analysis.

The plates were rolled to foils of different thickness (150 and 10 μm) at room and cryogenic temperatures. Deformation degree was $\varepsilon \sim 6$ (for 150- μm foil) and $\varepsilon \sim 7.5$ (for 10- μm foil). Further grain size reduction were failed to achieve during plates rolling. The average grain size was 5 μm for both 150- μm and 10- μm foils.

Mechanisms responsible for the intense refinement of the initial structure are processes of slip and dynamic recrystallization.

The highest values of yield stress ($\sigma_{0.2}$) and tensile strength (σ_B) for 1-mm plates were 132 MPa and 161 MPa, respectively. Mechanical properties of the obtained plates depend on the orientation of the initial columnar structure and correlate well with their varying degrees of texturizing. The best results of elongation-to-failure were achieved to 16% for plates and 20.5% for foil.

Based these results, we attempt now to obtain thin plates from AZ31 magnesium alloy using by different methods of low-temperature SPD.

The work results could be useful for application of magnesium to create membranes for biology and medicine, magnesium batteries for energy and biocompatible sensors for transient electronics.

Acknowledgments

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Impact of microstructure and geometric length scales on miniaturized tensile tests of advanced steels

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Keywords: microstructure, EBSD, steel, small scale, tensile test

In this work the mechanical properties of advanced steels are characterized by a miniaturized tensile test and compared to the results of other mechanical testing methods. Eleven steel grades were provided by *thyssenkrupp* and miniaturized specimens were cut with a dog-bone shape contour. The specimen dimensions have a constant gauge length of 4mm, the gauge width is varying from 0.16 mm to 0.50 mm and gauge thicknesses between 1.45 mm and 0.18 mm were used. For one type of steel the dimensions were severely changed to verify an occurring size effect. The microstructure of the materials was quantitatively investigated by EBSD.

The tensile test results were correlated to Vickers hardness measurements, average grain size and *thyssenkrupp* database values for the ultimate tensile strength. Some steels reproduce the macro-scale results well in miniaturized testing whereas others show a significant drop in the performance. The overall performance of the miniaturized tensile tests were evaluated by the ultimate tensile strength and the fracture strain for one type of steel by varying the geometrical dimensions. The results indicate the importance of the standard deviation of the grain size distribution for a more independent evaluation of the size effect.

Contribution of the comprehension of nanostructured $(\text{Fe}_{65}\text{Co}_{35})_{100-x}\text{Cr}_x$ ($x = 0,10$) some structural and magnetic properties

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Keywords: mechanical synthesis, ternary systems, VSM, hysteresis loops.

In this paper, structure and magnetic properties of nanocrystalline $(\text{Fe}_{65}\text{Co}_{35})_{100-x}\text{Cr}_x$ ($x = 0, 10$) powders produced by mechanical alloying have been investigated. Some parameter such as milling times (up to 36 h) and compositional ration (0 and 10% Cr) was investigated in this study. For this purpose Fe, Co and Cr of 99.9, 99.8 and 99.5% purity, respectively. Characterizations of the prepared powders were carried out by the X-ray diffraction (XRD), scanning electron microscopy (SEM) techniques and vibrating sample magnetometer (VSM). The results show the formation of the $(\text{Fe}_{65}\text{Co}_{35})$ and $(\text{Fe}_{65}\text{Co}_{35})_{90}\text{Cr}_{10}$ solid solutions completed in 12 h by milling time. High values of induction (B_r) and magnetic field (H_c) confirm that we got a hard magnetic system. The presence of chromium (Cr) in ternary $(\text{Fe}_{65}\text{Co}_{35})_{90}\text{Cr}_{10}$ amplifies the value of the magnetic field (H_c). The value of the induction (B_r) ternary alloys $(\text{Fe}_{65}\text{Co}_{35})_{90}\text{Cr}_{10}$ stabilizes around 12h of milling time.

Anisotropic influence of hydrides on fatigue properties of zirconium based nuclear fuel claddings

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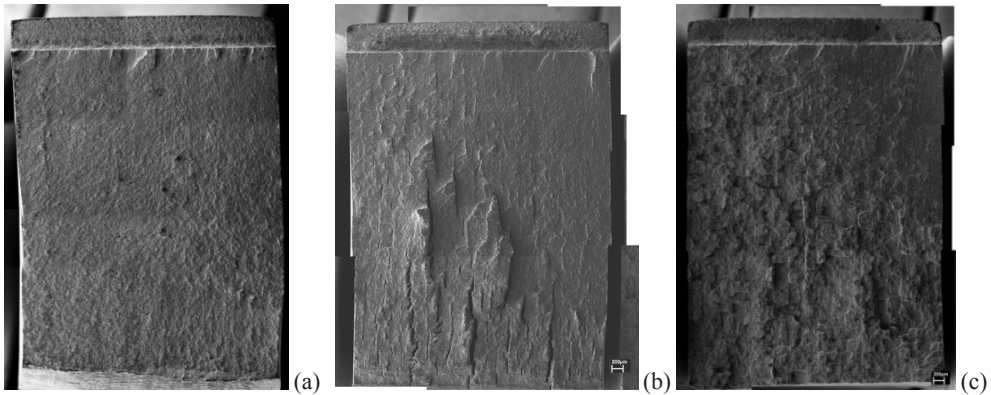
Keywords: zirconium, hydrides, orientation, fatigue

Zirconium alloys exhibit a highly corrosion resistant and protective oxide layer and are therefore often used in the chemical process industry. Due to their low absorption cross-section for thermal neutrons and reasonable mechanical properties they are also the material of choice for nuclear fuel cladding tubes for light water reactors. During the operation in the reactor, the zirconium alloy claddings take up a part of the hydrogen produced by the slow oxidation reaction between the reactor coolant water and the hot rod surface. With increase of operation time, hydrogen uptake may become a concern because of the precipitation of hydrogen-metal compounds, so-called hydrides, which are brittle platelets of few μm thickness and tens to few hundreds μm of lateral expansion. While the zirconium alloy has a hexagonal crystallographic lattice, the typical δ -hydrides are of face centered cubic type. When the spent fuel

is discharged from reactors, there is a potential loss of resilience of the cladding even under usual loading due to an unfavorable orientation of the hydrides platelets. Hydrides, especially those oriented in through wall direction, could raise the risk of respective crack incubation and propagation, thus reducing fatigue strength under vibration conditions like spent fuel transportation, eventually accelerating fatigue failure.

The objective of this work is to investigate the potential anisotropy of fatigue properties induced by different hydrides platelets orientations with respect to the zirconium alloy matrix in rolled flat samples. Fatigue tests of hydrogen-free samples in the as-received condition were performed at room temperature, where the S-N curve (applied stress amplitude versus number of cycles until failure) was fitted as a reference. Further, in-plane hydrides (parallel to loading direction) showed little effect on the fatigue properties although the SEM fractography presented a distinguished feature with split hydrides. Some fatigue degradation by hydrides perpendicular to loading direction was observed. Hydrides metallography and SEM fractography – considering the respective thermo-mechanical history – are employed in order to interpret the interaction between hydrides orientation, loading direction and crack propagation, but also texture / rolling direction effects.

Figures



SEM fractography of zirconium alloy fatigue samples, (a) hydrogen-free, (b) with in-plane hydrides (perpendicular to crack surface) and (c) reoriented hydrides (parallel to crack surface).

The effect of oxidized nanosized silicon nitride powder particles on the structural and mechanical properties of $\text{Si}_3\text{N}_4/\text{CNTs}$ composite by hot isostatic pressing

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Keywords: Hot isostatic pressing, Si_3N_4 , $\text{Si}_2\text{N}_2\text{O}$, CNTs, oxidation

The effect of oxidized Si_3N_4 nanosize base particles on the structural and mechanical properties of $\text{Si}_3\text{N}_4/3$ wt. % MWCNTs composites sintered by hot isostatic pressing (HIP) was studied. The $\alpha\text{-Si}_3\text{N}_4$ powders were oxidized for 10 and 20h at 1000°C in the ambient air. The oxidation of powder particles was confirmed by the different structural analysis. The amorphous oxide layer on the powder particles were found and thickness of amorphous phase was increased with the increase of oxidation time. An enough ZrO_2 was found in the powders after milling which originated from the grinding media. The effect of ZrO_2 in the starting powders on the properties of final composites was investigated as well. The 3 wt. % MWCNTs were added in the oxidized silicon nitride powders and mixed it with the attrition milling for 30 minutes at 600 rpm. The composites were sintered under 20 MPa for 3 hours. The milled and sintered composites were characterized by the XRD, SEM, EDX, HRTEM and TEM. 3-point and 4-point bending strength, Young's Modulus, Vicker's hardness and toughness of sintered samples were measured. The complete α to β Si_3N_4 transformation and presence of $\text{Si}_2\text{N}_2\text{O}$ phase were observed in all sintered samples. The oxide layer on the Si_3N_4 surface and its amount increased with the oxidation time. Higher amount of oxygen in the base powders results in the higher tendency of formation of $\text{Si}_2\text{N}_2\text{O}$ in the sintered Si_3N_4 .

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The development of titanium alloy powders produced via the Metalysis process

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The Metalysis process has previously demonstrated its capability to produce a wide range of metal powders from across the periodic table directly from their metal oxides via an electrochemical route. The attraction of this approach is that the steps associated with conventional metal powder synthesis are circumvented, resulting in a significant cost reduction. Work has been on going in the development of a synthetic rutile (beach sand) derived alloy. This alloy possesses comparable mechanical properties to Ti-6Al-4V. Further to this, work is ongoing using this synthetic rutile foundation to develop novel synthetic rutile derived alpha/beta-titanium alloys. The ensuing products have presented themselves well for application in developing powder consolidation markets, such as additive layer manufacturing and the Field Assisted Sintering Technology process.

Remarkable plasticity in iron-based metallic glass via finely-dispersed nanocrystals

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Despite ultra-high strength, elastic energy absorption capacity, the negligible plasticity in most metallic glasses has been an important barrier obstructing property optimization and their widespread use [1]. Yet, the novel bulk metallic glass (BMG) forming systems developed in the last two decades have broken this taboo by often showing extreme compressive plasticity (>15%) along with considerably high yield strength [2]. The large plasticity observed in the newly developed monolithic bulk metallic glasses under quasi-static deformation raises a question about the contribution of atomic scale effects. The main goal of this study is to resolve the origin of large plasticity, which is the multi-scale heterogeneities using a combination of an aberration-corrected high-resolution transmission electron microscopy (HRTEM) and nanoindentation techniques [3]. Since Fe and Ni atoms have a very small heat of mixing and similar atomic radii, the glass forming ability of this system decreases as the Ni content increases, which gives rise to nanocrystal formation. Pronounced variations in hardness and Young's modulus revealed by the nanoindentation tests and preferential thinning confirm the existence of hard and soft phases for this BMG. Furthermore, we conducted

systematic simulations of the HRTEM images at varying sample thicknesses, and theoretical model for the size estimation of the shear transformation zone. The findings suggest that the main mechanism behind the formation of softer regions are the homogenously dispersed nanocrystals, which are responsible for the hindrance and initiation of the shear transformation zones and hence, play a key role in the enhancement of mechanical properties. The controlled nanocrystal-induced plasticity with crystal sizes of a several nanometers can set a new precedent to understand the deformation mechanism in other BMG classes exhibiting extensive plasticity.

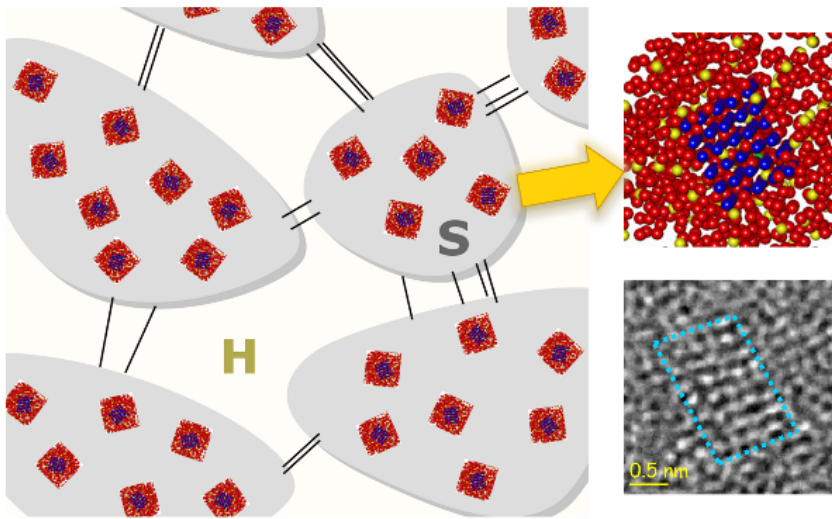


Figure 1. Multiscale deformation behavior in $\text{Fe}_{50}\text{Ni}_{30}\text{P}_{13}\text{C}_7$ BMG

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3-point bending fatigue testing of automotive bar steel grades at 20 kHz

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Keywords: VHCF, 3-point bending

The 20 kHz load frequency enables fatigue tests for very high cycle fatigue life, 10^9 - 10^{13} cycles, within conveniently short time, or significantly reduced test time and costs at shorter life. In automotive applications, many components are subjected to flexural loading and hence bending fatigue is an important test mode. Components exposed to surface stress gradients are also often conveniently bestowed enhanced performance by surface strengthening using mechanical, thermal or thermochemical processes, and carburizing is one such method often used.

Ultrasound fatigue test instruments have been used successfully in several assessments of fatigue strength and more commonly in uniaxial loading. Here, a 3-point bending fatigue test rig operating at 20 kHz load frequency has been constructed to test bar specimens with rectangular cross-section at $R = 0.1$ loading. In this project, the common ultrasonic test equipment including oscillator, booster and magnifying horn is completed with a specially designed mounting rig holding specimen in a fixed position. The design was achieved using FEM analysis to obtain a load train in resonance at 20 kHz and simultaneously reaching the desired stresses, Figure 1. The functions of the newly added mounting rig is to withstand the vertical force and to fix the plane position of the specimen and stop any horizontal movement caused by the ultrahigh frequency vibrations. Thus, a self-aligning lower fixture was developed.

Three different common automotive steels, 38MnSiV5, 50CrV4 and 16MnCr5, were tested. The 38MnSiV5-steel is a micro-alloyed ferritic-perlitic steel, the 50CrV4-steel is a quenched and tempered martensitic steel and the 16MnCr5-steel is a carburizing martensitic steel, with tensile strengths of 867, 1409 and 1175 MPa, respectively (regarding the carburizing steel the given tensile strength is of the core).

The new testing rig worked successfully, at least for two of the steel-grades. Unexpectedly, the 50CrV4 grade interacted with the supporting rods causing significant wear and test failure. The very high loading frequency allows us to achieve fatigue data to 10^9 cycles within 14 hours.

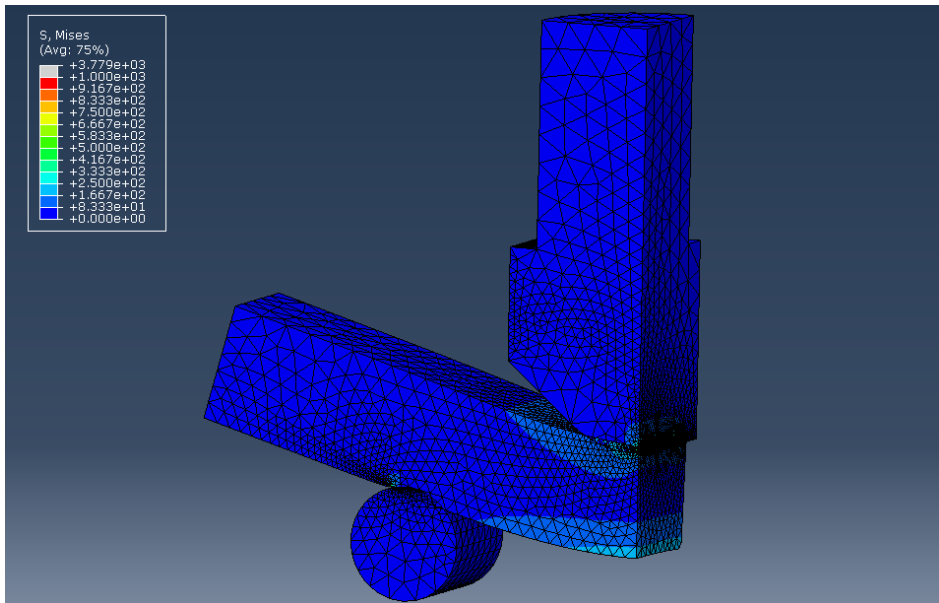


Figure 1. 3D stress plot of 1/4-symmetry three-point bending specimen with supporter and load-transferring tip.

Simulation of interactions between screw dislocations and radiation-induced defects in alpha-Iron using dislocation dynamics

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Keywords: molecular dynamics, dislocation dynamics, crystal plasticity, radiation-induced defects

Low-alloy ferritic steel (16MND5) is used for pressure vessels in French nuclear reactors, where radiation-induced defects are continuously nucleated during their operational lifetime. More specifically, such defects have a major influence on the mobility of dislocations, leading to a significant hardening and embrittlement. A multiscale modeling approach based on a combination of molecular dynamics and dislocation dynamics simulations is therefore adopted.

Interactions of edge dislocations with radiation-induced defects have been widely studied compared to those of screw dislocations. The later are nevertheless of great interest as their specific mobility has a significant impact on the mechanical properties of steels at low and

moderate temperatures. Furthermore, prior molecular dynamics (MD) studies have shown that their interactions with irradiation loops and precipitates lead to significant hardening.

In this study, a systematic investigation is made of the interactions between screw dislocations with $\langle 111 \rangle$ loops and coherent precipitates using a three-dimensional nodal dislocation dynamics (DD) code NUMODIS. The same configurations that were previously simulated with MD are successfully reproduced. This allows for a direct comparison between atomic scale and DD simulations to validate our multiscale strategy. Ultimately, the modeling of the collective behavior of dislocations properties controlling irradiation strengthening will be undertaken in order to identify and incorporate the relevant parameters in a crystal plasticity simulation.

Acknowledgments

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Formation of phases in Ti-20 wt. % Al mixture during reactive sintering

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Keywords: titanium aluminide, reactive sintering, mechanism

Titanium aluminides are promising materials for high-temperature applications, such as engine parts of airplanes or turbines. They are usually produced by melt metallurgy, which is quite inconvenient. This alloy has also high melting point, poor castability and moreover, melt reacts with crucible during melting. Optimization of properties and high cost of processing have been the barrier for the advance of practical applications. Despite of these disadvantages, casting technology is the most common processing route of aluminides. A number of methods have been also used in the synthesis and processing of TiAl intermetallic compounds, such as vacuum arc remelting (VAR), vacuum induction melting (VIM) and hot working techniques. However, powder metallurgy including reactive sintering was found to be a possible substitute for melt metallurgy. The greatest advantage is the possibility to control the phase composition. To optimize and control the process efficiently, mechanism and kinetics of the formation of intermediary phases have to be determined.

In this work, the mechanism of phases formation was studied by in-situ XRD and micro-structural analysis of the mixtures of Ti and Al elemental powders after heating in induction furnace and reaction was observed by optical pyrometer. Ti-20Al (in wt. %) alloy was studied in the range of temperatures from room temperature to 900 °C. Further, special model was used to determine, how the reactions are controlled. Phases formation was also studied at temperatures below the melting point of aluminium.

Acknowledgments

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PLC effect in austenitic TWIP steels

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Keywords: TWIP steels, thermomechanical treatment, martensitic transformations, PLC effect

Austenitic FeMnCr steels have high strength, high toughness and formability because of the martensitic transformation. In these steels the temperature as well as strain-induced non thermoelastic martensitic phase transformation takes place. This is the so-called TRIP (transformation induced plasticity) and TWIP (Twinning Induced Plasticity) effect. TWIP steel can deform by both glide of individual dislocations and mechanical twinning.

The PLC or Portevin Le Chatelier effect was seen in the tensile curves investigations during the thermo-mechanical treatments of the high strength austenitic TWIP steels. Many serrations of different intensities and amplitudes were observed in the tensile curves of the steel test samples and earlier it was believed to be the result of some mechanical parameters during the loading of the samples in the universal testing machine.

In our research work the TWIP steel samples used have different composition and it was interesting to figure out different behaviour of the PLC serrations in the tensile curves of the test samples. The true stress–true strain were characterised in more detail and various iterations of the test samples were used on the basis of temperature, composition and the deformation rate.

Influence of slm process parameters and heat treatments on the microstructure of inconel 625 superalloy

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Keywords: Selective Laser Melting, IN625, Energy Density, Microstructure, Heat treatments, Recrystallization, EBSD Analysis

Selective Laser Melting (SLM) is an additive manufacturing technology which allows to create mechanical parts by superimposing layers of selectively-fused metal powder one over the other.

The study is carried out in the frame of the FAIR French national project, leaded by Air Liquide Company. The objective is to manufacture and certify the first world exchanger-reactor created by SLM, and envisaged for different applications such as hydrogen mobility (production of hydrogen), animal feeding (production of molecules for food supplements) or heat generation.

In this work, a number of trials were carried out on simple geometries (with or without representative channels) to optimize the SLM process. The influence of the main manufacturing parameters (laser power, scan speed, focus spot, layer thickness ...), was studied with the first objective to generate samples combining a minimum of porosity with a minimal roughness, while respecting a good geometrical accuracy and a high productivity rate. Building strategies were also considered, such as the number of borders or steps order.

In a second step, a range of heat treatments was performed to evaluate their effects on microstructures and mechanical behavior of SLM samples. The goal was to check possible microstructural modifications through heat treatments and to understand the underlying origin of recrystallization in relation with the high solidification speed in SLM. Various samples were built to determine the effects of building parameters (at high or low Energy Density) and heat treatments conditions on recrystallization through EBSD analysis. Moreover, the precipitation in SLM samples were compared to materials from conventional processes.

Acknowledgments

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Softening of complex crystals by elastic heterogeneity

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Materials with complex crystal structures have many attractive properties for high temperature structural applications, including lower density than current alloys, high strength, creep resistance and resistance to oxidation. However, such materials are usually prohibitively brittle. Some complex crystals show anomalously low flow stresses, such as Ti_3SiC_2 , Nb_2Co_7 , W_2B_5 , Ta_2C and Ta_4C_3 . It is shown, with density functional theory, that these materials undergo heterogeneous elastic deformation at the unit cell level. This elastic heterogeneity is linked with the flow stress by atomistic modelling of dislocations, consistent with experimental observations of the MAX phases. The effect was observed experimentally in alloys of the structure type Ti_2Ni , a giant FCC structure. The effects can be substantial, suggesting this is the first step to controlling flow in complex crystals.

Adhesive properties of refractory materials

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Keywords: adhesion, refractories, phosphate binders

Unshaped refractory materials are divided by use and by the way of application (according to the already abolished standard EN 12475-1) into castables, torkreting materials, refractory mortars and other materials [1]. Among basic required properties of refractory materials belongs for example mechanical properties (compressive, tensile, bending strength), refractoriness or heat stability. Increased requirements on some special properties of refractory materials are based on their use. For example, low shrinkage, low thermal expansion, corrosive resistance, low bulk density, low coefficient of heat transfer or high adhesion. And just the study of adhesion of refractory materials, especially at the fresh state, was the aim of this work. High adhesion is important in case of torkreting materials or repair mixes, which are used to gluing cracks, cavities or even to missing parts of the wall that were formed during operation of thermal equipment.

The work is divided into two parts, the methodical and the experimental. The methodical part is focused on measurement of adhesion at the fresh state. Adhesion after solidification and hardening can be measured for example according to the standard EN ISO 9046 [2] via cyclic tensile stress of a connection of two pieces of refractory materials. But there is no standard for testing of fresh refractory mixtures. However it represents important property for easy application to the damaged surface with undesirable downfall. For mentioned purpose,

we used the procedure from the standard for measurement of traditional glues. Use of the so-called Probe Tack tester appeared as the best [3]. It is based on a steel probe (plate) with 50mm in diameter (in our case we used the probe with 35mm in diameter), which approaches to the specimen surface and then it presses the material by defined force. After a short delay, when the force is equal to zero, so an elastic tension could relax, the probe moves up from the specimen surface by defined rate and the loaded force is measured at the same time, as you can see in the figure 2a. Scheme of the experiment is possible to see in the figure 1. The highest loaded force is the most important value. The progression and arrangement were modified as, so it will be possible to measure mixtures with consistency from casting materials to relatively rigid repair mixes. Due to changing properties of curing refractory mixtures, mentioned Probe Tack tester was cyclically repeated. From gained data, it was possible to determine the setting time, workability time, maximal adhesion, average adhesion and impulse.

In the second part, we tested the modified Probe Tack tester method on a selected refractory material with chemical, phosphate binder. The binder was represented by the mixture of aluminous cement SECAR 71 with phosphoric acid and the aggregate was represented by mullitic Mulcoa 70. We measured the influence of the composition (content of the binder, content of the aggregate, content of liquid compound and its concentration) and condition (temperature) on progression of the setting.

Figures

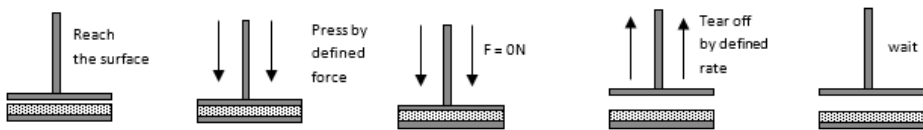


Figure 1 – Scheme of modified Probe Tack tester

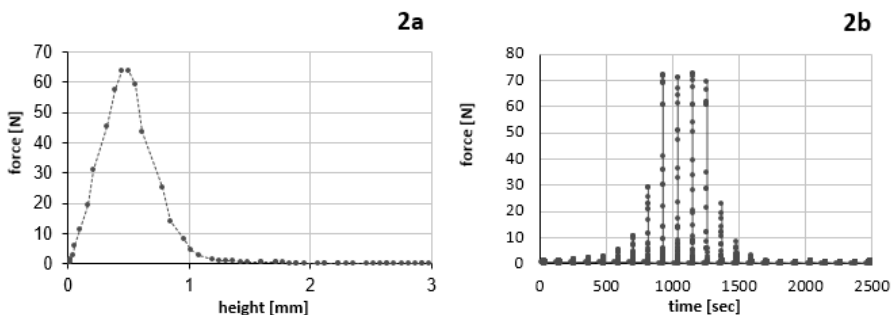


Figure 2 – a) Progression of loaded force during tearing off the probe. b) Progression of loaded force during cyclic tearing off the probe (Probe Tack tester)

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High temperature oxidation behaviour of Mo-Si-B-Ti alloys in dry and wet environments

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Keywords: Mo-Si-B alloys, high temperature oxidation, evaporation

Mo-silicide based alloys with high Ti concentrations are a new class of high temperature materials with a promising property combination. The good oxidation resistance of these alloys at temperatures above 1000°C relies on the formation of a protective silica layer. In dry atmospheres, this scale is remarkably durable. However in water vapour containing environments, silica evaporates leading to a substantial material loss over long period of time. Theoretical assessments reveal that Si(OH)_4 is the dominant vapour species as it possesses the highest value of vapour pressure at temperatures below 1300°C (see Fig.1). In our study, the stability of silica grown on Mo-Si-B-Ti alloys in water vapour-containing atmospheres was experimentally investigated. Oxidation tests were carried out at 1100°C in dry as well as wet atmospheres. In addition, two-steps experiments were conducted in order to investigate the silica stability in water vapor containing atmospheres. A protective silica scale formed during exposure to dry laboratory air (first step) was subsequently exposed to a wet atmosphere (second step). Oxide scales formed during oxidation were characterized using standard experimental techniques such as X-ray diffraction and scanning electron microscopy. It was observed that the formation of a protective silica layer was retarded in water vapour containing atmospheres leading to the deterioration of the oxidation resistance of the Mo-Si-B-Ti alloys. Possibilities to improve the oxidation behaviour of these materials are discussed.

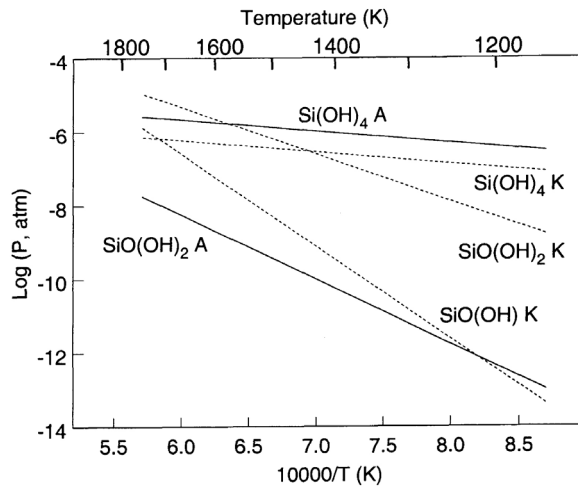


Figure: Calculated vapour pressure of Si-OH species over SiO_2 at $x(\text{H}_2\text{O}) = 0.37$ and $p_{\text{total}} = 1$ bar. The lines labeled K were calculated from thermodynamic functions taken from Krikorian's estimates based on the pseudo halide behaviour of the hydroxyl group. The lines labeled A were calculated from the thermodynamic functions taken from Allendorf's *ab initio* calculations [1]

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Natural silk fibre as a novel reinforcement for strong and tough epoxy composites

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Keywords: biopolymer, composites, toughness, sub-ambient, impact

Among all natural fibres, silk fibre is unique because of its continuous length of over 1000 m and protein composition, which results in much better elasticity and toughness than polysaccharide-based plant fibres[1]. Silk fibres also have other advantages such as low-density (1.3 g cm^{-3}), moderate strength ($\sim 500\text{--}700\text{MPa}$) and eco-friendliness. However, the use of natural silk fibres for engineering composites or biomedical composites has not been explored in detail.

We systematically studied silk fibre as a new reinforcement for thermoset epoxy resin, and fabricated a series of silk fibre reinforced composites (SFRP) using two silk species of common *B.mori*/*Bm* silk and uncommon *A.pernyi*/*Ap* silk[2,3]. Mechanical property characterization suggests (results shown in **Figure 1 (a, b)**): 1) both *Bm* and *Ap* silks with 60% volume fraction result in doubled specific modulus and specific strength compared to pure epoxy resin. 2) *Ap* silk with superior tensile elongations can absorb more breaking energy (11.7 MJ m⁻³) and induces much improved toughness of SFRP. Sub ambient temperature mechanical characterization in **Figure 1 (c, d)** shows: 1) the 60 vol.% *Ap*-SFRP possesses the highest specific strength and breaking energy at three sub-ambient temperatures (–50, –100 and –150 °C). 2) At –50 °C, the *Ap*-SFRP has a balanced strength of 471 MPa and a ten-fold increase in breaking energy from 1.7 to 24.3 MJ m⁻³. The silk reinforcement can bring down the tough-brittle transition temperature of SFRP to at least –50°C. **Figure 1(e)** shows a clear effect of the volume fraction of silk reinforcement on impact strength of SFRPs. When silk reinforcement becomes prominent (volume fraction > 50%) the impact strength surges to 71 kJ m⁻². In conclusion, silk reinforcement proves to enhance the mechanical strength and toughness of epoxy composites at both room temperature and subambient temperature, and with a dominant volume fraction, silk-epoxy composites achieves a boost in impact strength.

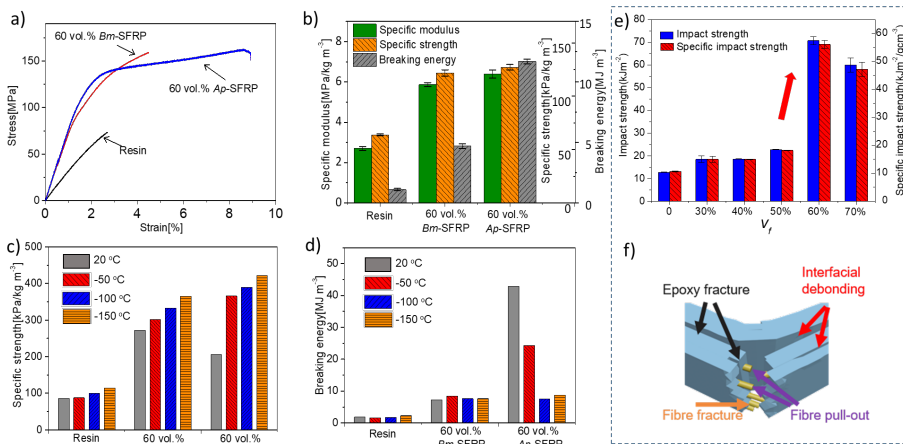


Figure 1: a) Comparison of stress-strain curves of epoxy resin, 60 vol.%-*Bm*-SFRP and 60 vol.%-*Ap*-SFRP. b) Derived tensile properties including specific modulus, specific strength and breaking energy of silk fibre reinforced polymer composites or SFRPs at room temperature. c,d) Derived specific flexural strength and breaking energy of SFRPs at sub-ambient temperature. e) Impact mechanical properties of different fibre volume fractions of *Bm* SFRPs.

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The research on ductile resistance of deformed copper in various temperatures and various velocity of deformation

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A number of properties determine the susceptibility to annealing of material, such as atomic structure (type, content, form and location of elements) which defines its chemical purity and the structure determined by the quantity and the size of grains. All the defect are very important, especially the ones that mostly determine the degree of material strengthening (dislocations). The wire rod's structure which undergoes the cold deformation process and after that the recrystallizing annealing process is shaped under hot deformation conditions in which the decisive role plays the dynamic recrystallization. In case of multi-stand line (Contirod® line) the static recrystallization between individual rolling stands appear. Thermodynamic conditions of the deformation process shape the final wire rod's structure at the macro, micro and atomic level which defines all properties of the wire rod, including its annealing susceptibility. The whole phenomena occurring in the described processes contributed in the program and scope of the experimental research.

The aim of the research was to assess the influence of the deformation processes at the elevated temperatures at a specific velocity on the flow curves of wire rods. These tests are a simulation of the dynamic recrystallization process which occurs during the continuous casting and rolling process. These tests were conducted using the Gleeble simulator. The deformation was carried out in the scope of 350°C to 500°C and the deformation velocity of 0,1 1/s and a total logarithmic deformation of 1,2. Differentiated susceptibility to the annealing process results in a different material structure and different values of ductile resistance of the material during the hot deformation processes.

Investigation of silicon carbide dispersion strengthened austenitic steels

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Keywords: ceramic dispersion, 316L austenitic stainless steel, attrition milling, Spark Plasma Sintering, structure

In this work 0.33 wt. % and 1 wt. % SiC nanoparticles were mixed in 316L austenitic steel matrix. The high efficient attritor milling provides size reduction of the 316L steel grains and homogeneous distribution of the SiC nanoparticles before sintering process. Spark plasma sintering (SPS) was used for compaction of milled composites. The effect of the SiC addition on the milling efficiency and the structure of the composites have been studied. It was found that the amount of ceramic addition influenced the efficiency of milling process, powder mixtures with flake-like grains have been obtained. The intensive milling assured an optimal coverage of 316L stainless steel grains with ceramic submicron sized particles in both cases. The sintered composites showed high densities with the presence of small amount of closed porosities. Structural, mechanical and tribological examination of 316L/SiC composites is presented in this work.

Poster

Structure-related properties of bionanocomposites based on poly(lactic acid), cellulose nanocrystals and organic impact modifier

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Keywords: bionanocomposites, poly (lactic acid), cellulose nanocrystals, lignin coating, organic impact modifier

There are ever increasing demands of modern society for products made from renewable and sustainable resources that are biodegradable, non-petroleum based, carbon neutral, and have low environmental, animal/human health and safety risks. Poly (lactic acid) (PLA) as a leading biobased, biodegradable and biocompatible thermoplastic with high strength and modulus still faces important industrial problems such as a slow crystallization, inherent brittleness, low thermal stability and low impact resistance to compete with synthetic commodity polymers. The slow crystallization rate of PLA results industrially in products with a very low crystallinity state. In this paper is the problem addressed by the addition of cellulose nanocrystals (CNCs) and lignin-coated cellulose nanocrystals (L-CNCs) bio-based nucleation agents to increase nucleation density, enhance higher crystallization rate and increase the thermal stability of PLA. However, the effectiveness of CNCs/L-CNCs incorporation strongly depends on the quality of the dispersion and distribution in non-polar PLA matrix. The two-step pre dispersion processes based on solution ultrasonication/mechanical stirring and than masterbatch preparation by melt mixing using an internal mixer has been used to maximize dispersion and individualize CNCs/L-CNCs agglomerates within the matrix. It is relatively easy to improve the ductility, on the other hand, it is much more challenging to increase the impact toughness that depends on many extrinsic and intrinsic variables. An organic impact modifier masterbatch has been used to investigate its influence on overall bionanocomposite properties. An injection molding machine has been used to prepare final samples and these were characterized by means of structure-morphology investigation and thermo-mechanical properties evaluation.

Comparison of FDM manufactured cavity insert and conventional injection moulding: An influence on crystallization, morphology and thermo-mechanical properties of injection moulded parts

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Keywords: additive technology, FDM, injection moulding

Injection moulding is a major mass-technology for production of high-quality thermoplastic and thermoplastic composite parts. Once the initial costs have been paid the price per produced part is extremely low and part is then created up to million times. However, it is a long-term process to scale up the production and in case of prototyping, additive manufacturing technologies are very quick and parts are literally created overnight in some cases. On the other hand, produced parts do not possess the required level of mechanical properties as their injection moulded counterparts due to technological processing conditions. Despite this, the main advantage of additive manufacturing technologies is the fast production that doesn't require expensive injection mould tooling.

Injection moulding cavity inserts made of certain polymers by an additive manufacturing technology offers advantages of both mentioned technologies. Production of parts with comparable mechanical properties as conventional injection molded counterparts is possible without making an expensive steel injection mould. On the contrary, polymer moulds as thermal insulators require longer cooling times. The aim of this paper is to compare injection molding into conventional and additively manufactured moulds/inserts. The influence of processing conditions on crystallization behavior, morphology and thermo-mechanical properties has been studied. Removable injection mould cavity inserts from Ultem 9085 (Polyether Imide) were made by FDM (Fused Deposition Modeling) technology and polypropylene (PP) was used as an injection molded polymer.

Evaluation of high-temperature degraded concrete with content of blast furnace slag by non-destructive approach

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Keywords: Non-destructive testing, concrete, cement, high-temperatures, destructive tests, cement, impact-echo, blast furnace slag

In the field of civil engineering a diagnostics acoustic non-destructive testing is widely used and in the past years finds application even in testing of high-temperature degraded concrete structures. This article is focused on non-destructive testing by Impact-Echo method, ultra-

sonic velocity pulse method and destructive testing of high-temperature degraded concrete test beams of dimensions $100 \times 100 \times 400$ mm fired at 200–1200 °C. Designed mixtures have same composition of fine and coarse aggregate, additives and water cement ratio but different cement is being used. The first mixture uses high-strength cement CEM I 42.5 R and second mixture use CEM II /A-S 42.5 N, which contains blast furnace slag. In recent study Karahan compared behavior of cement specimens with usage of cement with content of fly ash and blast furnace slag. The paper concludes, that cement test specimens with content of blast furnace slag better resist high temperatures up to 800 °C. Test specimens have higher residual strength and lower porosity than specimens with content of fly-ash or regular Portland cement. The presented paper aims to test these assumptions by non-destructive measurements.

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Mechanical properties and wear damage of silicon carbide composites with carbon nanotubes

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Four types of composites based on silicon carbide were prepared by hot pressing (1850°C/Ar/60 min/40 MPa) by CCVD method from SiC/CNT powder precursors. Addition of CNT was 0, 2.5, 5, 10 wt%.

Mechanical properties such a hardness and elastic modulus of the composites were investigated by nanoindentation using Berkovich indenter tip (applied load 500mN) and macro-indentation method with Vickers diamond tip under the load of 1kg (9.81 N). Indentation fracture toughness (K_{IC}) was calculated from the lengths of the radial cracks produced in the material under the load of 10 kg (98.1 N). The microstructure and chemical composition of the resulting materials were studied by SEM/EDX. The electric conductivity as function of CNT content was determined by the four point probe method. Tribological properties have been characterized by the ball-on-disc method at the ambient temperature and dry wear conditions at the 5N load, 500 m sliding distance with the static partner made from SiC. Area of the wear track cross-section was measured using 3D optical profiler and the wear loss volume was calculated according to the international standard ISO 20808.

Resulting materials were relatively hard $HV_1=24$ GPa and with presence of CNT moderate decrease down to $HV_1=17-19$ GPa. Similarly, the fracture toughness decrease with presence of CNT from 7 MPa.m^{1/2} down to 4 MPa.m^{1/2}. Nanoindentation shown that hardness H_{IT} of monolithic sample was around 26 GPa and with increasing amounts of CNT decrease down to the roughly same value 21 GPa. The samples with CNT had similar modulus of elasticity ($E_{IT}=337-348$ GPa) and for monolith sample $E_{IT}=434$ GPa. Electrical conductivity increases with amount of CNT (1.76 S/m for monolith, 420.2 S/m for 2.5% CNT, up to 2873.6 S/m for 10% CNT). Specific wear rate was same by all cases with the increasing tendency by increasing addition of CNT. Main wear mechanisms of created wear track were cracking and oxidation.

Acknowledgements

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Deformation behavior of harmonic structure designed SUS304L austenitic stainless steel at elevated temperatures

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Keywords: heterogeneous, homogeneous, strength, ductility, compression test

Human being have struggled with metals for long time and generally, it was believed that grain refinement was the best way. However, improving strength usually be inconsistent with improving ductility. Conventionally, coarse grained metals show low strength and high ductility, on the other hand, fine grained ones exhibit high strength and low ductility.^[1-3]

In recent years, to achieving such an antinomy, a unique heterogeneous microstructural design based on bimodal grain size distribution, called Harmonic Structure (HS), has been proposed for the strengthening of metallic materials. The HS design consists of a specific spatial distribution of fine and coarse grained regions, wherein coarse-grained areas are interlinked with a three dimensional interconnected network of fine-grained regions. The HS design extremely efficient in delivering a variety of high performance metallic materials, via achieving a combination of high strength and high ductility together with excellent reproducibility. The corrosion resistance as well as the fatigue properties are also superior to the conventional structure materials too.^[4]

In the present study, we applied HS design to an SUS304L austenitic stainless steel by means of Severe Plastic Deformation Powder Metallurgy Process (SPD-PM). An EBSD band contrast image of HS is shown in Fig.1. The HS designed SUS304L material exhibited superior mechanical properties at elevated temperatures compared to the conventional homogeneous (Homo) structure counterparts. The results of high temperature compression tests are shown in Fig.2. Such advantages in mechanical properties are attributed to the heterogeneity of the HS materials.

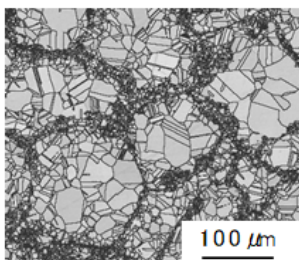


Fig1. An EBSD Image of the SUS304L compact with Harmonic structure

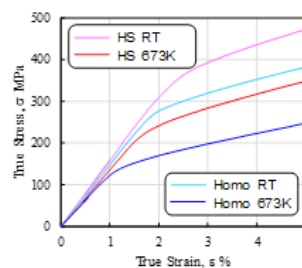


Fig2. High temperature compression test results of HS and Homo 304L materials

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Atom probe tomography study of nickel-based superalloy

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Ni-base superalloys are essential materials used primarily in the hot section of jet turbine engines [1]. Ones provide excellent ability to retain strength, to withstand creep and fatigue, and to resist oxidation at elevated temperatures above 540 °C [2–4].

High temperature strength of these alloys derives from a high volume fraction of γ' phase. The γ' phase exists in the alloys in the form of cuboids, which has an ordered L12 structure with coherent interfaces with the continuous fcc γ phase matrix, which presents as narrow channels between the γ' cuboids [5–6]. The highly ordered L12 structure gives high rigidity of the γ' phase and low dislocation tolerance, thus the dislocation mainly limited in the γ channels. Therefore, mechanical properties of Ni-based superalloys notably depend on morphology, volume fraction, nanostructure and composition of γ' phase [7]. Though, alloying elements and modes of thermomechanical processing may affect these parameters [8].

Atom-probe tomography provides information about the local chemical composition and distribution of atoms within the material in nanoscale. This information can increase understanding of phase transformations depending on the initial composition and heat treatment regimes.

In this work the results of atom probe tomography study of granulated nickel superalloy VV751P designed in OJSC "VILS" are presented. Samples with different heat treatment regimes were under our study. Both γ' and γ phases were detected in each sample. The composition and the volume fraction of both phases are analyzed. Furthermore, a nanoscale embryos of γ and γ' phases were detected in one of the sample.

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The microstructure evolution of bainitic steel after low-cycle fatigue tests

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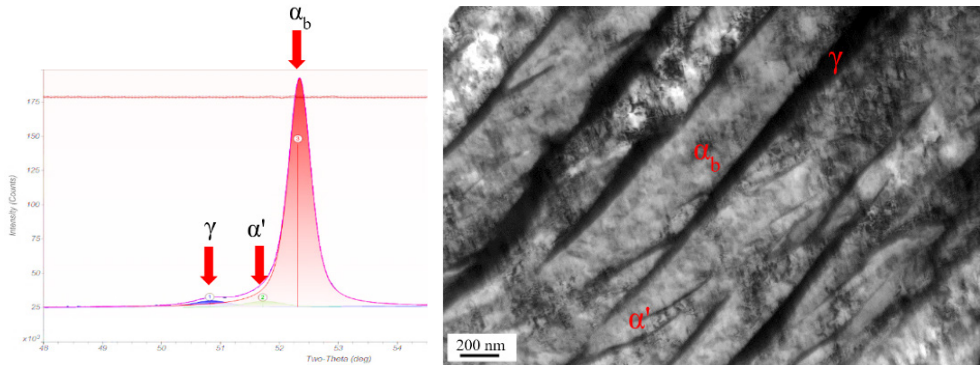
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Keywords: low-cycle fatigue behaviour, XRD measurements, microstructure evolution

The aim of the present study was to analyze the microstructure of low alloy steel after low-cycle fatigue (LCF) tests. The research carried out within the project was financially supported by the National Centre for Research and Development and consisted in developing of new high-durability materials for the railway industry. After appropriate selection of heat treatment conditions, bainitic steel containing minor additives of Mn, Cr and Si was produced by the ArcelorMittal Poland steelworks. The microstructure investigations were carried out on samples in the initial state, before deformation and after the LCF tests at room temperature and various strain amplitudes with four different levels of the total strain. The cyclic softening of fatigue curves was observed for all samples. The measurements were carried out on the transverse sections in the direction parallel to the force applied during LCF, near the fracture surface. The X-ray diffraction (XRD) method was used for phase analysis of the obtained microstructure. Results showed the presence of bainitic ferrite phase (α_b), retained austenite (γ) and deformation martensite (α'). It was noticed that the presence of certain phases depended on the area of measurement at the fracture surface. The obtained results were confirmed by the microstructure observations performed with the use of transmission electron microscopy (TEM). The presence of α' phase and the reduction of retained austenite after LCF test were observed.

Figures



Example of the microstructure of sample after LCF tests–TEM image and phases identification from XRD of bainitic ferrite (α_b), laths of retained austenite (γ), and deformation martensite (α')

Acknowledgments

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Microstructure characterization of the Inconel 686 clad layer after high temperature corrosion tests in aggressive gases and ashes

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Keywords: laser cladding, Inconel 686, high temperature corrosion, aggressive environments

Ni–base alloys are used as one of the most important coating material and can be applied in a different environments and parts of devices having various applications. To protect the surface of the substrate 13CrMo4-5 steel against aggressive environments the Inconel 686 as clad layer was used. The Ni–Cr–Mo–W alloy is characterized by the excellent high temperature corrosion resistance, good strength and good ability to work in aggressive environments. The corrosion process was performed only in oxidizing mixture of gases like O_2 , CO_x , SO_x . The second part of corrosion experiment concerned the corrosion test of the clad layers in reducing atmosphere of the specified gases with ashes, which contained e.g. Na, Cl, Ca, Si, C, Fe, Al. After corrosion experiment in both cases the oxide scales on the substrate and clad were created. The

scale on the 13CrMo4-5 steel had 70 μm thickness while the scale on the clad layer had less than 10 μm . The sulfur compounds were found on the top of overlay surface while in case of the application of ashes on the top of clad layer surface was observed the higher contents of silica compounds. The microstructure, chemical composition of the clad layer and scales were investigated by means of a light microscope, the electron microscopes (SEM, STEM, TEM) equipped with the EDS detectors.

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Application of surface engineering to reduce tool wear

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Keywords: wear, PACVD, coating, multilayer

Tool wear has large impact not only on production costs but also on the quality of the final product. Therefore the intensity of the tool wear should be reduced as much as possible. This leads to the development of new surface-engineering techniques in order to improve their wear resistance. In this paper new duplex treatment consisted of plasma nitriding and gradient multilayer PACVD coating was developed. The improvements of the treated surfaces in terms of wear and corrosion resistance have been analysed and new duplex treatment showed significantly higher wear resistance when compared to non-coated steel. Finally, new duplex treatment was successfully applied on the moulds.

On the effect of precipitates on intergranular corrosion of severely deformed 2xxx aluminum alloy

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Keywords: aluminum alloy, intergranular corrosion, severe plastic deformation, structure, aging

The effects of severe plastic deformation by isothermal rolling at a liquid nitrogen temperature with a strain up to $\epsilon \sim 2$ and subsequent natural and artificial aging on the resistance to intergranular corrosion (IGC) of preliminary quenched 2024 aluminum alloys of standard composition and with zirconium additions were investigated. Intensity and depth of corro-

sion in 3% NaCl+1% HCl water solution were analyzed by optical and scanning electron microscopy.

The microstructure of standard cast and homogenized at 370°C, for 6 hrs, alloy was represented by dendrite cells with an average size of about 400 μm , and by 2.4 ± 0.1 % volume fraction of $\text{Al}_{11}\text{Si}_5(\text{CuFeMn})_3$ and S (Al_2CuMg) excess phases, distributed mainly along cell boundaries. TEM analysis has also revealed near homogeneously distributed inside the alloy matrix Mn-rich T-phase ($\text{Al}_{20}\text{Cu}_2\text{Mn}_3$) of 50-200 nm in diam. Meanwhile, the Zr modified alloy has a grain type microstructure with a grain size of 300–400 μm , 1.1 ± 0.1 % volume fraction of θ (Al_2Cu) excess phases and coherent Al_3Zr precipitates of 10-50 nm in size inside grains.

Cryorolling has formed well-defined shear bands and resulted in pancaked grains with a thickness of 100–200 μm , especially in Zr modified alloy, with well-developed lamellar structure having a cell size of about 0.5 μm . Besides, strings of excess phases with low interparticle spacing along and across the rolling direction were formed. Concurrently, a brittle failure of phases resulted in increased density of particles along grain boundaries and on perpendicular contact surfaces of a specimen. The nature of the alloy structure transformations is discussed.

It was found that irrespective the alloy composition and aging conditions, the general path of corrosion attack lies along grain boundaries contacted with strings of excess phases; that was the main structural factor, influencing the alloy corrosion behavior. Zr additions, significantly increased the alloy IGC resistance in both naturally and artificially aged conditions, reducing its depth and intensity. The main reason of such a behavior is caused by absence of coarse excess impurity phases. Besides, Zr aluminides were acting as strong barriers of grain growth and dislocation rearrangements, decreasing the intensity of corrosion damage.

It was concluded that the effects of treatment, involving cryorolling, and of Zr additions was conditioned by synergy of phase and structural factors. Ambient temperature annealing of cryorolled alloy led to formation of Guinier-Preston-Bagaratsky zones (GPBZ) owing to aluminum solid solution decomposition. Because of their coherency with a matrix, difference in electrochemical potentials, consequently, the IGC driving force, was low, causing insignificant corrosion damage. Under artificial aging of cryorolled alloy, solid solution decomposition was influenced by recovery and recrystallization of the matrix, changing kinetics and sequence of aging, as morphology and distribution of its products. As a result, non-coherent precipitates of stable phases were formed in recrystallized areas instead of plate shape products and, apparently, intensified the alloys corrosion damage. In that case the effect of Zr was the most pronounced as their aluminides played their main role – suppression of the alloy matrix structure transformations, involving dislocation rearrangements.

Acknowledgements

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Investigation of mechanism of glycine nitrate processes for preparation of lanthanide oxides nanocrystallites

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Keywords: glycine-nitrate processes, lanthanides oxides nanocrystallites, SEM, FTIR

Glycine-nitrate processes, a simple and inexpensive laboratory method, have been known for more than dozen years. Yet, but the mechanisms of the particular chemical transformations remain still unclear. Our group have recently published an article [1] describing a method of the preparation of three dilanthanide trioxides (Gd_2O_3 , Sm_2O_3 , and Er_2O_3) in a shape of nanocrystallites via thermal decomposition of a transient complex formed *in situ* from suitable lanthanide nitrates. According to our findings, thermal decomposition (calcination at 600 °C for 1 hour) of the complex of suitable nitrate and glycine contains several meso-stages and intermediates. Thus, is not straightforward combustion. To bring a further insight into the chemistry of glycine nitrate processes, we have prepared a series of samples of glycine- $\text{Gd}(\text{NO}_3)_3$ aqueous solution and treated them at temperatures of 195 °C, 220 °C, 240 °C, and 310 °C to record possible intermediates. Moreover, we have prepared a gel-like matter from the aqueous solution by drying it at 120 °C. Thus obtained samples were characterized by using scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS) and infrared spectroscopy (FTIR). There is a clear trend of a slow transformation of amorphous gel-like structure with sporadic surface ruptures at lower thermal treatments to the more organized net-like structure apparently sourcing from the initial ruptures at higher thermal treatments visible in the SEM images. Furthermore, EDS analysis revealed a significant progressive loss of nitrogen in samples clearly associated with the increasing processing temperature, which corresponds well with the results from FTIR. This research is a part of our broader undergoing investigations aimed to reveal and explain the mechanisms of formation lanthanide-oxide nanocrystallites by glycine-nitrate processes.

Acknowledgments

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Anomalous strain hardening behavior of harmonic structure designed nickel

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Keywords: heterogeneous structure, homogeneous structure, Stress partitioning, Strain hardening

Grain refinement in metals by Severe Plastic Deformation (SPD) is efficient way to increase strength, but often lead to unacceptable reduction in ductility. One of the best solutions to solve this problem is creation of an unique “Harmonic Structure (HS)” . The HS design consists of a specific spatial distribution of ultra- fine-grains (UFG) and coarse-grains (CG), wherein CG “Core” are embedded in a three-dimensional interconnected network of UFG “Shell”. Such a controlled bimodal grain sized microstructure is a new material design paradigm allowing the improved mechanical performance of structural materials, that is, enhancement of strength without sacrifice its ductility [1,2]. Those HS materials are able to fabricate via a SPD Powder Metallurgy process. In the present study, deformation mechanism of the HS materials is studied using a HS designed pure Ni compact. Fig.1 shows an EBSD image of HS-Ni compact, and a Shell network structure can be observed. Fig.2 indicates tensile test results of a HS and homogeneous conventional structure (Homo) Ni. As can be seen, although the HS material demonstrates almost the same 0.2% proof strength as the Homo counterpart, the HS-Ni shows anomalous strain hardening. According to TEM observation results of the Shell region after 5% tensile deformation, a large number of dislocations are found in the Shell. This suggests that stress concentration and partitioning to the Shell region lead to an anomalous dislocation emission, and hence the strain hardening became larger in the HS material.

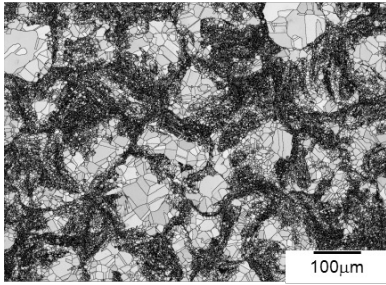


Fig. 1 An EBSD (band contrast + grain boundary) image of a HS-Ni compact.

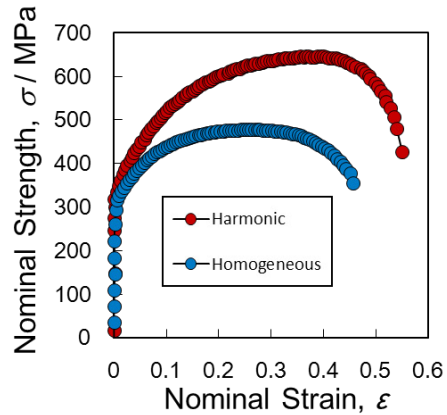


Fig. 2 Tensile results of HS and Homo Ni compacts

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Determination of cooling rates in iron meteorites by the structure of spinodal decomposition

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Keywords: structure, meteorites, cooling rate, spinodal decomposition

Metal phases of meteorites are an iron-nickel alloys. Slow cooling (one degree per million years) results in cloudy zone formation [1] due to spinodal decomposition at low temperatures (below 400 °C). This zone *consists* of high-nickel particles of FeNi (tetrataenite) embedded in low-Ni matrix. In iron, stony-iron and stone meteorites, the structure of the spinodal decomposition is formed during cooling in the area of M-shaped diffusion profile of nickel in the range from 30 to 42 wt.% Ni. The microstructure of the cloudy zone in these regions is formed in a practically pure Fe-Ni-Co meteorite metal after exsolution of minor elements such as S, C, and P at high temperatures in their sulphides, carbides and phosphide [2]. A detailed study of cloudy zone by electron microscopy makes it possible to estimate the rate of cooling of a metal and to study the processes leading to the formation of observable nanostructures.

In this work, we study the cloudy zone structure by various methods. Firstly, average high-nickel particle sizes was measured in accordance with the work [1, 3]. Secondly, interparticle distance in the cloudy zone was measured. These distances were of interest because spinodal decomposition is a wave process and one can estimate wavelength (or period) of such phenomenon using this distance. Fragments of several meteorites are the objects of this study: Seymchan (Pallasite, PMG), Canyon Diablo (Iron, IAB-MG), Odessa (Iron, IAB-MG). Studies of microstructure were carried out using the Zeiss Axiovert 40 MAT optical microscope and scanning electron microscopy Carl Zeiss Sigma VP with energy dispersive spectroscopy (EDS) and electron backscatter diffraction (EBSD) units.

The range of variation of the interparticle distance in the meteorites Seymchan, Canyon Diablo, Odessa are 140 nm, 230 nm and 160 nm respectively. The average particle size ranges in the meteorite Seymchan – 120 nm, Canyon Diablo – 200 nm, Odessa – 110 nm. Thus, we suppose that cooling rates, obtained with interparticle distances and average particle size [1, 3, 4], will differ. Further research in this area will improve methods for estimation of metallographic cooling rates and allow to explain thermal history of meteorites more accurately.

Acknowledgments

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Continuous production of ultrafine to nanocrystalline wires of pure titanium

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A description is given of a possible use of Conform SPD (Severe Plastic Deformation) continuous extrusion process by which ultrafine to nanostructured pure titanium can be produced on a continuous basis. The process has been derived from the ECAP technique but, unlike

ECAP, it offers continuous production of high-strength wire. Conform SPD is further combined with cold working. CP titanium processed by Conform technology exhibits improved mechanical properties and very favourable biocompatibility, due to its fine-grained structure. The article presents the current experience in the production of ultrafine CP titanium using this technology.

Mechanical test of 3D printed titanium samples with oriented inhomogenous material structure

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Keywords: 3D printed, metal, structure, mechanical test, production parameters

The 3D printing is an ideal technology to produce non-conventional samples for research projects before the industrial application of the produced parts or new material structures. The technology let us to influence the production parameters, and make samples even with different material parameters in each layer. Due to this it is possible to produce bulk samples with oriented inhomogeneous material structure. This possibility give a production solutions to individual parts with designed orientation and special mechanical properties. The application field of these parts is the medical and industrial field as well.

Small size mechanical test samples were designed considering to the ASTM-Additive Manufacturing standards. The design process of the samples do not included just the geometrical part, but the orientation structure also. That mean during the CAD process regions were designed into the bulk geometry. According to the final CAD model and the build material (in our case Titanium Grade 23 ELI) the process parameters were connected to the CAD model regions, and were sliced and 3D printed.

With the printed samples, different mechanical tests were executed, for example: tpb test, Charpy impact test, tensile test, etc. After the mechanical tests metallographic examination were perform, to examine the microstructure of the different regions.

The results shows that, the samples with oriented material structure have special microstructures, which highly influence the directional mechanical load capacity of the samples.

Evaluation of retained austenite in a TRIP steel using EBSD and X-ray diffraction technique

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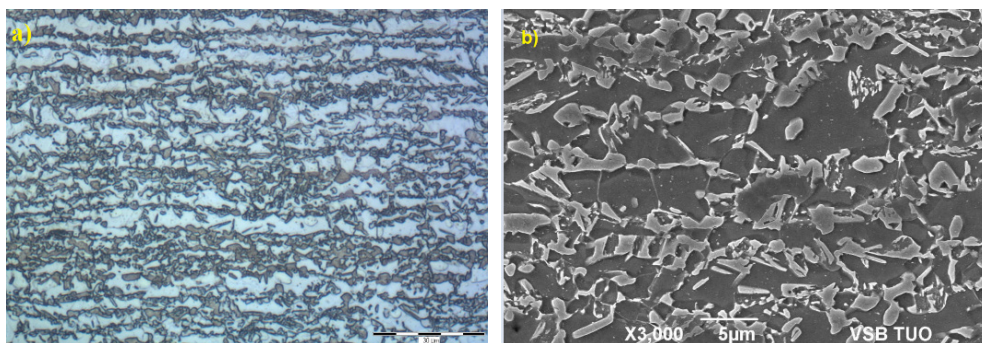
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Keywords: TRIP steel, retained austenite, EBSD

The presented paper is devoted to evaluation of retained austenite in a TRIP 800 C-Mn-Si-P with increased phosphorus content using EBSD (Electron BackScatter Diffraction) technique. EBSD is a useful technique for microstructural characterization and analysis of crystalline materials. Many of the structural parameters that control properties and performance of the materials can be derived from EBSD data, e.g. phase constituents, grain size, mechanical anisotropy and residual strain. The steel was studied after a standard heat treatment, including intercritical annealing and annealing in the range of bainitic transformation. This kind of steel exhibits a multiphase microstructure which undergoes a deformation induced phase transformation. 5% and 10% tensile deformation was applied to a part of the tested sheets. This resulted in microstructure changes (reduction of retained austenite content which transformed into martensite). Steel microstructures were analyzed by light microscopy (LM), scanning electron microscopy (SEM) and EBSD technique. The EBSD analysis results show that the volume fraction of retained austenite decreases with the increasing degree of deformation.

Figures



Microstructure of TRIP steel after 5% deformation, a) LM with Nital etch, b) SEM

Table: The chemical composition of the studied TRIP 800 steel (wt. %)

C	Mn	Si	P	S	Cr	Ni	Cu	V	Al	Nb
0,20	1,50	1,50	0,050	0,005	0,16	0,15	0,06	0,02	0,006	0,02

Acknowledgments

This paper was prepared with a contribution of the projects “SP2018/70 Study of relationships between the technology and processing of advanced materials, their structural characteristics and utility properties” and “SP2018/60 Specific research in the metallurgical, materials and process engineering”.

Harmonic structure design of 0.3mass% carbon steel

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Keywords: heterogeneous, homogeneous, strength, ductility, ferrite, perlite

Harmonic Structure (HS) has a heterogeneous microstructure consisting of bimodal grain size together with a controlled and specific topological distribution of fine grains area ('Shell') and coarse grains area ('Core'). In other words, the HS is heterogeneous on micro- but homogeneous on macro-scales. The most unique feature of HS is its continuously connected ultra fine grain (UFG) Shell structure. The HS design has been applied SPD powder metallurgy process. At a macro-scale, the harmonic structure materials exhibited significantly better combination of strength and ductility, as compared to their homogeneous microstructure counterparts. Those mechanical properties are essentially related to the ability of the HS to promote a large strain hardening and uniform distribution of strain during plastic deformation, leading to improved mechanical properties by avoiding or delaying localized plastic instability. Such outstanding mechanical properties of HS materials are attributed to the continuously connected UFG-Shell structure. This unique microstructure provides *stress partitioning* in the micro scale and *stress dispersion* in the macro scale [1, 2]. In the present study, we applied the HS design to 0.3mass% carbon steel (0.3C) by means of Severe Plastic Deformation Powder Metallurgy Process (SPD-PM). An EBSD band contrast image of the 0.3C-HS steel is shown in Fig.1. The 0.3C-HS steel exhibited superior mechanical properties compared to the conventional homogeneous (Homo) structure counterparts, as shown in Fig.2. In the 0.3C-HS steel, area fraction of perlite in the Core and Shell were 38.2% and 55.7%, respectively. The 0.3C-HS steel indicated higher strength and ductility than the Homo specimen, since larger amount of perlite formed in the HS material.

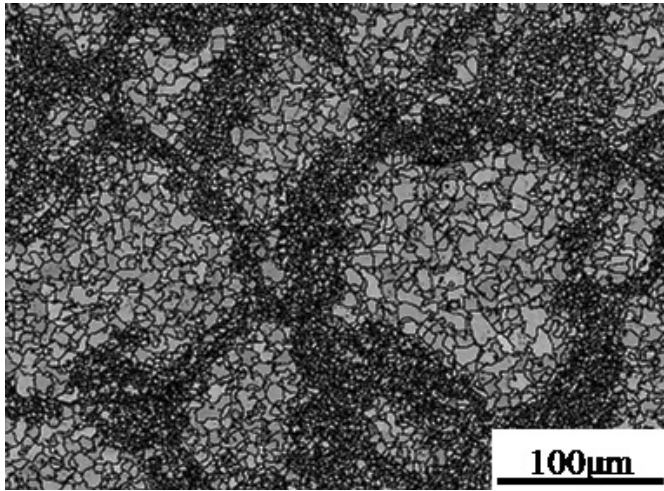


Fig.1 An EBSD band contrast image of the HS 0.3mass% carbon (0.3C-HS) steel compact

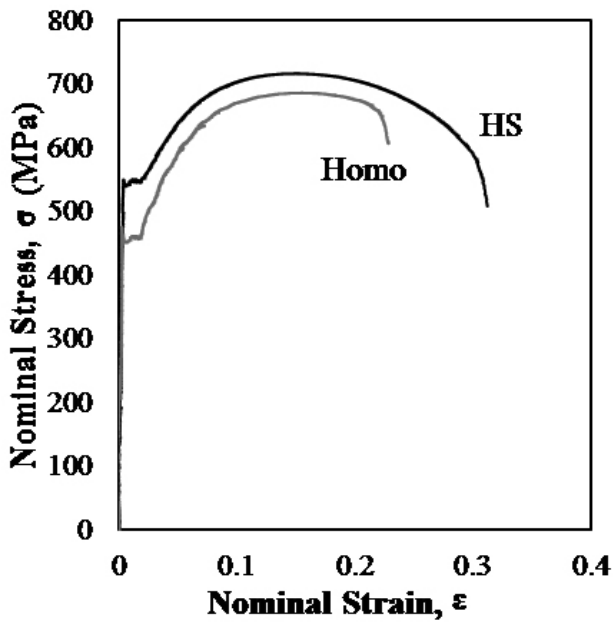


Fig.2 Tensile test results of the “HS” and “Homo” 0.3mass% carbon steels.

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Boron carbide/graphene platelets ceramics with improved fracture toughness, functional and tribological properties

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Keywords: Boron carbide, graphene platelets, microstructure, fracture toughness, conductivity, tribology

Boron carbide/graphene platelet (B_4C /GPLs) composites have been prepared with the addition of different weight percent of GPLs by hot-press processing technology at 2100 °C in argon. The influence of the GPLs addition on microstructure development, fracture toughness, electrical conductivity and tribological properties was investigated. The microstructure was studied by SEM, TEM, HRTEM, XRD and Raman spectroscopy. SEVNB method was used for fracture toughness and four-point Van der Pauw method for electrical conductivity measurement. Almost fully dense B_4C /GPLs composites have been prepared with lower wt.% of GPLs additives with relatively homogeneously distributed platelets in the matrix. With increasing amount of GPLs additives, the fracture toughness increased due to the activated toughening mechanisms in the form of crack deflection, crack bridging, crack branching and graphene sheet pull-out. The highest fracture toughness of 4.48 MPa.m^{1/2} was achieved at 10 wt.% of GPLs addition, which was ~50 % higher than the K_{IC} value of reference material. The electrical conductivity increased with GPLs addition with percolation threshold between 6–6.5 wt.% of GPLs and reached the maximum values at 8 wt.% GPLs addition. A significant improvement of electrical conductivity around two orders of magnitude up to 1526 S/m in the perpendicular direction and to 872 S/m in parallel direction was noticed. The friction and wear behaviour of B_4C /GPLs composites have been investigated using the ball-on-flat technique with SiC ball under dry sliding conditions at room temperature. The coefficient of friction for composites were similar, however the wear rate significantly decreased ~77 % in the case of B_4C +10 wt.% GPLs when compared to reference material at a load of 5 N, and ~60 % at a load of 50 N. Wear resistance increased with increasing GPLs content in regards to the present graphene platelets, which during the wear test pulled-out from the matrix, exfoliated and created a wear protecting graphene-silicon based tribofilm. For revealing and observation of the wear damages under the worn surfaces, focused ion beam (FIB) technique was used for the preparation of the cross section of wear tracks.

Acknowledgements

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Microstructure evolution of harmonic structure designed pure titanium compacts by thermo-mechanical processing

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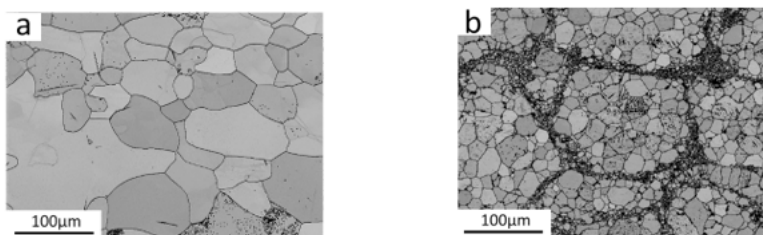
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Keywords: Harmonic structure, cold rolling, heat treatment, recrystallization, stress concentration

Through many years, conventional material developments have emphasized on microstructure refinement and homogeneity. However, “Nano- and Homogeneous” microstructures do not, usually, satisfy the need to be both strong and ductile, due to the plastic instability in the early stage of tensile loading. As opposed to such a “nano- and homo-” microstructure design, we have proposed the so-called, “Harmonic Structure (HS)” design concept [1],[2]. The HS materials have a heterogeneous microstructure consisting of bimodal grain size together with a controlled and specific topological distribution of fine and coarse grains, and demonstrate a significantly better combination of strength and ductility as compared to their homogeneous microstructure counterparts. It is attributed to the designed structure that coarse-grained (CG) areas (“Core”) are surrounded by three-dimensional continuously connected network of ultra-fine-grains (“Shell”). It is worthy to mention that the early stage of deformation of the HS is governed by the characteristics of interconnected network of the ultra-fine-grained (UFG) Shell region, especially by the volume fraction of Shell area. Therefore, increasing the fraction of Shell by thermo-mechanical processing (TMP), including cold-rolling and subsequent annealing process, will be an effective approach to obtain harmonic structure materials with high strength and ductility at the same time. In the present study, TMP is applied to Ti-HS compacts. The TMPs, i.e., 10% Cold rolling (CR) at room temperature (RT), followed by annealing at various temperatures between 873K and 1073 for 1.8 ks, are carried out. Fig.1 (a) and (b) show EBSD images of (a) as-HS and (b) after TMP (10%CR+873K, 1.8 ks), respectively. Remarkable is that UFG formation took place especially in the Shell region by the TMP. This strongly suggests that concentration of deformation was given mainly to the Shell region, and hence it lead to a predominant recrystallization in the Shell.



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Fracture toughness of ceramic hollow sphere filled metal matrix composites

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Keywords: Fracture toughness, Syntactic foam, Metallic composites, Porous materials

Metal matrix syntactic foams (MMSFs) or composite metal foams (CMFs) are high strength foams in which the porosities are ensured by hollow spheres. Nowadays, mass reduction and increasing loading in low-weight structures lead to the more extensive application of low density foams having high specific strength. As a consequence, their mechanical and fracture properties are becoming important. Al99.5 or AlSi12 MMSFs were produced by pressure infiltration [1]. As filler, ceramic hollow spheres (Globocer) were applied in ~65 vol% [2]. Three-point bending (TPB) samples for fracture toughness tests were machined from the infiltrated blocks, according to the standard [3]. The width of the samples was 25 mm, the span of the TPB apparatus was 100 mm, the diameter of the supporting rods was 10 mm. The notch was a 12 mm deep straight through notch, with a notch tip radius of 0.25 mm. The opening of the notch was followed by a double cantilever clip-in displacement gage. The load – notch opening curves were measured. At the load maximum a crack initialized in the notch tip and propagated, while the force decreased continuously. The average load maxima were 702 ± 25.5 N for Al99.5 and 845 ± 77.8 N for AlSi12 matrix, respectively. Due to the more brittle nature of the matrix, AlSi12 MMSFs showed higher scatter in their maximum forces. The load – notch opening curves were evaluated by the 95% secant method. A tangent line was fitted on the linear elastic part of the curve and a secant line with 0.95 slope was constructed. The intersection of this secant line with the original graph determines the conditional F_Q force that is required to calculate the conditional stress intensity factor (K_Q). The calculated K_Q values are fracture toughness (K_{IC}) if they satisfy the related criteria. For the last step the crack propagation was investigated. It was found that the crack initialized in the tip of the notch preferably propagated along the interfaces between the hollow spheres and the metallic matrix.

Acknowledgments

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Investigation on mechanical and flammability properties of high density polyethylene/pinna nobilis polymer composites

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Keywords: high density polyethylene, pinna nobilis, UL 94, flammability, limit oxygen index, mechanical properties

Shell waste, with its high content of calcium carbonate (CaCO_3) plus organic matrix, has a potential to be used as a bio-filler. Shellfish shell is a shellfish aquaculture by-product that has been listed worldwide as one of the worst environmental problems, as it is difficult to dispose the shellfish shells. It reduces the liming value and renders the waste difficult to recycle to land. The shellfish shell contains about 95% calcium carbonate in the form of aragonite and calcite and 5% organic materials such as glycoproteins, polysaccharides, glycosaminoglycan, chitin and other proteins.

In this study, high density polyethylene matrix contributes to ground pinna nobilis powder is handled as 0, 10, 20, 30 and 40 wt% ratio will be mixed in the extruder. Mixture obtained from the extruder to be granulated and then the granules will be obtained as appropriate standard test sample of the injection molding machine. Flammability and mechanical tests will be applied such as UL94, limit oxygen index (LOI), tracking index (CTI), elasticity modulus, yield strength, tensile strength at break, % elongation, Izod impact strength, hardness, density. Also, SEM examination will be conducted to evaluate the microstructure of pinna nobilis particles as well as material distribution in these experiments.

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Manufacturing metal-polymer hybrid joints using a non-conventional method

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Keywords: Joining, Metal, Polymer, Hybrid joint

As the performance and efficiency of internal combustion engines cannot be increased infinitely, and standards and directives (for example directive 2000/53/EC for car wrecks) demand more and more strict supervision of industrial processes and pose ever stricter require-

ments (environmental friendliness, emission standards, recyclability/reusability of materials and structures, etc.), manufacturers in the automobile industry seek out novel materials to integrate and methods to use in manufacturing their vehicles. Polymer materials are widely used in vehicles, as their light weight combined with outstanding mechanical properties (for example in the case of fibre-reinforced polymers) and acceptable heat tolerance make them suitable as the base materials of various components: engine and passenger compartment elements, or even structural elements. There are multiple, conventional joining methods that are used to join these parts to each other and to the structural elements of vehicle, the most used of which is insert-based injection moulding and adhesive bonding. In recent years, multiple research initiatives were started to find methods that can possibly be used to manufacture hybrid metal-polymer joints in one step, so that expensive, labour-intensive and time-consuming subtasks and methods (such as surface preparation in the case of adhesive bonding) could be replaced with methods that can be highly automated (such as laser welding, or ultrasonic welding).

However, based on our literature review, the effect that the specific properties of the polymer material (wetting ability, shrinkage, degradation of the material and the temperature dependence of these properties) had on the hybrid joint was never researched, nor investigated. In our work, preliminary results are published describing the mechanical properties of hybrid steel-polymer joints, wherein the joints were created by heating steel pins in a furnace to different temperatures and then placing the steel pins on top of rectangular polymer sheets. The mechanical properties of the so-formed joints were tested using a special clamping fixture and a Zwick universal material testing machine in tensile testing mode.

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Synthesis and properties of free-standing graphene nanosheets at atmospheric pressure conditions

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Keywords: graphene, sheets, carbon, microwave, torch, discharge

Last decade two-dimensional carbon-based nanostructures such as graphene nanosheets, nanoribbons and carbon nanowalls have attracted much research interest, due to their remarkable properties combining high mechanical strength and flexibility, exceptional electronic and thermal conductivity, high carrier mobility and optical transparency. Potential application in different fields of science and technology include hydrogen and energy storage, reinforcement of polymer composites, electronics, electrochemical devices, or gas sensors.

Our deposition process reported in this work is based on injecting ethanol vapours through a microwave plasma environment, where decomposition of ethanol molecules takes place. We used microwave plasma torch discharge (2.45 GHz, ~200 W, Figure 1) operating at atmospheric pressure conditions to synthesize graphene nanosheets with rectangular shape and typical size of hundreds of nanometres. Argon flowing through the central channel of the nozzle was used as the working gas to ignite the discharge. Mixture consisting of ethanol vapours and additional argon flow was delivered into plasma environment by outer channel of the nozzle. Wide range of deposition conditions resulted in synthesis of different types of carbon material. However, mastering the deposition process by setting specific set of deposition conditions led to synthesis of graphene sheets (Figure 2). Created samples were analysed by scanning electron microscopy, Raman spectroscopy and X-ray photoelectron spectroscopy to probe the morphological, chemical and microstructural features of the produced material.

Figures



Figure 1: Microwave plasma torch

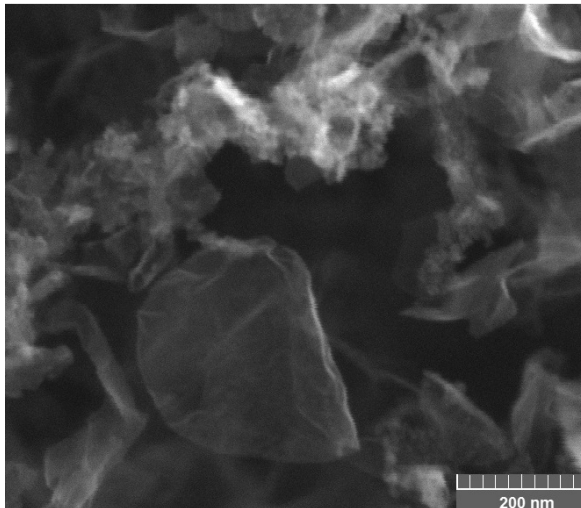


Figure 2: Synthesized graphene nanosheets

Acknowledgments

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Microstructure and mechanical properties of CP-Ti-1 processed by ECAP

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Keywords: ultrafine grained (UFG) titanium, equal channel angular pressing (ECAP), electron backscatter diffraction (EBSD), TEM, biomaterials

Biomaterials has become a crucial field of interest of many material scientists, biomedical engineers, pathologists and clinicians all over the world because these materials can improve the quality of human life and its expectancy. Biomaterials are artificial or natural materials, used to in the producing of implants, to replace the lost or diseased biological structure, to restore form and function [1]. Ultrafine grained titanium processed by equal channel angular pressing (ECAP) presents an obvious potential for biomedical applications because it provides the enhanced strength of titanium and doesn't contain any toxic alloying elements [2]. In this work electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) were applied to perform the microstructural analysis of grain size in commercial purity titanium (CP-Ti) Grade 1 in as-received state and after 4 and 6 routes of ECAP at 280 °C. The bars were 20 mm × 20 mm and 82.5 mm in length. The chemical composition of CP – Ti Grade 1 is shown in Table 1. The samples for investigation were cut from the central part of the bars and also from the near surface part. To provide a 3–D view of the microstructure, samples were taken from both longitudinal and transverse cross sections of as-received and deformed bars. A sub-microcrystalline structure of samples investigated after 6 ECAP routes exhibits promising mechanical properties, as determined by micro hardness measurements in different directions. The inverse pole figure map of the sample from the transverse central section of the bar is shown in Figure 1.

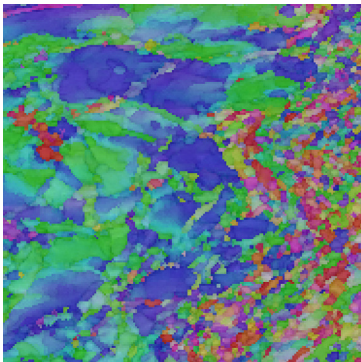


Figure 1 The IPF map of transverse section of CP – Ti – 1 processed by 6 routes of ECAP

Table 1 Chemical composition of CP – Ti – 1, wt.%

N	O	C	Fe	Al	V	Cr	Ti
0.004	0.068	0.008	0.03	0.01	0.01	0.01	Rest

Acknowledgments

This paper was prepared with a contribution of the projects No.LO1203 “Regional Materials and Technology Centre – Feasibility Programme”, “SP2018/70 Study of relationships between the technology and processing of advanced materials, their structural characteristics and utility properties” and “SP2018/60 Specific research in the metallurgical, materials and process engineering”.

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Morphological and structural characterization of thermally conductive HDPE/graphene nanocomposites

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Keywords: HDPE, graphene, nanocomposites, structure, morphology

There has been a growing interest in thermally conductive polymers such as thermal interface materials, electronics packaging and, plastic heat exchangers. Thermally conductive polymers are usually binary composites filled with high thermal conductivity additive in the matrix [1]. Carbon-based nanomaterials such as graphene nanoplatelets (GNPs) have been proposed as the next generation multifunctional nanofiller for the improvement of matrices [2]. A comparative study of GNPs at different diameters (5, 15, and 25 μm in diameter) for the melt mixed HDPE/graphene nanocomposites was carried out in order to investigate the influence of nanofillers on the morphological and structural properties of the matrix. The solid-state structure of nanocomposites was studied by X-Ray Diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). The high-resolution spectra of the C1s and O1s core levels were deconvoluted to separate the various bonds contributions. Fourier transform infrared spectroscopy (FTIR) measurements were performed to evaluate the chemical absorption, the thermodynamic interaction, as well as the crystallinity of graphene nanocomposites using the spectral bands of doublets. Finally, the microstructure of the nanocomposites was characterized using polarized optical microscopy (PLM), scanning electron microscopy (SEM), and high resolution transmission electron microscopy (HRTEM).

XRD results of HDPE/graphene nanocomposites confirm that the HDPE can exist in different polymorphs under atmospheric pressure such as the orthorhombic and the monoclinic forms. A sharp diffraction peak is also shown at $2\theta \sim 26.5^\circ$, which represents the diffraction of the (002) crystal plane of GNPs. XPS deconvolution of C (1s) reveals the presence of both sp^3 and sp^2 bonds because of the polymeric matrix and graphene fillers, respectively. FTIR spectra of HDPE/graphene nanocomposites suggests that the dispersion of GNPs in the HDPE matrix does not significantly alter the shape or the peak position of any of the absorption bands of polyethylene. PLM revealed that GNPs with smaller diameter promote the reduction of the spherulite mean size during crystallization compared to larger nanoplatelets. In addition, significant variations of GNPs distribution are shown in graphene nanocomposite with the larger diameter. GNPs with the larger diameter may present agglomerations which probably reduce its nucleation activity and support the development of larger spherulites. SEM and HRTEM analysis of the nanocomposites demonstrate that the GNPs with the smaller diameter are individually dispersed throughout the HDPE matrix. On the contrary, large aggregates can only be observed in HDPE nanocomposite with large GNPs.

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Application of bimodal powder process to harmonic structure design of SUS316L austenitic stainless steel

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Keywords: heterogeneous, homogeneous, mechanical properties, powder particle size control

Although SUS316L has been widely used for chemical plant and sea water related equipment due to the excellent combination of high temperature mechanical properties and corrosion resistance, the application is limited because of its low strength at room temperature.

In order to solve such a problem, a new concept of heterogeneous microstructural design called “Harmonic Structure” has been proposed. The Harmonic Structure (HS) is an extremely highly-controlled microstructure which consists of fine grains (Shell) with dispersed coarse grain (CG) islands. That is, Shell region is connected as a network structure surrounding the CG regions. The HS materials exhibited significantly better combination of strength and ductility, as compared to their homogeneous microstructure counterparts. The HS materi-

als were usually fabricated via severe plastic deformation – powder metallurgy (SPD-PM) process, which requires several ten hours mechanical milling procedure, forthemore it is difficult to control Shell/Core regions grain size and shell fraction.

In the present study, short term HS production process was applied. Different diameter powder particles, 140 μm and 7 μm , were mixed by Mechanical Milling and sintered by Spark Plasma Sintering to fabricate the Bimodal Powder Process (BPP) HS compacts. The BPP HS compacts of SUS316L was successfully produced. The BPP HS compacts exhibited good combination of strength and ductility, as compared to their homogeneous structure (Homo) counterparts. Fig.1 Shows EBSD grain size map of SUS316L BPP HS. The EBSD grain size map shows the Shell region (Grain size < 10 μm) established a continuous network surrounding Core regions. The BPP HS compact has Shell grain size of 6.0 μm and Core grain size of 22 μm . The Shell fraction is approximately 34%. Fig2. shows the tensile test results of these compacts. BPP HS compact shows both superior strength and ductility compared with Homo.

Figures

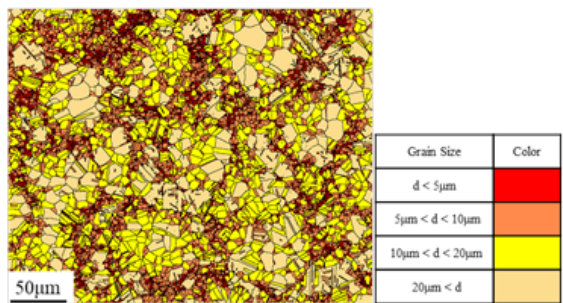


Fig1. EBSD grain size map of SUS316L BPP HS

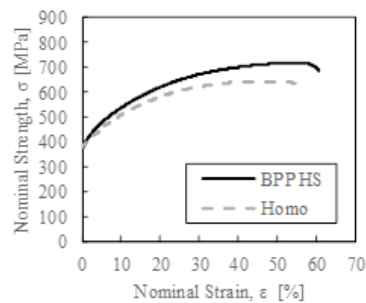


Fig2. Result of tensile test

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Fabrication and joining of the SiC fiber-reinforced SiC composites

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Keywords: SiC_f/SiC, Electrophoretic deposition, Joining, MAX phase

Owing to the wide convincing applications of the continuous SiC fiber-reinforced SiC composites (SiC_f/SiC) under extreme conditions, such as various structural components for high temperature, aerospace and nuclear reactor applications, significant effort is being paid to the fabrication of SiC_f/SiC. However, the main current limitation for producing a reliable SiC_f/SiC is in the lack of a suitable manufacturing technique, although a range of techniques have been developed to infiltrate the matrix phase into the fine voids of a fiber-preform to fabricate a dense and tough SiC_f/SiC. To develop a more suitable way for the infiltration of the matrix phase composed of SiC and sintering additive particles into a tightly woven TyrannoTM-SA3 SiC preform, therefore, both AC- and DC-electrophoretic deposition (EPD) combined with ultrasonication were examined in this study. On the other hand, the joining of SiC_f/SiC to produce a complicate structure for practical applications was also performed using a thin Ti₃AlC₂ MAX phase tape as a joining filler. After preparing a butt-joint configuration, the joining was performed by hot-pressing under 3.5 MPa applied pressure after inserting a thin Ti₃AlC₂ tape. In addition, an effort has been made subsequently to eliminate the joining filler layer via solid-state diffusion by exposing at high temperature. Experimental parameters, including joining temperature, holding time and filler thickness, were varied in a planned manner to obtain a sound SiC_f/SiC mechanical joint. The integrity of the joints was examined by microstructural observation in terms of elemental distribution, phase formation, evolution of the pores and cracks, and fractured surface after mechanical testing. Especially, the joining interface was indistinguishable for the SiC_f/SiC joined at 1900°C, while maintaining a good mechanical bonding between two neighboring base materials without presence of joining interlayer. The fracturing during the mechanical test occurred from the base material rather than the joining interface, indicating the the joining strength of 300 MPa approximately. These findings highlighted the possible elimination of joining interlayer in the joining of SiC using a Ti₃AlC₂ MAX phase, which might be an ideal for practical applications because the absence of joining region led to the preservation of excellent mechanical properties of the SiC base materials.



Fig 1. Images of the SiC_f/SiC having (a) planar 200×200×3 mm³, (b) tubular with 20mm diameter and 50 mm height fabricated by EPD combined with hot pressing, and (c) the joining SiC_f/SiC interface using Ti₃SiC₂ filler, showing the possible elimination of the joining filler by solid-state diffusion.

Acknowledgments

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A steel-like unalloyed ductile iron

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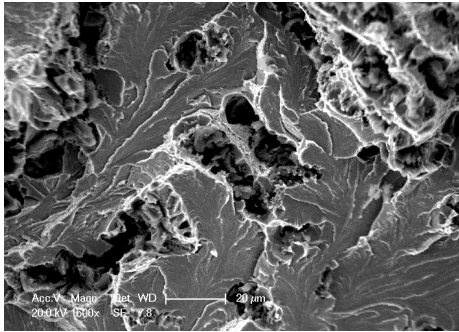
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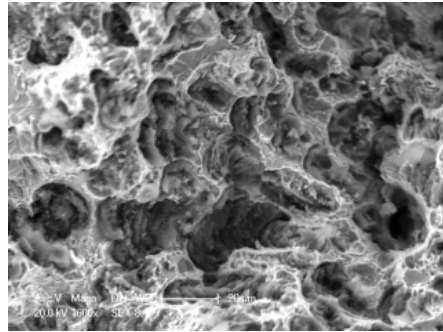
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Keywords: ductile iron, graphite morphology, multiple microstructure matrix, tensile strength

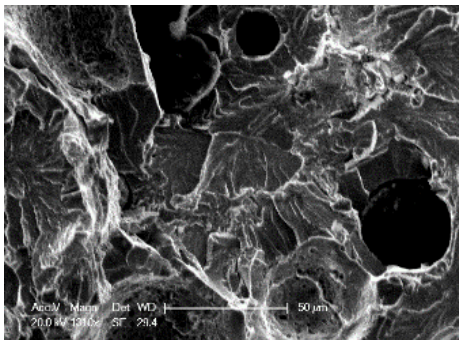
A key way of achieving sustainability of a product is to select a suitable manufacturing process by increasing the mechanical properties for improving materials efficiency and diminishing energy consumption. This work highlights a new process for direct manufacturing of ductile iron (DI) based on the innovative combined technology of quenching and partitioning and nanobainite at low temperature in high strength steels. In this process, a commercial unalloyed ductile iron is treated by initially rapid quenching after austenizing at 890°C for 20min and isothermal holding at 220°C for 4hrs. Additionally, four types of DIs with different matrix microstructures and graphite morphology are produced, namely ferrite and pearlite with vermicular graphite (sample A), ferrite and pearlite with nodular graphite (sample B), multiple microstructure consisting of prior martensite, bainitic ferrite and retained austenite with vermicular graphite (sample C) and multiple microstructure consisting of prior martensite, bainitic ferrite and retained austenite with nodular graphite (sample D). A maximum high tensile strength of more than 1600MPa and a hardness of HRC54 at an elongation in excess of 5% are achieved in sample D, which are comparable to those of a high strength carbon alloy steel. The results reveal that the synergistic strengthening effect (SSE) of multiple structure in the matrix and graphite morphology is the key for the excellent mechanical properties. The new developed technology provides a guidance on how to produce unique steel-like ductile iron with both high level of strength and reasonable toughness together.

Figure: SEM fractographs of different samples after tensile test

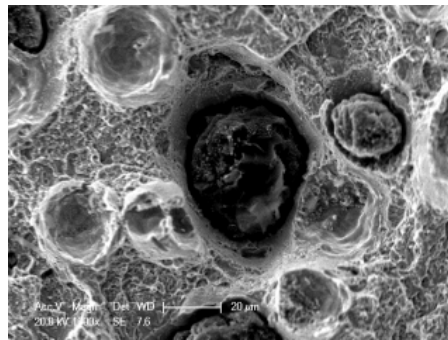
Sample A



Sample B



Sample C



Sample D

Table: Chemical composition of the ductile cast iron (wt.%)

C	Si	Mn	P	S
3.5	2.5	0.2	<0.0015	<0.0015

Design and Processing

Oral

Use of raw spelt hull (husk) as natural aggregate in a lightweight insulating concrete

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Keywords: spelt hulls, hemp hurds, plant-derived aggregates, lime binder, bio-based concrete, thermal conductivity, compressive strength

In developed countries, the global contribution from buildings towards energy consumption has risen sharply in recent decades and has even exceeded the other major sectors in EU and USA: industry and transportation. In this context, the development of eco-friendly materials containing locally available vegetable resources is a hot topic and interest has been paid to a wide range of agricultural waste which could potentially be used for this scope. The final goal is to valorise the entire biomass, in the spirit of the zero-waste concept. Bio-based building materials are already well established on the building market. One of the most widely used ones is Lime Hemp Concrete (LHC) which is made out of hemp hurd mixed with a lime-based binder. This study deals with the development of a similar insulating lime-based concrete in which spelt hulls are used instead of hemp hurd. From now on, this novel material is called Lime Spelt Concrete (LSC).

Having examined physical and structural characteristics of plant aggregates, specimens made out of whole spelt hulls and a lime-based binder were manufactured by mixing and mechanical tamping. Both thermal and mechanical properties of the final concrete materials were studied and the results were compared to those obtained for LHC manufactured with the same process.

The three different mixtures used and the main results are presented in Table 1. Thermal measurements show that LSC is comparable to LHC in terms of thermal insulation. These results are strongly linked not only to the concrete's apparent dry density, but also to its total porosity which values are comparable for LSC and LHC. Compression test results following 30 days of aging show that the mechanical strength of LSC is in all cases lower than that of LHC. These mechanical performances are possibly justified by considering the interface between the natural particles and binder. In LSC the interface should be influenced by the morphological characteristics of spelt hulls for which the bond is maybe only effective on their external convex surface. Before 1% strain, during compressive test, the microstructure of the lime-based binder plays a significant role. The results suggest that the setting and the hardening of the lime-based binder is less efficient in LSC. These observations could possibly be connected to a high amount of sugar extractives and hemicellulose in spelt hull which solubilized in the matrix and disrupted the hydration reactions. In fact, the cumulative amount of hemicelluloses found in spelt hulls is about two times higher than those in hemp hurd aggregates.

Table 1: Mix proportions and main results of the study

Concrete Name	Binder on aggregates mass ratio	Dry apparent density (kg/m ³)	Dry thermal conductivity (W/m K)	Compressive strength (MPa)
Lime Hemp Concrete (LHC)	1	200 ± 7	0.058 ± 0.005	0.16 ± 0.02
	2	286 ± 10	0.074 ± 0.003	0.14 ± 0.01
	2.5	326 ± 11	0.089 ± 0.005	0.18 ± 0.02
Lime spelt hull Concrete (LSC)	1	306 ± 10	0.073 ± 0.003	0.07 ± 0.01
	2	373 ± 13	0.076 ± 0.001	0.11 ± 0.01
	2.5	380 ± 13	0.083 ± 0.001	0.07 ± 0.01

Thermoplastic overmolding onto in-situ polymerization-based reinforced polyamides

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Keywords: overmolding, in-situ polymerization, polyamide

Injection molding – today’s most widely used plastic forming technology – is gaining popularity fast in the automotive industry, too. In addition to conventional technologies, more and more special technologies are gaining ground in the manufacturing of automotive parts, including gas- and water-assisted injection molding, bright surface molding and also reactive technologies. Reactive injection molding (RIM) and resin transfer molding (RTM), however, cannot be integrated into car factory assembly lines due to their considerably longer cycle times. In spite of this, more and more and larger and larger parts are manufactured with these technologies, for example, the whole body of the BMW i3 [1]. The technology can produce a functional part as a whole composite structure but recycling is a problem because these technologies produce cross-linked structures. For this reason, much research has been done in the past few years into the practical application of in-situ polymerization. The technology produces polyamide structures of a thermoplastic matrix [2]. The advantage of the technology is that the mold is filled with a low viscosity oligomer, which can also impregnate complex textile systems, then polymerization takes place in the mold, too [3].

My research goal is to solve the practical problems of in-situ polymerization. First, I designed a complex product and the mold for it. I impregnated the textile structure with a very low viscosity oligomer, then in the mold, I overmolded ribs from polyamide 6 onto this complex 3d surface geometry. The novel technology and the complex part generated problems that had to be solved before the mold was manufactured. To tackle these problems, first I designed and produced an experimental mold with which I injection molded ribs onto a polyamide plate which was produced in an earlier stage with in-situ polymerization. At this stage, I examined the overmolding performance of the ribs as a function of technological pa-

rameters. I examined and analyzed the effect of the state of the preform and the reinforcement content of the overmolded component. Based on these preliminary experiments, I designed a more complex experimental mold, which facilitated the analysis of local welding with the help of sensors inserted in the mold. In order to locally improve the quality of welding, I designed an injection compression function for the mold, with which local pressure can also produce a local pressure increase further away from the injection location. This function and the testing of this function was necessary because of the original large part, which will require ribs of good joint quality without warpage further away from the melt entrance, too.

My work combines large series, in-situ polymerization and conventional injection molding technologies, by exploiting the advantages of both technologies.

Acknowledgments

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The recrystallization behavior of extruded Mg-Zn alloys with Rare earths addition.

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Keywords: Extrusion, microstructure, texture, recrystallization

This research work focusses on the investigation of the microstructure and texture development of three different extruded Mg-Zn based alloys subjected to isochronal recrystallization annealing treatments. Additional elements Ce, La and Gd were used as microstructure and texture modifiers. Extrusion of round bars was carried out at 350°C and 0,1mm/s with an extrusion ratio of 25. These parameter settings allow to achieve widely non-recrystallized microstructures after extrusion as the initial material of this study. Samples from each alloy were annealed at the extrusion temperature for different times in order to increase progressively the fraction of the recrystallized microstructure. Optical microscopy (OM), X-Ray diffraction as well as electron-backscattered diffraction (EBSD) were applied for the characterization of the recrystallization process.

The initial microstructure consisted of large elongated unrecrystallized grains and fine recrystallized grains forming a necklace structure. During static recrystallization, Ce and Gd delayed further the recrystallization leading to the coexistence of equiaxed grains and the subsistence of elongated grains. Texture analysis revealed that all the alloys developed a strong prismatic fiber type texture, which is associated to the elongated grains in the as-extruded condition. During static recrystallization, Ce and La are more potent elements in retain the microstructure and texture development with characteristic texture components such as $[-11-21]$ than Gd, in which such a texture component disappeared. The growth of recrystallized grains was evident with the increase of time heat treatment with a concomitant weakening of the texture. The present results suggest that both, the alloying elements and annealing time have a different effect in the recrystallization behavior of Mg-Zn based alloys. This indicates that a careful control of the alloy composition in combination with proper annealing can help to control in a systematic way the microstructure and related properties e.g. mechanical properties.

Effect of printing direction on the mechanical properties of ABS compound manufactured by droplet deposition

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Keywords: Polymer Additive Manufacturing (PAM), Mechanical properties, Printing path

Polymer additive manufacturing (PAM) is known as an effective mean to manufacture 3D dimensional pieces directly from a computer-aided design model in a layer-by-layer style. Recently, a new additive manufacturing technology has been integrated into the Freeformer-Arburg machine [1]. It was inspired by injection molding technology and creates plastic parts using layers build up from tiny droplets of the polymer. The use of PAM components is not well accepted for load-carrying parts under static and dynamic conditions due to many processing parameters affecting the part properties [2–3]. The aim of this study is to characterize the manufactured polymer parts by perceiving how the individual PAM process parameters, particularly the printing path, might impact the performance of manufactured products under static and dynamic conditions. The quasi-static mechanical properties of samples are evaluated via uniaxial tensile tests in terms of Young modulus, strength and elongation to fracture. Besides, dynamic mechanical analysis (DMA) are performed in the single cantilever bending mode with sweeping temperature at different frequencies, providing properties such as maximum storage modulus, maximum loss modulus, peak of tan delta and flexural modulus. These properties are compared to those of the same ABS raw material processed by injection molding, considered in this study as a reference. Additionally, SEM microstructural analyses of tested samples have been performed to better understand the relationship between the manufacturing process and the final mechanical properties.

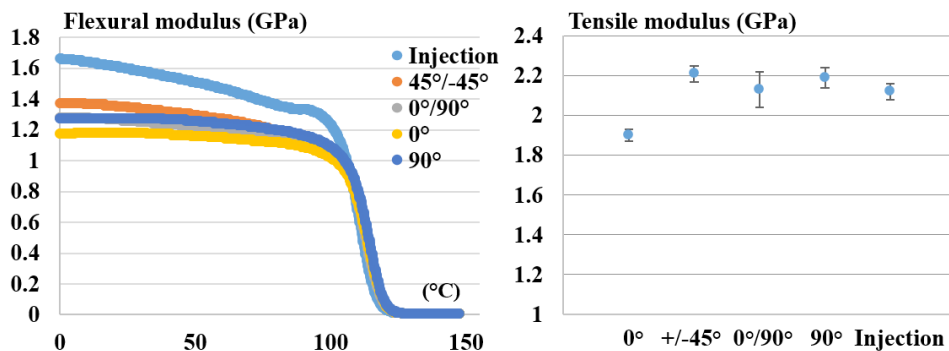


Figure 1: Comparison of Mechanical Properties Obtained by Two Techniques

The results have shown that the printing path significantly influences the performance of manufactured samples under static and dynamic conditions (Figure 1). It could be noted that the criss-cross printed specimens [45°/-45°] exhibit highest mechanical properties (tensile and flexural moduli) amongst all of the printed samples.

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Magnesium alloys containing RE considered as biodegradable materials

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Keywords: magnesium, biomaterial, mechanical properties, corrosion

Magnesium and its alloys make their place in the field of medicine as materials for biodegradable implants. Biodegradable materials should gradually degrade in a body after fulfilling their function without the necessity of the second operation to remove them. Unlike most of the materials designed for permanent implants, they have mechanical properties very similar to the bone tissue. Therefore, the problem with the stress shielding effect which occurs for materials with the materials with the high modulus of elasticity is eliminated. The greatest disadvantage of magnesium is its high corrosion rate which may lead to implant failure. The corrosion rate is often reduced by alloying elements. The most useful alloying elements of magnesium alloys are the rare earth elements. They improve mechanical properties signifi-

cantly even in a small amount. Even corrosion rate is usually much better compared to other alloys. However, it highly depends on in which form the alloying elements are present in the structure. Intermetallic phases usually work as cathodic places and may cause unwanted galvanic corrosion. Therefore the fine and homogeneously dispersed intermetallic phases are preferred in the structure. Such structure is usually obtained after thermomechanical processing, for example, by extrusion. During this process, individual grains are deformed and the dynamic recrystallization occurs. Intermetallic phases may be partially dissolved, precipitated or redistributed. They also work as nuclei for the recrystallization process. The knowledge of the impact of extrusion on the individual is therefore essential for their application.

In this work, four magnesium alloys (Mg, Mg-4Y-3RE, Mg-2Y-1Zn, Mg-3Nd-0.5Zn) were prepared by extrusion. Structure, mechanical and corrosion properties were compared. The pros and cons of individual alloys were discussed. Out of the obtained results, the WE43 alloy seems to exert the best mechanical and corrosion properties. The ultimate tensile yield strength was 300 MPa, while the corrosion rate was about 0.4 mg/cm²/day.

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Effects of particles sizes distribution on sintering behaviour and properties of hydroxyapatite bioceramic

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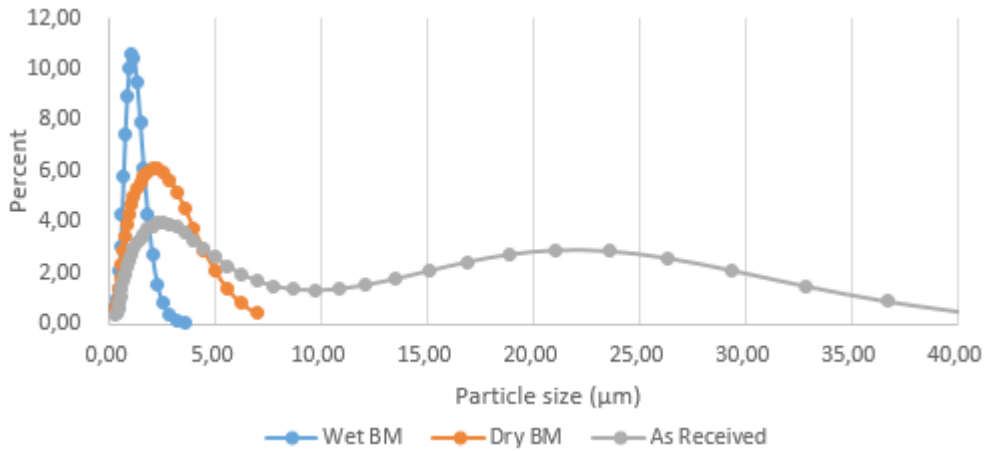
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Keywords: hydroxyapatite, sintering, dilatometry, microstructure, mechanical properties

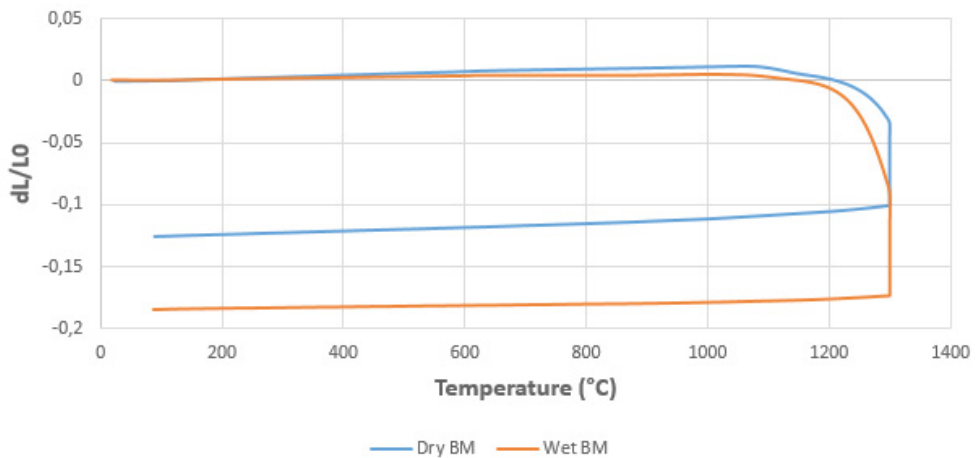
Hydroxyapatite powders with different particles sizes distributions was achieved by changing the ball milling conditions, from a commercial type of the ceramic. Dilatometry analyses of the resulted powders showed increase in sinterability with reducing the agglomerates sizes. It was also differences in the combination of final phases detected by X-ray diffraction method.

Effect of different heating rates on the sintering of the powders, also was studied using both dilatometry and direct heating in furnace. It was found that heating rate has a significant effect on grain growth and final densification and therefore, on the mechanical strength of the ceramic. Gelcast samples with near to complete densification and compression strength of around 400 MPa was obtained in optimum combination of particle sizes and sintering condition.

Figures



Effect of ball milling condition on agglomeration sizes



Effect of ball milling condition on sintering behaviour

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White cast iron reinforced with structural ceramics for wear resistance applications

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Keywords: Metal-ceramic bonding, white cast iron, wear-resistant cast parts

The metal matrix composites (MMCs) have been a subject of intense research and development essentially in the aerospace and automotive applications [1]. Comparatively with non-reinforced metals, the MMCs reinforced with carbides exhibit high tensile strength, improved elastic modulus and wear resistance, as well as fatigue and creep resistance. Due to the low processing costs and increased castability, locally carbide-reinforced ferrous alloys are promising materials for wear resistance applications [2–4]. There is currently, a considerable interest in developing MMCs with iron or steel matrices [5]. The attention has been directed towards the in-situ formation of ceramic particles during the casting process. The combination of self-propagating high-temperature synthesis (SHS) with the traditional casting process results in a potential process for achieving cast iron and steel MMCs components reinforced with ceramic particles. This technology is particularly suitable for the production of near-net-shape components of complex geometry for a wide range of wear applications due to a combination of wear resistance (conferred by the ceramic reinforcement) with toughness (conferred by the metal ferrous matrix). The brazing technique can prove an alternative in which the wear resistant reinforcement (WC-Co) is applied locally (where wear resistance is required) to the cast components surface (substrate) with braze alloys such as copper, nickel and silver based alloys, and multi-interlayers (Ti/Ni/Ti) [6–8].

This work is intended to develop metallic matrix composites reinforced with carbides, such as TiC and WC, through the introduction of green and sintered ceramic powder compacts in the mould cavity. The brazing technique is also applied to bond hardmetal (WC-Co) to white cast iron. The main goal of this research is understanding the bonding mechanisms established between the metal and the ceramic using different bonding processes. For this purpose, the microstructure along the cross-section of the bonding interfaces is characterized by SEM/EDS. The bonding mechanical properties are controlled by microhardness and shear tests.

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Preparation of zinc-based oxide ceramic nanomaterials by needle-less electrospinning

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Oxide ceramics like zinc and aluminum oxides, titanium and tin dioxides and many others are widely used as materials for structural and refractory applications, sensitive elements of sensors – especially gas sensors, catalysts supports and catalysts for chemical production, photocatalysts in dye-sensitized solar cells, and even water treatment. Due to the specific properties of nanofibers, such as high surface-area-to-volume ratio, high porosity, high substances loading capability and appreciable mechanical strength, these materials in nanofibrous form shows better characteristics and can be used in advanced composite materials and for specific applications.

In this paper needleless electrospinning is used for preparation of oxide ceramic nano/microfibers from polymer precursor blends. Because of its simplicity and flexibility the properties of prepared nanofibers made of above mentioned materials can be easily tuned to even some specific requirements, like dye-sensitized solar cells or sensors. Preparation route is simple and robust: organic or inorganic precursors are mixing with a polymer and appropriate solvent and then a blend is electrospun to form a micro or even nanofibers. Then composite

precursor fibers are being processed by conventional calcination, depending on the required properties, to receive ceramics. By simply changing the heat treatment atmosphere from oxidizing to inert, pure oxides (in air) or carbon-based composites can be obtained. Also by varying the treatment temperature, materials with different phase composition can be prepared. This route were used for preparation of wide range of oxide ceramic pure and doped materials – ZnO fibers, hybrid structures and composites (based on ZnO, Al₂O₃, SnO₂, TiO₂, C/TiO₂, CuO, Nb₂O₅ and others).

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Study of chromium nitride precipitation in a cast super duplex stainless steel

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Keywords: super duplex stainless steel, chromium nitrides precipitation, annealing temperature

Cast duplex stainless steels (DSS) offer an excellent combination of high mechanical properties and corrosion resistance in highly corrosive environments. The chemical composition of standard cast DSS grades can be modified by alloying additions of Mo, W or N, to enhance the pitting corrosion resistance. It is important to underline the particular case of N, that is also a austenite stabilizer [1, 2], which is able to partially replace some of the Ni content, by small additions up to to 0.4 wt%. In addition to the control of chemical composition, the thermal processing conditions are critically important in maximizing the desirable final characteristics. The annealing heat treatment is a necessary step of the process because it enables to adjust the volume fractions of ferrite (δ) and austenite (γ) close to a ratio of 1:1, which is essential to maximize the mechanical properties and corrosion resistance. In contrast to sigma (σ) phase and chi (χ) phase, which can be prevented by selection of cooling rates faster than the critical cooling rates for their precipitation, the chromium nitrides precipitate inevitably during quenching as the solubility limit of N is exceeded in the δ -phase, which is five times lower than solubility limit of N in γ -phase [3, 4]. As a rule, the extent of chromium nitrides precipitation depends on N content [5], however the heat treatment conditions (temperature, holding time and cooling rate) can modify the precipitation behavior. Thus, the present work investigates the influence of a short isothermal stage in the temperature range of 1040 °C to 1085 °C on the chromium nitrides precipitation of a cast 25Cr-7Mo-Ni-N duplex stainless steel. A set of samples were solubilized at temperature higher than 1100 °C and isothermally held at the temperature range of interest for a short stage of 15 minutes. In order to detect and measure the volume fraction of δ , γ and chromium nitrides, optical microscopy and scanning electron microscopy were employed. For the OM, the heat treated samples were pre-etched

by electrolytic etching in oxalic acid solution, with controlled current density (1 A/cm²) for 45 seconds.

The chromium nitrides precipitated within δ grains were successfully revealed with purpose metallographic procedure. Furthermore, the images collected during optical analysis permitted to accurately measure the volume fraction of the chromium nitrides, through the application of a threshold algorithm, able to segment the chromium nitrides precipitates from the matrix composed by δ -phase and γ -phase.

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Studies on microstructure, and mechanical properties of aluminium foam prepared by melting and casting routes

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Keywords: aluminium foam, melting and casting, wear, microhardness, compressive strength

In the present study, attempts have been made to develop foam aluminium using different space holders and gassifiers by melting and casting technique. Both pure aluminium and aluminium based alloy foam have been developed. Cenosphere and calcium carbonate have been applied as space holders and gassifiers for the development of aluminium foam. The main process variables were composition and mass fraction of space holders or gassifiers added for the development of foam, temperature of the bath, duration of melting and stirring velocity. A detailed investigation of the effect of process parameters on the microstructures and phases were undertaken to optimize the processing zone for the development of foam. The mechanical properties in terms of macro/microhardness, wear resistance and compressive strength have been evaluated in details. Finally, the deformation behaviour and wear mechanism have been established.

An Assessment of the effect of machining and surface microstructure on fatigue performance of aerospace titanium alloy Ti-6Al-2Sn-4Zr-6Mo

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Aeroengine parts are subjected to very demanding working conditions and strict standards, and it is key to ensure the structural integrity of critical parts like compressor disks during the total life of the component. The aim of this project is to understand how the features produced by upstream processes like forging, and the features produced by different machining conditions titanium alloy (Ti-6Al-2Sn-4Zr-6Mo), affect the fatigue life of the part.

This study is critical for ensuring the maximum quality of the compressor disks. Understanding the effects of machining and texture in fatigue life and how the different microstructural features are induced in the surface the material, makes possible to redesign the machining process to manufacture the disk under the optimal machining parameters and in a shorter period, without compromising the highest quality and fulfilling all mechanical specifications required.

Laser printing of silver nanoparticle inks: Simulation and high speed visualization of the jet dynamics

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Keywords: Silver nanoparticles inks, LIFT, jet dynamics, High speed visualization

Current technological trends in the field of micro-electronics have highlighted the requirement to use cost-effective techniques for the precise deposition of highly resolved features. The use of metal nanoparticle inks and their ability to be directly printed on flexible substrates has given great impetus in this direction.

Laser-induced forward transfer (LIFT) is one of the very promising direct printing techniques and has already been applied for the direct printing of devices and components. However, in order to improve the process reproducibility and printing resolution, further research has to be conducted, regarding the rheological characteristics of the printable fluids and their jetting dynamics. In this work, we employ time-resolved and high-speed imaging in order to

investigate the formation and expansion of the liquid bubble, as well as the liquid jet's propagation, over a wide range of inks' viscosities. Velocity and acceleration of the liquid jet, as well as its dynamics, were deduced by calculating its temporal and spatial evolution, while at the same time a phenomenological analysis of the jet's propagation was carried out from which conclusions about the behavior of varying viscosity inks were drawn.

Furthermore, a computational model is utilized in order to gain more insight on the transfer mechanisms of the process. The simulation predictions are validated against experimental results, being in good accordance with the latter ones.

Figures

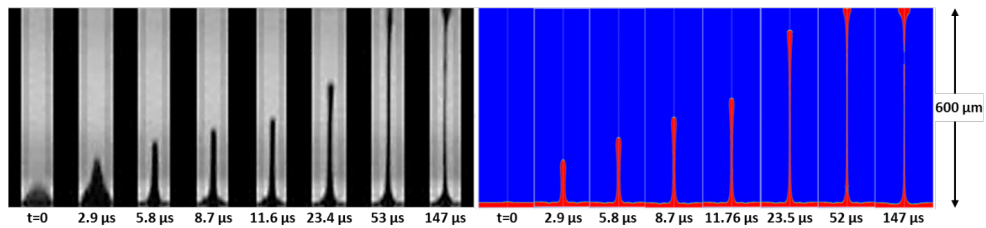


Figure 1: Silver nanoparticle ink jet's propagation during LIFT printing process. Comparison of experimental and simulation results.

Acknowledgments

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Poster

Along the process chain of AlZnMg(Zr) alloys

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Keywords: High-strength aluminium alloys, homogenization, dispersoids, extrudability

High-strength aluminium alloys of the 7xxx-series (AlZnMg(Zr)) are used commonly in the aircraft and sport industry. Increasingly they are also applied in automobiles as b-pillars or bumper systems. Due to their high strength and crashworthiness, they can replace steel components and make cars lighter. However, the extrusion of the material is challenging because of high press forces. To improve the extrudability, research on the material microstructure evolution along the process chain is necessary. A promising approach is the optimization of the homogenization heat treatment of the as-cast material. Here the influence of different homogenization heat treatments on precipitation and hot flow stress was investigated. It was found that the extrudability depends strongly on the forming temperatures.

Acknowledgments

The author thanks the AIT Austrian Institute of Technology and Hammerer Aluminium Industries (HAI) for the idea, the material and the permission to present some of the results at FEMS Junior EUROMAT 2018. This work was supported by the Austrian Research Promotion Agency (FFG).

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Banded Heat Affected Zone (HAZ) microstructures in LMD INCONEL718 repairs

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Keywords: Nickel alloys, Laser Metal Deposition, Digital Image Correlation

Laser Metal Deposition (LMD) is a low heat input additive technology that offers advantages for the repair of high value aerospace components. Banded microstructures often arise in LMD manufactured materials and their appearance is associated with the overlap of Heat Affected Zones (HAZ) developed during each deposition pass. The purpose of this work was to determine the effect of the deposition parameters on their occurrence in LMD manufactured INCONEL718 samples, especially in terms of their thickness, microstructure, chemistry, and hardness. Digital Image Correlation techniques were used to map the strain over the area of samples sourced from different build heights. Differential Scanning Calorimetry (DSC) was also performed at different build heights, on samples subjected to different post weld heat treatments and the results were used to quantify the amount of strengthening precipitates present in each sample. Hardness maps were then produced to demonstrate the mechanical heterogeneity of the material. Additionally, the microstructure was assessed using Scanning and Transmission Electron Microscopy, to further focus on the microstructural banding.

Preparation and characterization of organic-inorganic complexes modified epoxy resins

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Keywords: ultra-sonicated, epoxy resin, agar, lysine, barium nitrate, cadmium nitrate, zirconium nitrate

Relying on biotechnological substances, fundamental chemistry and material science we have developed some composite materials based on both physical and chemical properties of organic and inorganic substances that we use. The study aims to investigate an ultra-sonic method of dispersion in order to form polymeric materials modified with agar, lysine and inorganic compounds and, also characterization of obtained materials. Agar and lysine were used as organic modifying agents that could form organic-inorganic complexes with certain nitrates. Both organic and inorganic compounds (in determined amounts) were added to the epoxy resin and dispersed in certain conditions to obtain a doping level of: one molecule of

agar at one barium atom; two molecules of agar at three cadmium atoms; one molecule of agar at two zirconium atoms and the same but replacing agar with lysine. Ultra-sonication represents the key step of the present research in order to disperse the organic and inorganic complex into epoxy resin. Better mixing between composite components can provide higher strength and stiffness whereas poor mixing is seen to decrease those properties. The structure of these materials is unique and their characterization is of a great interest. The results of this study might be of great interest in nano-structuring an epoxy resin by developing chemical reactions into the pre-polymer mixture and placing together the barium, cadmium and zirconium atoms (as in the case of gel method for nanoparticle preparation) using their nitrates.

Acknowledgments

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Influence of composition and processing on strain ageing in high strength pearlitic wires

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Strain ageing is responsible for an undesirable change of mechanical properties in pearlitic wires. The project focuses on high carbon (~0.80 wt.%) wires. Dissolution of cementite contributes to the large increase in interstitial carbon atoms found in the ferrite during service. Interstitial carbon atoms interact with dislocation substructures in the ferrite to produce an increase in hardness, and an associated reduction in toughness. This can lead to wires failing prematurely in service. The project aims to reduce the effect of strain ageing via the addition of alloying elements or processing parameters. The project also aims to increase current understanding of the mechanisms responsible for strain ageing.

3D-printed droplet-microreactor for chemical synthesis of nanoparticles

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Keywords: microreactor, droplet, 3D printing, nanoparticles

The technology of highly monodisperse droplets is widely used in a range of chemistry related applications including synthesis of polymer beads, nanoparticle synthesis, precisely controlled crystallization or multiphase reactions. Droplet microfluidics offers an excellent platform to conduct a reactions where droplets act as isolated micro/nano-reactors. This method provides several advantages compare to batch or continuous-flow processes used in chemical synthesis such as proper mixing of reactants by recirculation of fluids within the droplet, low reagents consumption, perfect reaction temperature control due to large surface-to-volume ratio and safe operation of microreactor. Traditionally used techniques for manufacturing of droplet microfluidics devices such as dry or wet lithography, micromilling, hot stamping or micro-injection molding are time consuming, require precision treatment and final product is usually very expensive. However, 3D-printing has been seriously improved in recent years and can be used for fabrication of very precise droplet microfluidic reaction devices with a fraction of price and time compare to traditional methods. We apply FDM (fused deposition modeling) 3D printing technology to fabricate microfluidic device with different geometry of chips, capable to performing reactions in droplets in this work. This microfluidic device was successfully tested for reactions where separate droplets with different reagents were merged in a controlled environment to induce reactions inside a fused droplet to produce monodisperse nanoparticles. Results showed in this work points out large potential applications of 3D-printing in a production of droplet based microreactors.

The effect of thermomechanical processing on the microstructural evolution of nickel-based alloys

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Keywords: nickel alloys, thermomechanical processing, SEM, TEM

Nickel-based alloys, such as Alloy 625, are often used in demanding environments due to their enhanced corrosion resistance and strength at elevated temperatures [1]. For these reasons they can find applications in advanced nuclear power systems; however, processing

these alloys in such a way as to maintain consistent and uniform properties is often challenging. This is particularly the case in temperature regimes of second phase precipitation, which should ideally be avoided [2]. Therefore, the objective of this project is to determine the thermomechanical processing conditions that will produce nickel-based alloys with consistent, predictable and desirable properties. Variables being considered include temperature, strain and strain rate.

Processing is being simulated with laboratory scale Plane Strain Compression (PSC) deformation at the University of Sheffield. PSC (schematically shown in Fig 1) allows for flow-stress data to be obtained [3]. Ultimately, using PSC, a promising set of processing conditions may be obtained and applied to a wide range of nickel alloys. In this presentation, baseline characterisation performed at the University of Manchester (including SEM, TEM and Vickers Hardness) is shown for the as-received material supplied by Rolls-Royce plc. Additionally, initial results from PSC will be presented to illustrate how deformation conditions can influence the resulting microstructures.

Figures

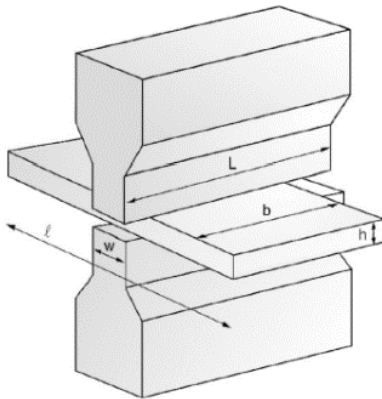


Fig 1. Schematic diagram of a plane strain compression test. Adapted from ref [4].

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Electrospun silver coated polyacrylonitrile nanomembranes for water filtration applications

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Natural scarcity of drinking-water as well as the contamination of water sources in under-developed countries are serious problems that need immediate low-tech and low-cost solutions. Polyacrylonitrile (PAN) fibrous membranes have been widely researched recently for their potential application in the preparation of water filters. [1-3] The beauty of the filters produced offer a vast choice of membrane fibre diameters that can be varied based on the choice of application. [3] [1,2] As compared to other techniques, electrospinning offers various advantages like being fast and an inexpensive way to produce membranes. This study aims to present the potential of polyacrylonitrile (PAN) fibrous membranes coated with silver nanoparticles (AgNP) as an affordable filter membrane. The membranes were prepared by electrospinning PAN solution and treated in hydroxylamine (NH₂OH) aqueous solution to form –C(NH₂)N–OH groups that were used for functionalization (Ag⁺ ions) of the membrane. The coordinated silver ions were then converted to silver nanoparticles (AgNP). The functionalized membrane's morphologies, structure, water permeability, antimicrobial effects (using *Escherichia coli*) and particulate filtration capabilities were studied. The study verified that the membrane demonstrated a 100% reduction for gram-negative bacteria with an effective filtration rate 8.0 ml/cm²min. Furthermore, the membrane was able to eliminate 61.4% of latex beads as small as 50 nm and over 81.2% of the 2 µm beads via gravity filtration. This study demonstrated PAN-AgNP membranes as an economical approach to develop antimicrobial membranes that can be employed for filtration of water in underdeveloped countries.

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Microstructure of additive produced parts:

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Due to the advantages of additive manufacturing (AM), it has been increasingly integrated into many industrial sectors.

The application of AM materials for safety-critical parts requires the detailed knowledge about their microstructure stability under thermo-mechanical or mechanical load and knowledge on ageing process mechanisms. Ageing processes are characterized by change of the material microstructure that is to be initially investigated. This work deals with the investigation of 316L stainless steel manufactured by selective laser melting (SLM) and laser powder cladding (LPC). Describing parameters must be defined and applied on the microstructure of these materials in their initial state and after loads were applied. The findings of this work form the basis for the investigation of AM material ageing.

The influence of severe plastic deformation and magnesium content on the corrosion resistance of Al-Mg model alloys

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Keywords: corrosion resistance, hydroextrusion, severe plastic deformation, aluminum alloys, Al – Mg alloys

Two model Al – Mg alloys, containing 1 and 7,5% wt. of magnesium, were subjected to severe plastic deformation by means of hydroextrusion. This method allows to uniformly deform the material, for during the process a surrounding pressurized liquid is pushing it through the die, resulting in the extrusion. The diameter of both materials was reduced from 20 to 10 mm, giving the deformation degree equal to 1,39.

In order to determine the influence of deformation on the size and shape of grains the microstructure of both alloys in an initial state and after extrusion were examined with the use of light microscopy (LM). The overall influence of Mg content on the microstructure was also considered. Corrosion resistance of the materials was measured in a standard three-electrode setup, consisting of an examined sample as a working electrode, a Pt counter electrode and an Ag/AgCl reference electrode. All the tests were carried out in 3,5% wt. sodium chloride water based solution in an ambient temperature. Open circuit potential and potentiodynamic polarization behavior of materials was determined, characteristic corrosion parameters were calculated by Tafel extrapolation method. Surface of the samples after corrosion tests was further examined with the use of scanning electron microscopy (SEM), which allowed to determine the extent and character of its degradation.

The microstructure of both materials significantly changed after the hydroextrusion process. Equiaxial, uniformly distributed grains were replaced with an underdeveloped and non-uniform structure having slightly reduced grain size. Moreover, the alloy with a higher content of magnesium in an initial state and after deformation had a noticeably lower grain size, which could have an influence on its corrosion resistance. Electrochemical measurements and SEM observations revealed that all analyzed materials underwent pitting corrosion. The passive films which were formed on the samples, protected the substrative materials from the detrimental impact of chloride ions up to the pitting potential when the local corrosion was observed. It was also noticed that the variation of magnesium amount and the deformation degree strongly influenced the corrosion behavior in analyzed conditions.

Manufacturing and Recycling

Oral

Microstructure and corrosion resistance in TIG welded joint of Hastelloy C-22 alloy

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Keywords: TIG welding, Hastelloy, Microstructure, SEM, Corrosion

Nickel alloys are used because of their corrosion resistance, heat resistance and high and low temperature properties. In this article types of nickel-chromium-molybdenum alloys are described and guidance is given on welding processes and techniques which can be used in fabricating nickel alloy components without impairing their corrosion or microstructure properties. Tungsten Inert Gas of 7 mm thick nickel based alloy HASTELLOY C-22 has been conducted in butt joint configuration. The need for complex and detailed preparation of plates prior the welding, and welding discipline has been presented. Microstructure of the weld metal and heat affected zone was examined using scanning electron microscope. Composition of the weld metal and heat affected zone was examined using X-ray fluorescence analyser. Comparative electrochemical corrosion tests were conducted to determine the influence of welding on corrosion resistance properties.

Effect of needle characteristic on fibrous PEO produced by electrospinning

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The technique of electrospinning have been researched for several decades. All parameters almost have been investigated in the past years, e.g. solution parameters, process parameters and environmental conditions. Among the solution parameters, the viscosity of the polymer solution is an extremely important factor for fiber formation and morphology. In general, however, viscosity of the polymer solution is mostly controlled by the solution concentration or by the molecular weight of the polymer. In this paper, it is shown that the needle characteristic (the length and diameter of needle) can have an influence on the fiber morphology. This is because that when the polymer solution is flowing through the cylindrical needle, it is subjected to a shear load that depends on the needle geometry. Polyethylene-oxide (PEO) with a molecular weight of 400,000 g/mol was dissolved in a mixture of ethanol and water with a proportion of 1:3. The relationship between the viscosity of the polymer solution and shear rate was characterized by a plate-plate rheometer. The solution was electrospun and the sample obtained on the collector was examined by scanning electron microscopy, the fiber diameter determination was carried out by image processing software. A model describing how the needle characteristic determines the shear rate was proposed. Based on the above analysis of viscosity, shear rate and needle characteristic, a model of needle characteristic determining the solution viscosity was obtained.

The kinetic description of texture variation during annealing in the 3103 aluminium alloy

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Keywords: texture, ODF, rolling, recrystallization, aluminium

The properties of cold and hot formed semi-products highly depend on the crystallographic texture. It is important to know their crystallographic texture since usually they undergo further shaping (and heat treatment) before reaching their final condition. In this research, the texture of the 3103 type aluminium alloy was investigated as a function of annealing temperature and time. The aim was to describe the kinetic of the recrystallization processes through the crystallographic texture. The initial state was hot rolled sheet, which was cold

rolled to 1 mm thickness. Subsequent annealing was carried out on the cold rolled sheets where the temperatures and the holding time was varied. The resulting textures are characterized by the volume fractions of the main texture components, which were calculated from the orientation distribution function (ODF).

Effect of heat input in the fusion zone of electron beam welded commercially pure aluminum and AISI 304 stainless steel

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Dissimilar joining of commercially pure aluminum and AISI 304 stainless steel was carried out using electron beam welding machine (12 kW) under three different parameters. The aim of this work is to evaluate the quality of weld joint through microstructural analysis, hardness measurement, and porosity calculations. Microstructural analysis as well as quantitative elemental analyses demonstrated increased thickness of the weld zone and interlayer by increasing heat input. Scanning electron microscopy together with area mapping has been done to analyze the microstructure of the samples. The optimized process parameters mainly show the variation of beam current and scan speed, while keeping beam voltage (55 kV) and beam offset (0.7 mm in Al side) constant. Mainly AlFe and AlFe₃ are the intermetallic phases formed during joining.

How the nozzle position affects the geometry of the melt pool in directed energy deposition process

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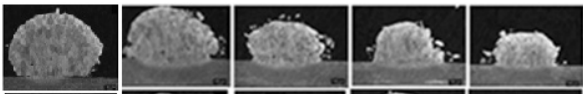
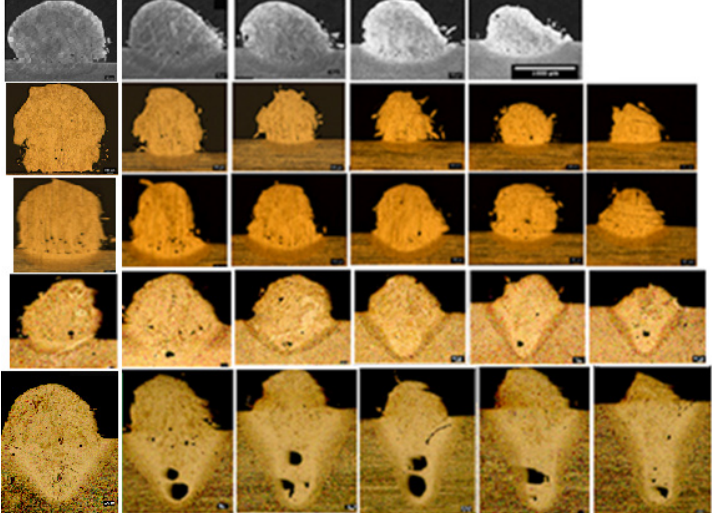
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Keywords: Directed Energy Deposition, Nozzle, Ti-6Al-4V, Single track, Melt pool

Additive Manufacturing (AM) is considered as a fast alternative technique for serial production of components with complex geometry. Among AM technologies, this work deals with Directed Energy Deposition (DED) that is a process in which the material (wire or powder) is delivered directly into the melt pool. When the powder is used, several nozzles such as lateral, coaxial and 4-nozzle have been developed to deliver it into the melt pool. However, the position of the nozzle, in particular in the case of lateral one, plays a key role in the geometry of the melt pool and subsequently in its porosity content. Therefore, in this work to investigate the effect of the position of the lateral nozzle on the geometry of the melt pool

three different sets of single tracks of Ti-6Al-4V were deposited by a DED process with two combinations of process parameters at different nozzle positions. Dimensions of single tracks were analysed, and geometrical characteristics of melt pools were evaluated after polishing and etching of cross-sections. It was found that helpful information regarding the selection of the optimal position of the lateral nozzle can be achieved from the melt pool features. It is found that by choosing a far position of the nozzle, the deposition rate would be lower and most of the melt pools have deeper fusion zones which contain keyhole porosity which is not desirable for the final properties.

Figures

Manual	Laser Speed (mm/s)						Power (W)
	30	40	50	60	70	80	
Manual A							980
Manual B							1500
							980
							1500
Manual C							980
							1500
							980
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							1500

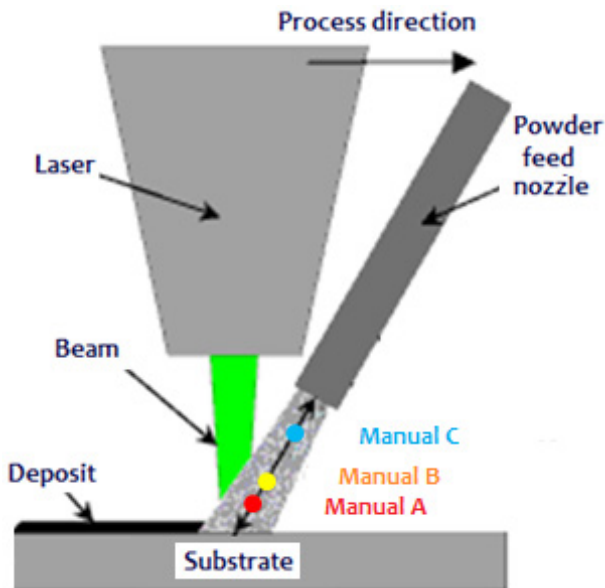


Figure.1 Geometry of Ti-6Al-4V melt pool at different positions of lateral nozzle

Acknowledgments

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Effect of beam oscillation on bead on plate electron beam welded pure niobium

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Keywords: Electron beam welding, Electrical resistivity, SRF cavity, Pure niobium

Niobium is the preferred material for making SRF cavities due to its highest critical temperature among the elemental superconductors. Electron beam welding is the widely used technique to build SRF cavities. In present work, bead on plate welding of pure niobium (RRR-300) samples has been carried out using 60 kV (15 kW) electron beam welding unit (Model LARA 52 supplied by M/s Techmeta, France) under varied process parameters. Followed by welding, a detailed microstructural characterization has been carried out using

optical and scanning electron microscope that shows substantial grain growth in the melted zone. A detailed phase analysis shows no contamination or formation of any new phase after melting. Lattice strain and residual stresses developed in the melt zone has been evaluated using X-ray diffraction technique. Electrical resistivity measurement has been carried out in the temperature range of 20-300 K using four point probe method, which shows an increase in resistivity when beam oscillation has been used. The variation in resistivity has been correlated with the amount of volume defects present using x-ray micro CT-scan. EBSD analysis has been done to evaluate the change in texture and its effect on properties after welding.

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Conform of titanium wire from particulate feedstock

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The continuous extrusion process Conform has been used to extrude titanium alloy powder directly into rod. The process has historically been used to process rod feedstocks in lower strength aluminium and copper alloys for the power generation industries. However, research conducted at the University of Sheffield has enabled the extrusion of high strength, high value metal powder feedstocks through a Conform machine. This work demonstrates a potential step change in the cost reduction efforts for a range of applications ranging from lightweight automotive structures through to Wire Arc Additive Manufacturing feedstocks.

Starting out from the small scale, this work begins at initial investigations into the way the powder behaves under shear (making use of a bespoke annular shear cell) and how different tooling materials behave under friction (using a wear tester). Both of these lab-scale experiments help us understand the larger Conform process and how tooling and powder choices affect the extruded product.

A number of full scale trials have been completed on the Conform machine, each producing 10 mm diameter rod made from a Ti-6Al-4V HDH powder feedstock, which is one of the largest diameter titanium products produced in this manner.

This project investigates the production of Ti-6Al-4V wire from a reasonably low cost feedstock using the Conform process, with the intention of using the product for wire-fed additive manufacturing processes.

The effect of vacuum degree on the porosity and mechanical properties of die cast (AlSi9Cu3(Fe)) aluminum alloy by life data analysis

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In cold chamber die casting of aluminum alloys, air and other gases are often trapped in the metal, because of the turbulence of the alloy as it is forced into the die cavity at a high pressure. This phenomenon can cause porosity in the casting, that may affect the mechanical properties of the product. AlSi9Cu3(Fe) aluminum alloy castings were produced by conventional high pressure die casting (HPDC) and vacuum assisted high pressure die casting (VPDC) processes under atmospheric and two different absolute pressures of 250 mbar and 80 mbar. The influence of absolute pressure in the die cavity on the porosity and mechanical properties of the die castings were investigated and compared with traditional casting method. The two-parameter Weibull distribution model was applied to deal with the variation in mechanical properties of the die cast flat tensile specimens. Porosity of castings was assessed on the basis of X-ray observation and the density measurements performed by the method of hydrostatic weighing.

The investigations proved that the volume of gas porosity and the pore sizes in the castings can be significantly reduced by using vacuum assistance during die casting. As a result, the density and the mechanical properties, particularly the tensile strength and elongation were significantly improved. The specimens contained larger pores under higher absolute pressure. Meanwhile the shape of pores is found to be also an important factor affecting the mechanical properties. In general, higher vacuum degree contributes to reduce the porosity that would be the basis to improve the mechanical properties of die cast parts.

Poster

3D collagen scaffold fabricated using a bio-printing process for tissue regeneration

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Keywords: 3D collagen scaffold, tissue regeneration, bio-printing

While the human body has limited self-healing ability, severe injuries with massive loss of tissue are hard to regenerate by itself. Basic concept of tissue engineering suggests that fabrication of biological substitution can challenge this medical problem [1]. Especially, mimicking the native extracellular matrix (ECM) has been a strategy for scaffold fabrication to enhance cell adhesion, migration, proliferation and differentiation. The main components of ECM are proteoglycans and fibrous proteins. Collagen is the main component and most abundant fibrous protein in ECM [2]. For this reason, several researches have been studied to fabricate collagen scaffolds for tissue regeneration using freeze drying [3], 3D printing [4], and electrospinning [5]. 3D printing has advantages in fabrication of complex geometry and control of macro pore, which is an important factor for tissue regeneration. However, it has been challenging to fabricate collagen scaffolds using 3D printing due to low viscous nature of collagen. In this study, pluronic F-127 (PF-127), which is a biocompatible material, was mixed with collagen type I in order to enhance printability. 3D mesh structures were fabricated with collagen/PF-127 mixture and cross-linked chemically. 3D collagen scaffolds were obtained after the removal of PF-127 by washing with ultra pure water. To evaluate cellular activities, MC3T3-E1 cells (pre-osteoblast like cell line) and adipose derived stem cell (ASCs) were seeded onto the fabricated scaffolds. It showed outstanding cell adhesion, proliferation compared with conventional collagen scaffold. We expected that this scaffold can be used for tissue regeneration.

Acknowledgments

This study was supported by a grant from the Ministry of Trade, Industry & Energy (MOTIE, Korea) under Industrial Technology Innovation Program (No. 10063541: Development of bioceramic 3D printing materials and low temperature (<40 °C) process customized by implant sites) and was also financially supported by the National Research Foundation of Korea Grant funded by the Ministry of Education, Science and Technology (MEST) – Republic of Korea (NRF-2018R1A2B2005263).

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The effect of carbon nanosheet from natural waste materials on aluminium matrix composite properties

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Keywords: Carbonization, Chemical activation, Carbon nanostructured, Composite materials

Natural waste materials are a new trend for producing nanostructured materials by different processes. Plants are a natural material consisting mainly of cellulose, hemicellulose, and lignin. Cellulose (C₆H₁₀O₅)_n is an organic compound with carbon which derived from plant structure. Carbon is a good reinforcement material and carbon composite materials have various applications. This investigation aims to develop high-performance low-cost aluminium matrix composites (AMCs) reinforced with synthesized carbon nanosheet from natural waste materials by powder metallurgy technique to improve the properties of AMCs. In this study, AMCs were reinforced with carbon nanosheet produced from nettle or from peanut shell. Carbon nanosheet was synthesized by chemical and thermal treatment process at 800 °C under argon atmosphere in a low-cost and uncomplicated process, which is a high added-value of advanced carbon materials from natural waste materials. The microstructure and morphology of the samples were investigated by scanning electron microscopy (SEM) and the chemical compositions by energy-dispersive X-ray spectrometry (EDS).

Issues and main solutions in FSW of Al alloys and steel sheets

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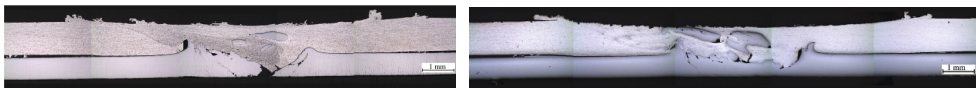
Keywords: friction stir welding, steel sheet, aluminum alloy sheet, welding defects, automotive industry

Unlike fusion welding, friction stir welding (FSW) is a technique that has the capability of joining metals in a semi-solid state. FSW is successfully used in several industries (e.g. aeronautic field) for joining aluminum (Al) alloys since it ensures the formation of sound and resistant joints. Differently, FSW of Al alloys and steels pose some serious issues because of their different mechanical, chemical, and physical properties. Particularly, the formation of large intermetallic compounds (IMCs), severe tool wear, and weld defects (e.g. voids, incomplete penetration, weak hook effect) are common drawbacks of FSW joints.

The low solubility between Al and steel often causes the formation of large brittle IMCs (type Al_xFe_y) in the weld nugget and thermo-mechanically affected zone (TMAZ). These phases usually promote crack nucleation and/or limit mechanical strength of joints [1]. The amount and size of IMCs are strictly influenced by process parameters. Low tool rotational and travel speeds are often the main solutions to prevent or limit large IMCs layer. Zinc coated steel sheets are less sensitive to the formation of Al_xFe_y compounds since zinc has a better affinity with Al, forming less harmful precipitates (type Al_xZn_y), [2]. Post annealing treatments at moderate temperature ($< 350\text{ }^{\circ}\text{C}$) are an effective way to relieve residual stresses in FS weld and may contribute to the nucleation of very small IMCs, which seems to improve joint strength [3].

FSW tools undergo significant stresses and heat during joining of Al and steels, which are responsible for a severe tool wear. In this regard, improper worn surfaces cause the occurrence of pores throughout the weld surface, nugget, and TMAZ [4]. Therefore, expensive hard materials (e.g. WC, W-Re, P-CNB alloys) and/or coatings (e.g. TiC, TiN, multiple layers) are needed to improve wear resistance of such tools.

Improper welding parameters have instead a main influence on joint integrity. Proper design of experiments (e.g. factorial, Taguchi) are helpful to identify proper weldability windows to avoid, for instance, incomplete penetration, Fig. 1a, or internal defects, Fig. 1b.



Incomplete penetration
Internal defects

Fig. 1. Some typical defects in FSW Al/steel joints.

Overall, an overview of the typical issues about FSW of Al alloy and steel sheets in the automotive industry and their main solutions are outlined and addressed in this study.

Acknowledgments

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Correlation between the laser focus and regularity of deposition in DED of AISI 316L

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Keywords: Directed energy deposition, Stainless steel, deposition, laser focus

Additive manufacturing (AM) technologies refer to the type of flexible processes to produce objects with different and complex geometries from a CAD model without using any tool and mold. AM technologies are bottom-up processes which produce objects by using the material in a layer by layer way. These technologies allow processing a wide range of materials such as titanium, stainless steel, nickel-based superalloys. As a matter of fact, it is found that the microstructure and mechanical properties of materials produced by AM technologies are superior with respect to the traditional methods. In general, Laser powder bed fusion (LPBF) and Directed energy deposition (DED) are two types of laser additive manufacturing. Although they are based on the same principles, but there are some differences from the metallurgical point of view, such as solidification rate, melting situation and mechanical performances. Production of large component is a big challenge for LPBF process and this challenge can be addressed by means of DED technique. Process parameters in DED include the laser power, laser speed, powder feeding rate, shielding gas and carrier gas flow rates and laser focus. Among them, laser focus plays an important role in deposition of materials by changing the laser spot size. In fact, changing the laser spot size modifies the energy density of laser and consequently fusion zone and melt pool dimensions. In this study, the effect of the laser focus on the regularity of deposition in DED of AISI316L has been investigated

through the top side and cross-section analysis. According to the findings in this work, it can be concluded that it would be possible to deposit AISI316L more regular at the higher laser focus as a consequence of lower input energy and also less turbulence in the melt pool.

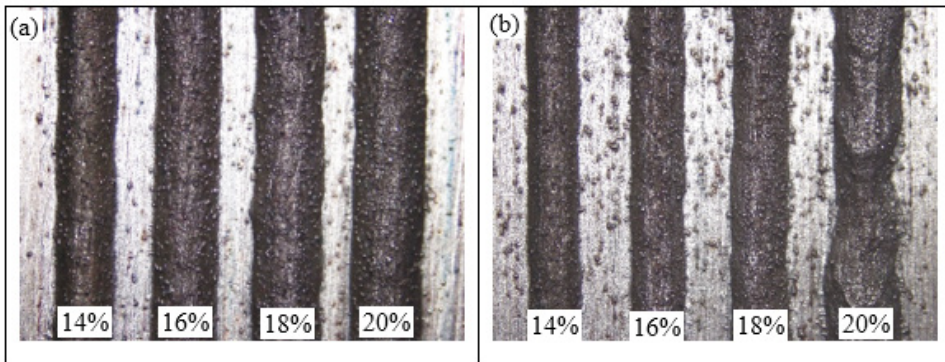


Figure 1. Single scan tracks of AISI 316L deposited by DED at (a) high (7.5 mm), (b) low (2.5 mm) laser focus.

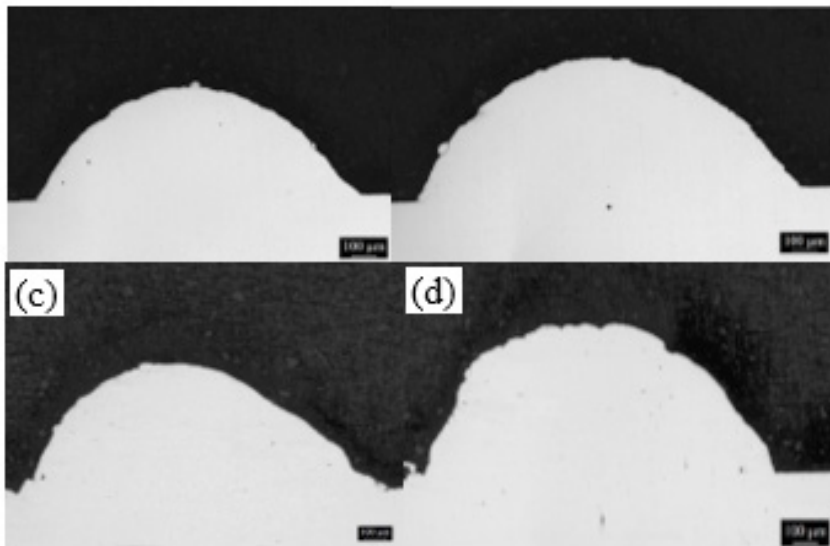


Figure 1. Cross-section of single scan tracks of AISI 316L deposited by DED at (a,b) high (7.5 mm), (c,d) low (2.5 mm) laser focus.

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Aligned cell-laden alginate fibrous structure fabricated by an electrospinning process for muscle tissue regeneration

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Keywords: electrospinning, scaffold, alginate bioink, muscle regeneration

Electrospinning is a versatile tool to build a micro/nanofibrous structure with controllability of topological cues. The fabricated micro/nanofibers are similar to the structural geometry of extracellular matrix (ECM), so it has been widely applied to regenerate various tissues [1]. Typically, aligned topological cue is a critical factor to regenerate skeletal muscle which are composed of myotubes in anisotropic parallel bundle structure [2]. In this concept, electrospinning using cell-laden bioink provides a great advantage to directly immobilize and align cells within fibrous structure [3]. Thereby, we report generating aligned fibers using alginate/PEO solution in the effects of various processing parameters such as flow rate, voltage,

nozzle-to-electrode distance, and electrode-to-electrode distance. To examine the feasibility of electrospinning process using cells, C2C12 cells were encapsulated into alginate/PEO solution as a bioink. The bioink was electrospun to fabricate cell-laden fibrous structures which were studied on *in vitro* cellular activities like cell viability, proliferation, and differentiation. Here, the cell-laden electrospun fibrous structure in aligned manner is proposed for its possibility to be utilized in tissue engineering applications.

Acknowledgments

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Energy, Transportation and Environment

Oral

Thermography of hydrogen sorption-desorption processes in La-Ni based metal hydrides

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Keywords: metal hydrides, hydrogen, energy storage, renewable energy, fuel cells, thermography, La-Ni based alloys

Metal hydrides are one of the types of functional materials that allow safe and compact storage of a large amount of hydrogen, which is increasingly used today as an alternate fuel or energy source. The possibility of obtaining the initial energy necessary for the production of hydrogen by electrolysis process from renewable energy sources, such as solar panels and wind generators, makes hydrogen energetic quite attractive and rapidly developing industry sector but hydrogen storage and transportation to the end consumer still remain the open questions. Development of powder metallurgy provides new possibility for investigations in the field of alternative way of hydrogen storage. Solid form of hydrogen storage with the possibility of reversible sorption, gives opportunity for creation autonomous energy storage systems and use hydrogen like a modern energy carrier.

Lanthanum-nickel based alloys allow hydrogen storing at ambient temperatures and pressure not higher than 15 bar, which makes the application of these alloys quite practical. The disadvantages of these alloys are the high cost of the initial powdered pure metals and the relatively low mass content of hydrogen in the final alloy. Despite this, La-Ni alloys are quite interesting and prospects for further study and modifications.

The additional issue of using hydrogen storage systems based on metal hydrides is the importance and accuracy of the thermoregulation process during hydrogen sorption and desorption and thermography of the fuel cells during hydrogen conversion to electricity.

Influence surface quality of EN AW-1370 wires on material fatigue strength

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Keywords: fatigue strength, surface quality, aluminium wires, drawing process, topography of surface

Fatigue of the material is a process of forming and developing defects in the material as a result of multiple changing (in cycles) loads. Fatigue process of material is connected with the lowering the value of the mechanics of material which is connected with the fatigue limit. Fatigue is a general cause of premature failures of construction and the term in general refers to the finished number of load cycles which set material is able to bear. Fatigue process is not well-known and elaborated. One of the theories proves that the starting point of fatigue phenomenon is anisotropy and irregular placement of material's grains. At the beginning local ductile deformation are found and its existence can be proved using the microscope because slip bands are visible as dark band around the grain. They develop as the number of cycles rises forming a pile-up and bundles which results in creating cracks and their binding. These cracks are usually forming at the surface and top layers of the elements.

Scientific objective (the problem which applicant is trying to resolve, questions or research hypotheses)

The scientific objective of this project is to determine mechanisms responsible for the initiation and propagation of fractures in aluminum wires after high cycle fatigue process. In particular, this research should accomplish to determine the influence of surface quality (various topography), various microstructure (size of the grain) and various types of work hardening (amount of dislocations) on the locations of fractures and its propagations due to material's fatigue process.

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The electrochemical properties of Na- β "-alumina originated from single crystal alumina prepared by vapor phase process

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Keywords: Na-S battery, Electrical conductivity, Na- β "-alumina

Na-S battery has gained an great attention due to its advantages such as the low cost and long-term stability. Na- β "-alumina, the composition of $\text{Na}_2\text{O} \sim 6\text{Al}_2\text{O}_3$, is a typical material applied in Na-S battery as the electrolyte and the separator due to its high sodium ion conductivity and chemical stability. Generally, the ionic conductivity of poly crystalline Na- β "-alumina at 300 °C is ~ 0.25 S/cm. Conventional method for the formation of Na- β "-alumina is that calcinated mixture (Na_2CO_3 , LiNO_3 (or MgO) and $\alpha\text{-Al}_2\text{O}_3$ powders) is converted to Na- β "-alumina at high sintering temperature (1400~1500 °C). However, NaAlO_2 can be formed in the conventional process and it remains at grain boundaries of Na- β "-alumina. Formation of NaAlO_2 is one of the biggest reason for decreasing conductivity causing the reduced performance of the Na-S battery. Recently, the vapor phase process has been reported that the poly crystalline $\alpha\text{-Al}_2\text{O}_3$ is exposed to Na_2O gas to convert into β "-alumina. This process converts the $\alpha\text{-Al}_2\text{O}_3$ /YSZ composite into a Na- β "-alumina/YSZ composite. NaAlO_2 is not formed in this process and β "-alumina prepared by vapor phase process exhibits high chemical and mechanical stability. In this study, the electrochemical properties of Na- β "-alumina originated from single crystal Al_2O_3 have been systematically investigated. It is the first report to confirm the eletrochemical properties of Na- β "-alumina by using single sapphire crystal. The Na ion conductivity related to structural and microstructure changes will be discussed.

A study on correlation between improved oxygen permeability and coating materials in fluorite-rich dual-phase membranes.

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Keywords: Oxygen transport membranes, Oxygen permeation flux, Coating layer, Oxygen surface exchange kinetics, Oxygen diffusion

Reducing carbon dioxide emissions is essential to mitigate global warming, since carbon dioxide emitted during the burning of fossil fuel is severe concern for the global climate change. The CO₂ capture and storage technology is a promising technology to reduce carbon dioxide emissions from coal-fired power plants and is mainly classified into three concepts (pre-combustion, post-combustion, and oxy-fuel combustion capture). Recently, the mixed ionic and electronic conducting membrane has gained an attention owing to their potential application to supply pure oxygen to the power plant for CO₂ capture. The perovskite oxide-based single-phase membranes have been mainly studied to achieve high oxygen flux. However, chemical and mechanical instability of the single phase membrane is a major obstacle for commercialization. In order to solve the problem of single-phase membrane, fluorite oxide-based composite membrane have been intensively studied to enhance overall stability of the membrane.

It is important to understand the role of surface modification in the oxygen permeation behavior since the active coating layer on the ceramic membrane is important function in the improvement of the oxygen permeation flux. In this study, the role of coating layers on the oxygen permeation in a fluorite-rich dual-phase membrane [80 vol% Ce_{0.9}Gd_{0.1}O_{2-δ} (GDC): 20 vol% La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-δ} (LSCF)] has been investigated as a function of membrane thickness and temperature to explain the enhancement of oxygen flux. The various materials such as ionic, electronic, or mixed ionic and electronic conductor have been applied to the coating layer for improving the oxygen permeation flux. The role of surface modification in the dual-phase membrane to enhance the oxygen permeability has been systematically researched in the viewpoint of electronic conductivity, surface exchange kinetics and bulk diffusion.

Effect of attritor milling and sintering temperature on the thermo-mechanical properties of YSZ/MWCNT composites

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Several energetical applications are known where yttria-stabilized zirconia (YSZ) and zirconia / multiwall carbon nanotube (MWCNT) composites were useful used as parts of solid oxide fuel cells (SOFC), photovoltaic solar cells, supercapacitor or hydrogen storage materials. The Ni – YSZ cermets are widely accepted as anodic materials in SOFCs with high catalytic activity and electrical conductivity. Some research works showed the drawbacks in hydrocarbon fuels as carbon build-up, sulfur poisoning or low tolerance to redox cycling. These irreversible processes damage the microstructure of anodes and reduce the cell performance, therefore Ni-free anode materials are continuously researched and developed to overcome these problems. In this work, the design of milled and sintered YSZ / MWCNT composites was studied. The detailed structural investigations confirmed the MWCNT clusters in all cases, while the best homogenization was obtained in the case of YSZ / 1 wt% MWCNT composite. The Raman measurements showed unanimously results with structural observations. The apparition of the G and D bands for all the composites at $\sim 1589\text{cm}^{-1}$ and $\sim 1356\text{cm}^{-1}$ confirmed the structural integrity of MWCNT after the milling process. The effect of attritor milling and spark plasma sintering (SPS) at 1200°C, 1300°C and 1400°C on the structural and thermo-mechanical properties of 8 mol% yttria-stabilized zirconia (8YSZ) composites with 1wt%, 5 wt% and 10 wt% MWCNTs addition has been investigated.

Asymmetric oxygen transport membrane prepared by phase inversion tape casting

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Asymmetric membranes with a porous support and a dense membrane layer have attracted huge interest for oxygen separation from air. However, severe rate limitations are reported due to gas diffusion through the tortuous support pores. Among the technique of preparing

asymmetric membranes, phase inversion method provides an advantage of obtaining straight pores, which could facilitate O₂ molecular transport. In this work, flat BSCFZ asymmetric membrane supports were prepared with the phase inversion combined tape casting method. Dense membranes layers were applied with the screen printing method. The microstructures were characterized with SEM and light microscopy. The oxygen permeation rate of BSCFZ samples prepared with phase inversion tape casting method were twice that of 1 mm thick bulk BSCFZ membranes. In addition, bulk diffusion, support limitation and surface exchange limitation were investigated by different thickness of membrane layer, different porosity of support and activation layers, respectively.

Enhanced nanoporous carbons as adsorbents for mixed phenolic acids

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Keywords: enhanced nanoporous carbon, pharmaceutical contaminants, salicylic acid, benzoic acid

Nanoporous carbons are often recommended as the adsorbent materials for pharmaceutical contaminants. Phenolic compounds are only one group of numerous contaminants which are excellently adsorbed on the classical activated carbons. However, mixed adsorption of these contaminants is complicated case which demands a different approach in estimating the material's applicability in particular filtration technologies. Salicylic and benzoic acid adsorption was studied on special types of enhanced nanoporous carbons produced on bases of the Balkan-originating cellulose waste. Detailed adsorption kinetics is discussed in the frame of the thorough carbon material characterization results.

Mixed adsorption was also tested through the model for prediction based on Dubinin's theory and the Myers-Prausnitz's ideal adsorption theory. From the results it is suggested that model could be applied in prediction of adsorption parameters with reasonable accuracy. Experimental results are compared with theoretically calculated values for two systems and plotted and discussed.

Hydrocarbon energy, environment & water resource management

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Keywords: Energy, Water Resources, Hydraulic Fracture, Environment

In an era of a competing global energy mix - where alternative energy sources are playing significant role in some countries, hydraulic fracturing is a huge contributor in making shale gas the key to escalating the role of natural gas. It has been reported that hydraulic fracturing is used on 90% of all oil and gas wells drilled in the United States. However, associated water requirements are enormous and remain a major challenge and necessity. Water requirement for hydraulic fracturing operation in Texas varies from somewhere 1 million gallons/well to 13 million gallon/well depending on the types of shale.

An important aspect of the hydraulic fracturing process is identifying and securing sufficient water resources. With the ever-increasing demands for higher quality water resources for domestic and agricultural needs, Water withdrawals could have significant ecological impacts especially in water-stressed areas. In regions where there is strong competition for water resources, drawdown in hydraulic fracturing water-supplying wells could result in; the lowering of aquifer water-table, serious effect on biodiversity and the exposure of indigenous ecosystem to harm. Productive activities like agriculture and other consumptive uses of water by host communities might be affected, due to reduced or limited water availability. Similarly, transportation of large volumes of water in truckloads could degrade local infrastructure (roads, bridges) and lead to road congestions and accidents. However, maintaining the energy demand is also as important as the sustainable environment.

Hydraulic fracturing has generated a tremendous amount of controversy in recent years. Many countries in Europe and globally are calling for a temporary moratorium or a complete ban on hydraulic fracturing due to concern over environmental, social, and public health concerns. Water abstraction in shale gas development is governed by regulations. In Europe, shale gas exploitation and water consumption is regulated under general EU water legislation on water resources such as the Drinking Water Directive, the Water Framework Directive and the Groundwater Directive. The United states oil and gas industry, including shale gas, are variously regulated under federal, states and local laws while in China, water withdrawals for shale gas, is controlled under the Water-Drawing Permit and Water Resources Charges regulation. However, enforcement gaps persist. An efficient, project-specific and localized water-requirement plan is recommended, putting into consideration; water availability, if water recycling is necessary for the project, and the possibility of reducing the use of chemicals.

This paper reviews the regulatory framework governing shale gas plays around the world with respect to water abstraction. Water Resource management is as important as the hydrocarbon energy extraction for the modern life. Furthermore, how the hydrocarbon industry is preparing for the challenges and synthesis of existing research will be discussed.

The effects of engine oil degradation by OME-diesel fuel blends on friction and wear

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Keywords: lubricant degradation, oxymethylene ether, friction, wear

Non-fossil fuels for the commercial and passenger vehicle sector are gaining more importance, due to their positive effects on exhaust gas emissions. Ongoing research investigating a broad variety of biofuels, synthetic fuels, and regenerative fuels shows that a reduction of CO₂ emissions can be possible without major modifications to the existing vehicle infrastructure. An internal combustion engine is a complex system of physical and chemical mechanisms, all of which contributing to performance output, exhaust gas composition, durability and longevity of the engine. Modifying the fuel will not only result in differing chemical reactions and thermodynamic efficiency but also will affect friction and wear through the dilution and degradation of the lubricant.

This study aims to investigate the variation of friction and wear in a model system due to the introduction of an oxymethylene ether – diesel blend to the lubricant. High-frequency reciprocating rig experiments will be carried out on 100Cr6 steel specimen in fully formulated commercially available SAE 0W-20 and SAE 0W-30 grade engine oil with different amounts of OME-diesel blends. The contaminated lubricant samples will be subjected to chemical analysis. The surface of the steel specimen will be analyzed using optical microscopy, interferometry, and scanning electron microscopy.

Influence of a dopant on birnessite-MnO₂ and electrochemical activity for oxygen reduction reaction (ORR)

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Manganese oxides, including MnO, MnO₂, and Mn₃O₄, are intriguing materials and have been used in wastewater treatment, catalysis, sensors, supercapacitors, and alkaline and rechargeable batteries. Particularly, birnessite-type MnO₂ materials have attracted great interest as supercapacitor in batteries for their high theoretical capacity, environmental benignity, and other special properties. Low cost and high activity electrocatalysts for oxygen reduction reaction (ORR) are necessary for the development of fuel cells and metal-air batteries. The

birnessite structure consists of layers of manganese-oxygen octahedra (MnO_6) with interlayer cations (Na^+ , K^+ etc.) and water molecules positioned between the MnO_6 layers. Replacing interlayer cations of the birnessite for Co^{2+} , Ni^{2+} or Cu^+ can lead to higher stability during the electrochemical cycling, increased activity for electrocatalytic mechanism, and improved electrochemical oxygen reduction activity in fuel cell system.

Here we report a simple and cost-effective method to synthesize Fe and Cu intercalated birnessite with high surface area and improved catalytic activity for oxygen reduction reaction (ORR). The intercalation of the metals (Fe, Cu) was verified by XRD and Raman spectroscopy, the morphology was studied by scanning electron microscopy, the chemical composition was measured by energy dispersive X-ray spectroscopy (EDX), the crystal structure was determined by transmission electron microscopy (TEM), the band-gap energy was calculated using diffuse reflectance spectroscopy in the ultraviolet-visible region (DRS-UV-VIS), the specific surface area and pore size distribution was measured by nitrogen adsorption, the thermal properties were investigated using thermogravimetry and electrochemical measurements were performed in a three-electrode system.

Our study demonstrates a successful intercalation process to produce metal doped birnessite with increased specific surface area and improved electrocatalytic activity for oxygen reduction reaction.

The relationship between surface finish and performance for stainless steel intermediate level nuclear waste containers

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Intermediate Level Waste (ILW) containers face the challenge of withstanding corrosion while in interim storage and disposal in a geological repository. These uncoated, 2.33mm thick, stainless steel (316L and 304L) containers rely on the superb corrosion resistant qualities of their component materials.

Surface finish can profoundly affect the corrosion of steels. Here we present results from electrochemical studies used to determine the effect surface finish has on the performance of 316L and 304L stainless steels in both simulant interim storage contaminate electrolyte and a MgCl_2 solution.

Under-droplet atmospheric, stress corrosion cracking was generated in the lab to study general cracking behaviour, with a focus on surface finish related effects on crack initiation.

This work coupled with a thorough characterisation of various surface finishes, including; roughness, morphology, physiochemistry and the presence of inclusions hopes to show a relationship between surface finish and corrosion behaviour, and go some way to explaining why such effects take place.

Poster

Investigation of the microstructure of autoclave-formed zirconium oxide via top-down EBSD

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Zirconium alloys are used almost exclusively by the nuclear industry to clad nuclear fuel, providing structural integrity and preventing the escape of fission products into coolant water. Zirconium alloys are selected for this role due to their low neutron absorption cross-section (therefore allowing for a more efficient reaction); good corrosion resistance; and adequate mechanical properties. [1] One of the life limiting factors for Zr alloy cladding is oxidation and hydrogen pickup caused by the high temperature, high pressure aqueous environment. It is therefore vital to understand the underlying mechanisms behind the corrosion behaviour of Zr alloys in order to lengthen the lifespan of core components and increase plant efficiency. [2]

Previous work has highlighted the importance of oxide grain size, morphology, phase, and orientation on the corrosion performance of Zr alloys. [2,3] It has been observed that increased corrosion rates are associated with small, equiaxed oxide grains due to a high proportion of high-energy grain boundaries. Observations have also shown that large, columnar, monoclinic oxide grains which are oriented parallel to the oxide growth direction form an effective barrier against corrosion. [4]

The microstructure of zirconium oxide is routinely examined adjacent to the metal/oxide interface via TEM, showing the entire oxide thickness and underlying metallic substrate. [5] The current work utilises an alternative approach of top-down EBSD to observe the orientation, phase, and morphology of oxide grains parallel to the metal/oxide interface. Autoclaved Zr alloy samples were mechanically polished in increments to gradually remove the oxide layer. Between these polishing stages EBSD maps were produced, highlighting the variation in oxide microstructure throughout the thickness of the oxide.

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A new approach to avoid errors from electrode overpotential using an in-plane geometry in Hebb-Wagner method

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Keywords: Hebb-Wagner polarization method, Electrolyte, Overpotential, Electronic conductivity

Oxygen ion conductors have been extensively studied for application of electrochemical devices such as solid oxide fuel cells and gas sensors. Among the class of oxygen ion conductor, doped-ceria has been widely used as the solid electrolytes due to its high ionic conductivity. However, in reducing atmosphere or high temperature range, it is a problem that the electronic conductivity increases due to the formation of oxygen vacancies and electrons. The change of electronic contribution in the electrolyte materials can induce variation of electrochemical properties. Therefore, it is highly important to exactly determine the electronic conductivity in the solid electrolytes for appropriate utilization. The Hebb-Wagner polarization method is most widely used to separate and measure the partial electronic conductivity due to the technical simplicity and as well as its high accuracy of measurement. In this method, the electrode consists of ion-blocking and reversible electrodes as an asymmetric electrode configuration. Since the ion-blocking electrode blocks the ionic current, only electron conductivity by a minor charge carrier can be estimated. Two electrodes configuration has been typically used to measure the partial electronic conductivity with Hebb-Wagner polarization method. However, the contribution of the electrode over-potential cannot be excluded in the 2-probe method. Thus, it is very difficult to obtain the accurate partial electronic conductivity by using 2-probe polarization method. In this study, new approach has been proposed to overcome the electrode overpotential problem in the 2-probe Hebb-Wagner polarization cell by dramatically increasing the shape factor of the cell. In order to accurately estimate the electronic conductivity, the thick-film cell has been fabricated using the tape casting technique, which dramatically increases the shape factor of the cell. This approach can be also applied to Li or Na conductors as well as the oxygen ion conductor materials.

A new approach to accelerate ionic transport of Na- β "-alumina with pre-added phase stabilizer in vapor phase method

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Keywords: Beta alumina, Solid electrolyte, Ionic conductivity, Vapor phase method, phase stabilizer

Na- β "-alumina solid electrolyte (Na-BASE) with the nominal composition of $(\text{Na}_2\text{O}) \cdot (\text{Al}_2\text{O}_3)_x$ ($x = 5 \sim 7$) has played a crucial role for sodium high-temperature rechargeable batteries, such as sodium-sulfur (Na/S) and sodium-metal halide (Na/MX) batteries, due to its fast ionic conduction and high thermal stability. Conventional β "-alumina solid electrolytes (BASEs) are synthesized with a mixture of sodium precursors, phase stabilizer (Li, Mg sources), and alumina sources, and sintered above 1500 °C. The role of phase stabilizer is to increase sodium concentration for enhanced ionic conduction on the conduction plane by compensation mechanism [1]. One of the major technical issues in BASE fabrication is sodium volatilization on high temperature firing due to the high vapor pressure of Na_2O [2]. Recently, to resolve the issue, the vapor-phase conversion of α - Al_2O_3 /yttria-stabilized zirconia (YSZ) composite into the BASE at lowered temperature (<1450 °C) was introduced [2-5]. The conversion process is based on coupled diffusion of sodium and oxygen ions from $\text{Na}_2\text{O(g)}$; The ions are transported through the converted Na- β "-alumina and YSZ, respectively. However, in the previously reported processes, the supply of the phase stabilizer depends only on the diffusion from the cation sources; It was not examined whether the supply of the content of the phase stabilizer is sufficient [2-5]. In this presentation, the ionic conduction in Na-BASE prepared by vapor phase method will be discussed as a function of lithium stabilizer contents which was added before sintering process.

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Characterisation of novel low activation high entropy alloys for high temperature and fusion plasma-facing materials applications

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Keywords: high entropy alloys, plasma-facing materials, characterisation, fusion energy applications, microstructure, mechanical properties

Nuclear fusion energy has the potential to supply a baseload source of clean electrical energy with little or no radioactive waste, but significant materials challenges need to be successfully resolved. Development of high performance structural materials that can withstand the intense thermomechanical stresses and severe bombardment of high energy neutrons is therefore essential to enable the advancement of fusion power.

High Entropy Alloys (HEAs) constitute a relatively new field of metallurgy comprised of novel metallic alloys which, due to their reportedly superior mechanical properties including: high strength and ductility, high temperature softening resistance and corrosion resistance, have been proposed as potential candidates for high temperature fusion structural applications. Contrary to traditional alloys which are based around one or two primary elements, HEAs comprise of a combination of several elements in similar amounts. Little research so far has focussed on HEA compositions of low activation (tendency to become radioactive when exposed to a fusion reactor environment).

In the present study, the design, fabrication, and characterisation of novel quaternary HEAs for the application of plasma-facing materials was carried out. Specimens were prepared via vacuum arc melting on high purity (<99.5 %) elements. Characterisation for composition, microstructure, and mechanical testing has been investigated by a combination of X-ray diffractometry, X-ray fluorescence, scanning electron microscopy, and microhardness measurements; subsequently an assessment is made for the suitability as candidate structural fusion materials.

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Comprehensive characterization of the automotive brake wear debris is crucial for understanding of its environmental impact

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Keywords: brake wear debris, friction composites, airborne particles, nonairborne particles, chemical analysis, phase analysis, particle size distribution

Automotive brake wear debris are released into the environment during the friction process and represents a major source of non-exhaust emissions from road traffic. Brake linings are multicomponent composites which contain more than 10 constituents (metals, polymer matrix, etc.). Chemical and phase composition of initial brake pads is confidential and not commonly provided by manufacturers. Braking process is accompanied by high temperature, high pressure and mechano-chemical interactions. These conditions together with applied friction force cause release of wear debris whose composition significantly differs from initial brake pad materials due to chemical changes. Produced mixture of wear particles can be in the form of single elements, inorganic and organic compounds with different sizes and shapes. Amount of released material is increasing together with growing global car park. Enormous quantities of released wear debris require a better understanding of the negative impact on the environment and human health related to the several potentially hazardous materials present in brake pad materials. Contribution of brake wear to the environmental pollution is still not fully understood and there is not standardized procedure for the characterization of composition and evaluation of their environmental impact.

The first step to assess for determination of the potential impact of brake wear particles to the environment is comprehensive characterization of their elemental and phase composition, size and shape. Airborne and nonairborne wear particles were collected after standard brake dynamometer test from the environmental chamber and filters of full-scale dynamometer (Link M2800). Elemental and phase composition of the collected brake wear debris was analyzed by scanning electron microscopy with energy dispersive X-ray spectroscopy, transmission electron microscopy, X-ray fluorescence analysis, and X-ray powder diffraction analysis. Particle size distribution was determined, as well. In all cases wear particles have heterogeneous character and very similar basic elemental composition with a majority of carbon and iron. Airborne particles are several times smaller in diameter than non-airborne.

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Aggregation behavior of silver nanoparticles in biorelevant conditions

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Silver nanoparticles (AgNPs) are heavily investigated due to their unique optical, catalytic and biological properties that can be controlled by their size and shape. To assess the potential of AgNPs in various therapeutic applications, their physiological effects are widely investigated in *in vitro* model systems. However the aggregation behavior of AgNPs in bio-relevant conditions like varying pH, ion-, and biomolecule concentrations is rarely examined, although it would be highly relevant prior utilizations.

In this study, three silver nanoparticle samples were synthesized with different methods; chemically reduced particles stabilized by sodium citrate (AgNP@C) and the surface of MCM-41 type mesoporous silica nanoparticles (AgNP@MSN), in addition, a green synthesized sample prepared using green tea extract (AgNP@GT). Particle size and morphology were characterized using transmission electron microscopy (TEM) and the aggregation measurements were carried out on different pH values, sodium chloride, glucose and glutamine concentrations, furthermore, particle behavior was investigated in the presence of Dulbecco's Modified Eagle's Medium (DMEM) and Fetal Bovine Serum (FBS) cell culture medium components using UV-Vis spectroscopy and dynamic light scattering (DLS). Viability assays were also carried out to investigate the influence of nanoparticle aggregation grade on toxicity.

Our results show that high aggregation grade in AgNP samples attenuates their toxic effects towards living cells. Their colloidal stability is mostly dependent on pH and salt concentration, while the presence of FBS can improve particle stability due to the biocorona effect. AgNP@GT proved to be the least prone to aggregate in the examined conditions most likely due to the expansive biological matrix provided by the green tea extract acting as a stabilizer. AgNP@MSN showed heavy aggregation in the majority of the experimental conditions highlighting the crucial importance of surface functionalization of silica carriers in drug delivery systems.

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Bioceramic microspheres based on $\text{Si}_3\text{N}_4 - \text{Ca}_3(\text{PO}_4)_2$

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The article describes material prepared for biocements for medical use in human body. Biocements are using for example for bone implants or as a filling of bone cavities. The main method of preparation of Si_3N_4 microspheres in this article is based on flame synthesis. Different compositions of Si_3N_4 and $\text{Ca}_3(\text{PO}_4)_2$ powder mixtures were prepared and synthesized in $\text{CH}_4 + \text{O}_2$ flame. The aim was to characterize the influence of the proportion of Si_3N_4 and $\text{Ca}_3(\text{PO}_4)_2$ and the preparation of microspheres on their resulting chemical and crystalline phase composition and to determine the effect of these changes on the biological characteristics of the obtained microspheres.

Electrochemical characterization of bioceramic coatings on metallic implants

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The main requirements for medical implants are excellent mechanical strength, chemical stability, corrosion resistance and biocompatibility under physiological conditions. The corrosion stability of Ti based implant materials is mainly due to the stable TiO_2 layer formed on the surface, but it cannot be regarded as biocompatible material. The application of hydroxyapatite [$\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, HA_p] and other amorphous calcium phosphate coatings onto the surface of metallic implant devices offers the possibility of combining the strength of the metals with the bioactivity of the ceramics. Recently, much research interest has evolved in the pulse electrodeposition (PED) technique since this technique holds the potential to achieve highly crystalline deposits even at the low temperatures with low solubility in body fluids and low residual stresses.

The realized biodegradable coatings can gradually be dissolved, absorbed, consumed or extracted after the bone tissue healing. Normally the human bone contains trace amount of mineral phase such as sodium (Na), magnesium (Mg), strontium (Sr), zinc (Zn) and silicon (Si). The synthetic HA_p structure is also flexible in acceptance of mineral substitutions. The metallic and ionic silver can be incorporated into or co-deposited with some biomaterials such as Zn, Mg, Sr and hydroxyapatite. One major advantage of hydroxyapatite coatings loaded with silver and other biocompatible minerals is that they promote bone growth, accelerate the wound healing process increasing the biocompatibility ability of the implant materials.

In this work we deposited hydroxyapatite (HAp) and other amorphous bioactive Ca-phosphate layers doped with multi element nanoparticles by pulse current applying different deposition parameters onto commonly used Ti alloy implant material (TiAl6V4) from appropriate solutions. The effect of deposition parameters (composition of electrolyte, anode material, pulse current t_{on} and t_{off} time, current density) on morphology as well as chemical and degradable properties of deposited layers have been investigated.

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Mimicking biological mechanical behavior by a bioactive lactose-modified chitosan

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Keywords: bioactivity, mechanotransduction, lactose-modified chitosan, stress-induced reticulation.

Mechanical factors are able to influence biological processes according to a mechanism termed mechanotransduction.¹ There is a growing effort to recreate mechanical properties of natural tissues by exploiting synthetic polymers.² CTL60 (also termed as Chitlac) is a lactose-modified chitosan, which showed interesting biological and physical-chemical features; for instance it was found to form highly homogeneous colloidal coacervates.³ Its bioactivity was demonstrated *vs.* different cell types such as chondrocytes,⁴ osteoblasts⁵ and neural cells.⁶ The biological significance of CTL60 traced back to its interaction with Galectin-1, which bridges polymer chains and cell surface integrins.⁷ Rheological behavior of CTL60 upon treatment with boric acid (used as cross-linking agent) was explored and an uncommonly high dependence in the scaling law between the zero-shear viscosity and the concentration of CTL60 emerged, pointing to interesting potential implications in the field of viscosupplementation.⁸ When CTL60 was treated with boric acid, strain hardening effects and non-linear response to stress were also noticed. Such trends are considered as hallmarks of cytoskeletal and biopolymer networks.⁹⁻¹⁰ Resulting matrices pointed at hybrid sol-gel samples and

showed rheological properties similar to collagen and neurofilaments.⁹ The elastic modulus was found to markedly increase after stimulation at constant stress and frequency for few hours. Mechanical properties of the solicited sample were higher than the non-stimulated sample; the stimulated sample behaved like a weak gel and, therefore, a stress induced re-tilation occurred. By NMR it was possible to identify the interaction sites of boric acid binding to polymer. Mannitol was selected as competitor for boric acid binding to slow down gelation kinetics. Surprisingly, mechanical properties were slightly higher by using a certain mannitol concentration.

Optical Tweezers were used as micro-rheology technique on dilute polymer solutions; experimental findings showed that dynamic viscosity of the polymer treated with boric acid was about 20% higher than polymer alone. Giving the peculiar properties of the obtained system, bioactivity of CTL60 could be linked to a mechanotransduction mechanism mediated by the presence of cross-linking agents.

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Novel printed biodegradable fiducial markers – how the preparation process influence on the physical properties

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Keywords: biodegradable polymers, X-rays imaging, contrast properties,

Nowadays, there is huge interest in monitoring of implants by diagnostic techniques. Metallic and ceramic biomaterials have different radiopaque properties than tissues of human body what makes them easy in visualisation. However, in the case of polymeric biomaterials, there are difficulties in distinction of the material from surrounding soft tissue [1]. When compared to bone – they are radiolucent. Presented problems were the reason to try to increase the radiopaque properties of widely known biodegradable polymer – polycaprolactone (PCL). Similar approach may be used for any other biodegradable polyester.

Presented study was focussed on investigation how different parameters of preparation process of PCL-iodohexol (IHEx) influence the properties of final printed product. IHEx is an organic iodinated compound widely used in X-rays diagnostics. Its nature makes it perfect

candidate for this application. Compared to other potential contrast fillers (like barium sulphate, gold nanoparticles etc.) it is easily soluble in polar solvents, what can be an advantage in obtaining the desired microstructure of material.

In this study series of PCL-IHEX were prepared. The influence of IHEX incorporation method (one or two solvents methods), solution mixing speed, time of adding the polymer, the concentration of IHEX and the order of procedure on the materials properties were analysed. In two solvents approach modified nanoprecipitation method was used. Two miscible solvents: one to dissolve the PCL, second to dissolve the IHEX were used. After mixing all compounds, the solution was dried. Bioscaffolder (SYSENG) was used to fabricate the structures with keeping the same printing parameters for all materials. Then, processability, microstructure, molecular mass distribution, thermal properties, degree of crystallinity and visibility in X-rays were investigated.

Results showed that molecular mass distribution was not affected by the preparation process. However, the influence of several parameters on microstructure as well as on processability was observed. Summarizing, the PLC-IHEX is promising contrasting biomaterial. Nevertheless, the microstructure of material in very important parameter from stability of visualisation and degradation point of view.

Figures

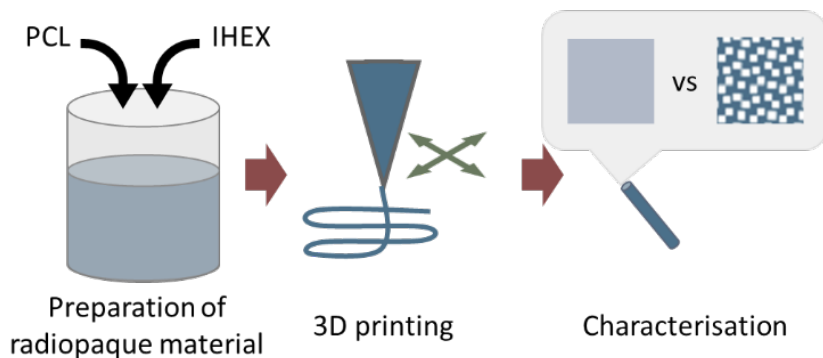


Figure 1. The scope of research

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Enhancing storage stability and bioactivity of small-molecule biopharmaceuticals via nanocomplexation with polyelectrolytes

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Keywords: nanoparticles, amorphous, drug complexation, polyelectrolytes, biopharmaceuticals

While the numerous therapeutic bioactivities of small-molecule biopharmaceuticals (e.g. organic herbs, plasmid DNA, vaccine) have been well established, their efficacy upon their clinical applications has been greatly limited by their poor physicochemical stability, particularly during long-term storage in their shelf-life, resulting in the loss of their bioactivities. Even worse, some biopharmaceuticals, such as plasmid DNA, are only stable if they are continuously stored in cold-chain systems. Several strategies have been pursued to improve the physicochemical stability of small-molecule biopharmaceuticals and also to reduce the cold-chain dependence of biopharmaceuticals. Among these strategies, encapsulation into carrier particles exhibiting high physicochemical stability (e.g. polymers with high glass transition temperature) has been the most widely pursued. However, this strategy has a major drawback in its low encapsulation efficiency, resulting in a significant loss of raw materials and also low payload of the active ingredients. Herein we present a new strategy to enhance the stability of biopharmaceuticals via their complexation with natural polyelectrolytes and well-known stable polymers (e.g. hydroxymethylcellulose, polyvinylpyrrolidone). The complexation was driven by either electrostatic or hydrophobic interactions, resulting in the self-assembly formation of nanoparticle complex (or nanoplex in short). The roles of the polyelectrolytes and polymers are (i) to molecularly bind the active ingredients to limit its molecular mobility during storage, and also (ii) attributed to its presence on the nanoplex surface, the polyelectrolytes also acts as enveloping matrix providing additional stability protection. The advantages conferred by the nanoplex system compared to encapsulation are (i) high complexation efficiency of the active ingredients (less wastage) and (ii) high active payload (less dose requirement). The present work used only natural polyelectrolytes from polysaccharide group (e.g. chitosan and dextran sulfate) to ensure the nanoplex biocompatibility. Two model systems will be presented, i.e. small-molecule organic herbs and plasmid DNA vaccine (potentially). The stability study was performed at an accelerated storage condition of 40°C and 75% relative humidity, which was equivalent to twelve-month storage at normal condition of 25°C and 60% relative humidity. The stability was examined by (i) thermal analysis, (ii) powder x-ray diffraction, (iii) spectroscopy, (iv) physical characteristics, and (iv) dissolution characteristics after storage. The results show that the incorporation of stable polymers, such as hydroxymethylcellulose, is crucial to enhance the long-term storage stability of biopharmaceuticals. Its inclusion into the nanoplex formation has minimal effects on the physical characteristics of the final products, where only the payload decreased slightly. The present results successfully established nano-complexation with polyelectrolytes as a new avenue to stabilize small-molecule biopharmaceuticals.

Effect of metal ion doping on electrical conductivity and antibacterial activity of ZnO nanoparticles

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Keywords: ZnO nanoparticles, Antibacterial effect, Electrical conductivity, Impedance spectroscopy

Zinc oxide is a typical n-type semiconductor with its wide band gap (3.37 eV) and has received attention due to its optical, electrical and chemical properties[1-2]. As the antibacterial effect of ZnO nanoparticles (ZnO NPs) has been recently reported, many studies have been conducted to understand the antibacterial mechanism of ZnO NPs. In addition, there have been lots of attempts to improve antibacterial activity through additional metal doping (e.g., Mn^{2+} , Al^{3+} and Ce^{4+}) on ZnO NPs for application in the medical field [3]. It is highly important to understand the effect of dopant on ZnO NPs. However, so far, very little attention has been paid to the mechanism of antibacterial activity by additional dopants.

In this study, the antibacterial mechanism of doped ZnO NPs has been systematically investigated in the view point of positive charge carriers (oxygen vacancies, electron holes or proton) via electrochemical impedance spectroscopy measurements. The particle size and structure of synthesized ZnO NPs were confirmed by XRD, XPS and SEM. The antibacterial effects of pure ZnO NPs and doped ZnO NPs on *Staphylococcus* (*S. aureus*) and *Escherichia coli* (*E. coli*) and the correlation between electrical properties and antibacterial activity will be discussed.

Acknowledgments

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Effect of various synthesis parameters and characterisation of a new family of calcium phosphate amorphous biomaterials

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Keywords: bioactive glasses, calcium phosphate, soft chemistry, gel, amorphous materials

Silicate-based bioactive glasses are one of the most promising materials used in the fields of bone and soft tissue substitution [1-2]. Despite promising properties [3], phosphate-based glasses have been poorly studied especially when synthesised in mild conditions. Very recently, a new family of calcium ortho-/pyro- phosphate glasses has been prepared by soft chemistry using salt precursors, water as solvent and low temperature [4]. The objective of the present study is to evaluate the effect of various synthesis parameters such as the initial ortho-/pyro- ratio (o/p) in solution, the gel washing step and the final drying temperature on the obtained material. The material is prepared using calcium and pyro/orthophosphate salts separated solutions (CaCl_2 and $\text{Na}_4\text{P}_2\text{O}_7/\text{Na}_3\text{PO}_4$). The calcium solution is added to the phosphate solution. The mixture is then centrifuged and a gel is obtained after a washing step. Finally, the gel is dried. The effect of the initial o/p in the phosphate solution (0 to 8), of the number of washing steps (0 to 2) and of the temperature of the final drying treatment (ambient temperature to 200°C) has been investigated. The as-prepared materials have been characterised by SEM (Fig. 1), XRD, ^{31}P solid-state NMR, Raman and FTIR spectroscopies. We showed that depending on the initial o/p in solution, the material can be crystalline (o/p = 0), amorphous for the lowest ratios ($0 < \text{o/p} < 8$) or partially crystalline for the highest ratio (o/p = 8). A thorough study using ^{31}P solid state NMR has led to qualitative and quantitative data on the structure and composition of the different materials prepared. It was demonstrated that the evolution of a transient amorphous phase towards a crystalline phase could be avoided by the washing step. The amorphous structure could be explained by the degrees of freedom of pyrophosphate entities (P-O-P angle) and the inhibitory effect of orthophosphate ions on calcium pyrophosphate phase crystallisation and vice versa. This study demonstrated that the o/p in solution and the gel washing step are two crucial parameters that can lead to amorphous, crystalline or mixed materials. In addition, we showed that these amorphous materials are stable up to quite high temperature (200°C) which opens interesting perspectives for their processing. Even if further investigations will be necessary to fully understand the formation process and structure of these materials, this new family of amorphous materials leads to promising and versatile materials to be used as monolith or powder for bone filling applications.

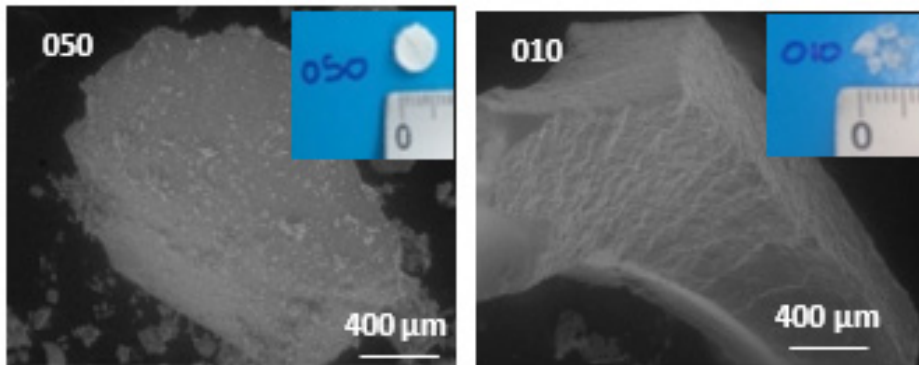


Fig. 1: Macroscopic and microscopic photos of two different compositions of ortho-/pyrophosphate amorphous materials

Acknowledgments

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Microstructure and mechanical characterization of thin bioactive PEO coatings fabricated on UFG CP Ti

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Plasma Electrolytic Oxidation (PEO) coatings were deposited on Ultra-Fine Grained Commercially Pure Ti (UFG CP Ti). The objective of the deposited coatings is to provide enhanced bioactivity to the protective anodic TiO₂ film by the incorporation of Ca and P ions. Three coatings were deposited under the same processing conditions using three electrolytes containing Ca and P species with different pH levels. All the deposited coatings were characterized by a high surface roughness and high porosity. Nanopores and random-shape micro-cavities were found through their thickness. However, the surface and through-thickness morphologies developed were different from one coating to another. The composition and the phases present were also evaluated by means of EDX and XRD, respectively. The

presence and the amount of Ca and P elements depended on the electrolyte. Crystalline TiO₂ anatase and rutile phases were found, being Anatase the predominant phase in all the coatings. The sample corresponding to the lowest pH electrolyte did not present the rutile phase. In the other two samples, rutile had a significant presence in one of the samples while in the other one its contribution was minimal. The mechanical characterization of the coatings was carried out by nanoindentation. This represents a challenge, due to the inherent porosity and large roughness characteristic of PEO coatings. To overcome this challenge, low-depth nanoindentations (~150 nm) were performed on the cross-section of the coatings, and care was taken to minimize the influence of micro-cavities. The correlations between the mechanical properties and the phase composition of the coatings will be presented.

Comparison of the dissolution behavior of silicate, borosilicate and borate bioactive glasses under static and dynamic conditions

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Keywords: borate, borosilicate, bioactive glass, dynamic dissolution

Key properties of bioactive glasses include their ability to dissolve and release ions when immersed in an aqueous environment, as well as the formation of an apatite surface layer on the glass during dissolution [1]. Due to the lower chemical durability of borate glasses and their ability to transform rapidly to hydroxyapatite, an increasing amount of research has started to focus on the use of borate glasses. Additionally, borosilicate glasses were developed by replacing partly silica by borate. By varying the ratio of silica and borate, it is possible to tailor the dissolution and transformation rate [2].

To evaluate the bioactivity of materials used in medical applications, the standard procedure is to immerse the materials in simulated body fluid (SBF) for a desired time under static conditions [3]. However, the human body is not a static environment. Therefore, the focus in this study was on the dissolution behavior of silicate, borate and borosilicate glasses in SBF under dynamic conditions in comparison to static conditions.

On the basis of the well-known 13-93 silicate glass, a borate glass and a borosilicate glass, where two-thirds silicate are replaced by borate, were produced. Prior to dissolution studies, the glasses were characterized using FTIR, SEM/EDX as well as XRD. Then, the dissolution of the particles (500-300 µm) were measured with a continuous flow-through-cell connected with a peristaltic pump with a pumping rate of 0.2 ml/min. Additionally, the samples were placed in SBF under static conditions following the standard protocol by Kokubo [4]. In order to compare the ions released under dynamic and static conditions, ICP measurements were done after immersing the glasses in SBF for up to 48h. The results show that the silicate

glass dissolves five times faster, whereas the borate glass dissolves 10 times faster under dynamic conditions compared to static conditions. Furthermore, differences in the release profiles were found.

Acknowledgments

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High performance magneto-fluorescent nanoparticles based on lanthanide (III) complexes for bioapplications

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Keywords: Nanoparticles, lanthanide complexes, bioimaging, luminescence, single-ion-magnetism.

Multifunctional nanoparticles (NPs) have shown great promise in targeted imaging and therapy. Their core/shell nanostructure is versatile for improving or integrating diverse functions. Lanthanide ions and their complexes exhibit not only magnetic properties but also unique optical properties that are a prerequisite for the fabrication of luminescent cores for NPs to be used as diagnostic markers in magnetically guided drug delivery and photodynamic therapy. The narrowness and the characteristic nature of emission bands, large Stokes shift, a long lifetime of the excited state and independence of the position of the emission bands on the ligand environment are the main advantages of the Ln(III) complexes compared to others. A covering of lanthanide complexes by silicate¹ or polyelectrolyte² shell is a promising approach both in terms of reducing their toxicity and also improving the hydrophilicity of the compounds.

We herein present a new family of lanthanide-based templates into polyelectrolyte capsules as a facile route to obtain stable in time luminescent colloids. The synthesis procedure of luminescent colloids has been performed as previously published²: a solution of the Ln compound in dimethylformamide was poured into the aqueous solution of (PSS - 1 mg/mL) and NaCl (0.5 M) at intensive stirring. The resulted aqueous dispersion has been further precipitated by centrifugation and the obtained precipitate has been separated and redispersed in aqueous solution. Dynamic light scattering measurements were performed and the effective hydrodynamic radius (R_H) calculated by the Einstein–Stokes relation, $D = k_B T / 6\pi\eta R_H$. Previous characterization of the Ln complexes using X ray powder diffraction, thermogravimetry and differential scanning calorimetry amongst other techniques will be presented. Luminescent and magnetic properties of the nanoparticles will be shown. Preliminary *in vitro* biological tests will be presented and discussed.

Acknowledgments

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Polysaccharides-based coacervates as drug delivery system for inflammatory diseases

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Keywords: polysaccharides, hyaluronan, coacervates, cartilage

The repair of damaged articular cartilage, which rarely heals spontaneously and develops osteoarthritic complications, is a common clinical issue^[1] and none of the available therapies can restore the hyaline cartilage surface beyond just fibrous repair. Over the last few years, ionically cross-linked colloidal polyelectrolyte (PE) coacervates have been in the limelight as a system for the encapsulation and delivery of drugs or active biomolecules^[2]. Water-soluble, biodegradable, polyelectrolyte coacervates offer numerous advantages over the conventional delivery forms, particularly so regarding the self assembly in aqueous solution without organic solvent, the rapid formation and high loading capacity, and the possibility to use biopolymers as native extracellular matrix protein such as heparin, elastin, hyaluronic acid, and chondroitin sulfate, that help to reduce toxicity and side effects^[3]. Polysaccharide-

based coacervates have, hitherto, been applied in the field of tissue engineering to enhance tissue regeneration by sustained and localized release of appropriate biological molecules, incorporated within the biodegradable nanostructure^[4]. Starting from this point, this project is mainly focused on the development and characterization of polymer-based nano-complexes, as a first step in the fulfillment of an injectable composite material for cartilage regeneration. Among different polymers, hyaluronan (HA), Chitosan and its derivatives seem to be very promising for their structural and well established biological role. The two differently charged polymers were used for the preparation of a binary mixture solution by mixing different amounts of a HA solution and a Chitosan/derivatives solution, respectively, hence obtaining different HA weight fractions. The different formulations were prepared by tuning physico-chemical parameters such as ionic strength, temperature, pH, charge density, weight ratio and molecular weight of the two interacting polysaccharides to find the best condition for coacervates preparation. First, the Transmittance (T) of the different solutions was measured; then the same solutions were investigated by Dynamic Light Scattering (DLS) for the evaluation of Z-average size, zeta-potential, polydispersity index. After that, the best formulation was selected to evaluate the stability of complexes in physiological conditions. Coacervates were tested *in vitro* for their biocompatibility and scavenger activity in order to better understand the capability of the system. Overall, these coacervates appear as promising biomaterials with interesting biological and physical properties for cartilage regeneration.

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Poster

Adsorbents for Arsenic Removal from Drinking Water

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Keywords: Arsenic, batch study, adsorption

The World Health Organization and the USEPA have determined that inorganic arsenic is a human carcinogen. Arsenic contaminated groundwater affects millions of people across the world in a range of countries. Arsenic exists in water as arsenite (As^{+3}) and arsenate (As^{+5}), with arsenite being the more toxic and more difficult to extract (Ntim and Mitra, 2011). Many poor and underdeveloped countries that use sand as their main filtration media face issues due to poor water quality, such as an excessive number of arsenic poisoning cases. Sand can remove many impurities from water, but is ineffective at removing arsenic. Arsenic levels in Bangladesh are four hundred times greater than the allowable level of 10 ppb set by the USEPA. Thus, there is a dire need to develop cost effective treatment technology for arsenic removal. The main objective of this study was to determine if arsenic can be readily adsorbed by iron oxide-coated sand, zirconium oxide-coated sand, activated carbon and carbon nanotube (CNT) adsorbents. The study also investigated combinations of these adsorbents for enhancing adsorption capacity. Batch adsorption studies in glass vials were conducted with an arsenic solution of 100 micrograms/liter and a varying range of adsorbent mass (10-250 mg). The Langmuir, Freundlich, Tempkin and Dubinin-Radushkevich (D-R) adsorption isotherm models were investigated for data analyses. The Langmuir isotherm model provided the best results for all the experimental data. Results indicated that iron oxide-coated sand had significantly higher adsorption capacity compared to the other adsorbents and their various combinations. Iron oxide-coated sand allowed for the removal of arsenic to drinking water limits and is the most cost effective adsorbent investigated in this study.

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Metal based particles detected in cytological mucus samples from the hypertrophic tissue of the inferior nasal turbinates

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Keywords: metal particles, cytological mucus, hypertrophic tissue, scanning electron microscopy, Raman microspectroscopy

The study addresses detection and analysis of metal based solid particles in cytological mucus samples from the surface of hypertrophic tissue in the inferior nasal turbinates in patients with chronic rhinitis, by scanning electron microscopy and Raman microspectroscopy. Since upper airways is the first part of the respiratory tract which is exposed to inhaled particles, it can be assumed that these particles may be deposited in this region. Particles detected by the above mentioned methods were in most cases in the size below 1 μm . Scanning electron microscopy revealed presence of solid particles based on iron in the majority of the analyzed samples of cytological mucus. Moreover, particles based on zinc, barium, chromium, manganese, titanium and nickel were found. Raman microspectroscopy as a suitable technique for phase microanalysis also revealed the presence of compounds based on especially amorphous carbon, graphite, calcium carbonate, titanium dioxide, ankerite, and barite in hypertrophic tissue in the inferior nasal turbinates. The obtained results showed that some solid particles detected in the mucus from the surface of hypertrophic tissues with their composition resembled the particles found in the hypertrophic tissue itself.

Acknowledgments

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3D printing of hierarchical porous/fibrous collagen structure for rapid remodeling of cell-laden scaffold using biodetergent hydrogel

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Keywords: 3D printing, collagen bioink, hierarchical scaffold, remodeling, tissue engineering

Conventionally in cell printing process, cells are imprisoned or immobilized in the 3D cell-containing hydrogel scaffolds due to crosslinking process [1, 2], which is one of the drawbacks of crosslinked polymer hydrogels for the cell migration and proliferation [3]. In this study, therefore, a biodetergent hydrogel has been used in the collagen-based bioink as a remodeling material for the collagen scaffold after printing. This hydrogel was used as 3D printing materials before and during printing. In addition, the remained collagen scaffold after the removal of the hydrogel has shown hierarchical porous and fibrous structure, such as micro pores and collagen nanofibers, which can enhance the cell activity including cell proliferation and cell release of human osteoblastlike cells (MG63) in the scaffold. Cell viability, migration, and proliferation were evaluated, as well as other effects of the biodetergent hydrogel on the collagen structure, such as variance in mechanical or rheological properties, were also assessed. We believe that these results may become a standard for more rapid and effective in vitro culture system by enhancing the cell activities, including cell migration and proliferation, of the cell-printed regenerative biomaterials.

Acknowledgments

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Deposition of thin metal layers on chitosan films

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Keywords: chitosan, plasma treatment, sputtering, silver nanoparticles

Chitin is one of the most abundant polysaccharides on Earth. It is very important for production of chitosan, because chitosan occurs in nature very rarely. Chitosan is produced by chemical or enzymatical deacetylation of chitin which is found in invertebrates as crustacean shells or insect cuticles. Nowadays, chitosan is well known polycationic biopolymer with a wide spectrum of biological activities including antibacterial and antifungal effects and is used in both industry and pharmacy. Chitosan is also non-toxic, biocompatible and biodegradable. The composition is often advantageous, because is very similar and in some cases may even coincide with the tissue of human body [1].

The present work is focused on preparation of chitosan films which were modified by plasma treatment and sputtering the silver nanoparticles which made silver layers. The films were doped with silver nanoparticles for enhancement of antibacterial properties. Properties of these films were measured and analyzed with a few techniques. The presence of silver nanoparticles was studied by X-ray photoelectron spectroscopy. Wettability and the surface morphology of the films were evaluated. Antibacterial activity of solid films with silver nanoparticles was tested by drip test on two bacterial strains, Gram-positive *Staphylococcus epidermidis* and Gram-negative *Escherichia coli*. The films exhibited strong antibacterial activity against both bacterial strains. Solid films were dissolved and then the solution was observed by transmission electron microscopy. Dissolution tests of prepared films were evaluated in different pH range. Concentration of the AgNPs released into the solution during dissolution was studied by atomic absorption spectroscopy. The presence of AgNPs was confirmed both in the solid films and in the solutions by the above mentioned methods. Our research was aiming on use of these films in medicine as a new type of wound dressing with antibacterial properties. These films could be used as a wound dressing, antimicrobial packaging material or as a long-term storage of AgNPs for various applications.

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Metal nanostructures for biological applications: Comparison of Ag and Pd biological suitability and material properties

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Keywords: silver, palladium, nanostructures, biocompatible polymers, sputtering, annealing, antibacterial effects, cytotoxicity, medical applications

Nowadays, it is well known that nanostructured noble metals, especially coatings-forming ones, exhibit strong antibacterial activity against broad spectrum of microorganisms, concurrently without bacterial resistance. Therefore surface modifications of biocompatible polymers by nanostructured antibacterial coatings are effectively used in medical applications, in which silver is a “gold” standard. This type of polymer treating might not only provide significant antibacterial effects, but also enhance biocompatibility of the resulting composite. Relatively easy way producing the simplest nanolayers (NLs), often having flat surface with limited bactericidal effect, is sputtering. One can, however, increase the surface area of such coatings by their thermally induced transformation into nanoisland-like structures (NIs), which might lead to increased antibacterial effects and biocompatibility of resulting composites. Moreover, cell cultures generally prefer rougher substrates, leading to their better adhesion and proliferation. Thus, the surface modifications enable a direct control over the biological suitability of polymeric materials, which is in most, significantly enhanced [1].

Silver and palladium NLs were sputtered on the surface of polyethylene naphthalate (PEN), material frequently used in health-care industry for the preparation of strong and thin catheters and other medical devices. Subsequently, those NLs were annealed and transformed into corresponding NIs. The surfaces of all types of nanostructures (both Ag and Pd ones), were broadly characterized by x-ray photoelectron spectroscopy and atomic force microscopy. Transformation of continuous NLs into isolated NIs homogeneously distributed over PEN surface was confirmed. Moreover, size and shape of metal NIs might be effectively controlled by the thickness of NLs preceding the annealing process. The biological suitability of commonly used Ag was compared with Pd by their mutual antibacterial and cytotoxicity testing using drop plate method with Gram-negative bacteria *Escherichia coli* and Gram-positive *Staphylococcus epidermidis*, and WST-1 assay with mouse embryonic fibroblasts L929, respectively. Surprisingly, in case of both examined metals, the increase of their surface roughness unfortunately did not lead to enhanced antibacterial effects, nor biocompatibility improvement. The first mentioned phenomenon often emerges due to so-called „curtain effect“, in which embedding of metal clusters into the polymer interior, while overlapping the formed islands with a thin film polymer reaching almost the tops of individual metal islands, occurs. The latter one is often observed due to the relatively high sensitivity of L929 cell line to rougher surfaces.

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Characterization of electrosprayed nanohydroxyapatite coatings on Si_3N_4 ceramic

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Synthesized nanohydroxyapatite (nHA) has high bioactivity and biocompatibility since the structure of this mineral is very similar to the inorganic constituent of bones and teeth. Using a bioactive and highly biocompatible coating on the implant's surface could help avoid the rejection in the early few days, after the operation. The aim of this study was to produce thin nHA coating on silicon nitride (Si_3N_4) implant material with electrospray deposition (ESD). In recent years Si_3N_4 bioceramic is developed for use as femoral heads in joint replacements and is being considered for use as dental implants. First, the Si_3N_4 substrates were realized by hot isostatic pressing and as the second step the nanosized nHA particles were produced from eggshells. The applied electrospraying process showed an unstable Taylor-cone due to the low electrical conductivity of the Si_3N_4 , therefore continuous voltage adjustment was necessary for the coating process stabilisation. The nHA coating was non-uniform on the Si_3N_4 surface, and did not form a continuous layer. Electrical conductive Si_3N_4 was also introduced in our study. In order to increase the electrical conductivity, we doped the Si_3N_4 ceramic with carbon nanotubes (CNT). As a result, 5 μm thick nHA coating was formed on the CNT- Si_3N_4 substrate.

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Modelling and Characterization

Oral

Damage mechanisms in machinery components of advanced high strength steels and their correlation to laboratory testing

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Keywords: Impact fatigue, Sliding wear, Swift, Softs

In several industrial applications, mechanical components are subjected to a combination of dynamic impact loading and sliding, leading to Impact Fatigue and Wear (IFW). The fatigue damage may either be a part of, or induce wear, or wear may induce an initial damage that later may continue into fatigue damage and failure. Components used in percussive rotating rock drilling devices and punching of high strength steel are examples of such. In the present work two different custom built test rigs, Sliding Impact Wear Tester (SWIFT) and Slider on Flat Surface (SOFS) are employed to emulate wear characteristics occurring during IFW. The test rigs allow combined impact at 50Hz and rotational sliding and also sliding varying sliding speed, single- or reciprocal sliding and friction monitoring, respectively. Damaged surfaces of the tested specimens and their surrounding areas were investigated in order to characterize the damage mechanism. The tested materials, carburizing steels and powder metallurgical high strength steel grades, were evaluated at different test conditions based on their performance in impacting and sliding contact conditions. The damage mechanisms observed in the present study were fatigue microcracking, plastic deformation and sliding wear. The results were found to be in a good agreement with damage mechanisms observed on the failed components used in rock drilling and punching applications supplied by industrial partners.

Oxidation behaviour of roughing mill work rolls and effect on thermomechanical fatigue

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Keywords: oxidation, steel, work roll, thermogravimetric analysis, thermomechanical fatigue, simulation

Due to the growth of high-strength steel production, rolls for hot rolling mills are facing higher mechanical loads and increasing contact temperatures. Therefore, continuous improvements of wear behaviour and process safety are required. The rolling process itself depends strongly on the entire tribological system, giving rise to abrasive and adhesive wear, rolling fatigue and tribochemical reactions. So a roll material improvement needs to consider all these aspects. Work rolls are in contact with the hot strip and therefore their surfaces are exposed to high alternating temperatures. In combination with water vapour, caused by roll cooling, this leads to an accelerated high-temperature corrosion of the material. In state of the art four-high rolling stands the work roll is also in contact with the backup roll. Hence, the strip-induced alternating temperature occurs in combination with backup roll-induced alternating stresses. Consequently, thermomechanical fatigue might be a suitable approach for a process simulation. In roughing mills, where several high deformation passes at highest temperatures are carried out, this approach becomes even more valid. Also, the impact of oxidation on the thermomechanical fatigue is of particular importance.

The objective of the study presented is to improve the understanding of damage mechanisms and damage evolution in rolling processes. By means of the finite element method (FEM) the periodic variation of temperature and stress at the roll surface needs to be calculated. Based on the results obtained, thermomechanical fatigue experiments must be carried out on a typical roughing work roll alloy. In order to understand the effect of oxidation processes, the high temperature corrosion behaviour of the alloy has been investigated first. This was done applying isothermal and thermally cyclic conditions in both, dry air and air mixed with water vapour. The oxidation kinetics were determined by means of continuous thermogravimetric analysis. The microstructure as well as the oxides formed were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) combined with energy dispersive X-ray spectroscopy (EDX). Furthermore, samples from worn out work rolls were taken and analysed, in order to compare real damage mechanisms with those occurring under laboratory conditions. The results of the microstructure analyses and the observed oxidation behaviour of the alloy will be presented and discussed in this contribution.

Equiaxed growth of Al-Cu dendrites: 3D phase-field simulations

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Keywords : Al-Cu, solidification, 3D simulations, phase-field, dendrites

We report on equiaxed dendritic solidification of Al-Cu alloys under isothermal conditions; the thermal gradient through the sample is almost zero. We focus on the interactions between two primary dendritic arms growing toward each other. Taking into account these conditions, a 3D phase-field code is developed and the numerical results [1] are compared to the experimental ones obtained in [2]. Because of the large computational cost when experimental concentrations are considered, samples with low concentrations are studied (fig 1, left) and growth laws are found. One can see that the tip velocities V and growth durations t are functions of the available distance between two primary arms growing toward each other L and the initial concentration of the sample c (fig 1, right). However, the product Vt is only function of L . We also show that the growth is always far from equilibrium because the sample is cooled down at a constant cooling rate during all the simulations, and as a result the product $\rho^2 V$, where ρ is the tip radius, is never constant. Finally, we show that the interactions between the arms of two dendrites become strong enough to stop the growth when the distance between them is about two solutal diffusion lengths and the time remaining to reach the end of the domain is of order of the diffusion time.

Figures

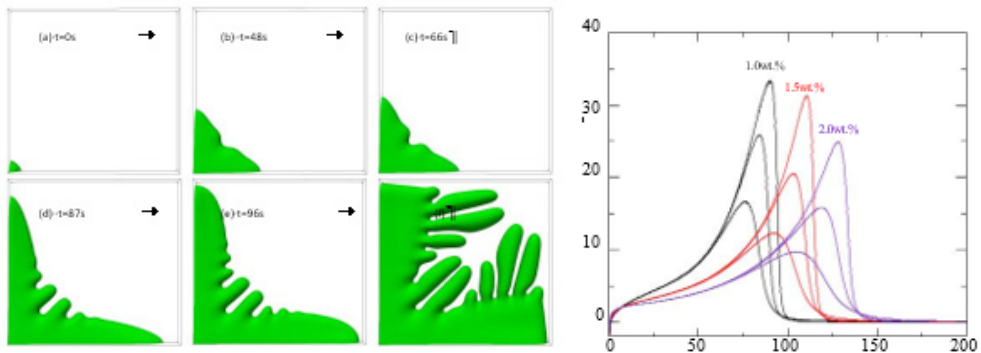


Figure 1: Six snapshots showing the growth of an Al-Cu dendrite simulated in a thin domain (left), numerical results for the velocity V of the dendrite tip as a function of time (right). [1]

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Microstructural characterization of an IN718 linear friction weld

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Linear Friction Welding is a solid-state joining process already in use as a niche technology for the assembly of bladed disks in aero-engines. The weld is obtained by the friction of a moving part in a reciprocating motion on a stationary part under a force. The local heat generation at the interface causes the materials to flow expelling along oxides and surface contaminants with plastically deformed material allowing parts coalescence in a thin metallurgical bond. Aerospace industry is showing a growing interest in the potential of LFW technology as a substitute in aircraft parts production to massive machining by material removal and is in concurrence with additive manufacturing processes. Moreover solid state welding is an interesting alternative avoiding solidification problems as grain boundary liquation, detrimental phase formation or micro-cracking problems in fusion zone.

In this study butt-weld test campaigns were carried out implying the joining of Ni-Fe-Cr superalloy Inconel 718 (10 x 80 x 70 mm³) solid blocks with a 10 x 80 mm² weld surface dimension. Alloy Inconel 718 is a γ'' (Ni₃Nb) precipitation hardenable material extensively used for service in extreme environment applications due to its good mechanical properties at high temperature and its excellent corrosion resistance. Two material heat treatment conditions were friction welded : ST and STA. The base material in ST condition consists in a microstructure composed of D=50 μ m γ -fcc austenitic grains with δ -orthorhombic intergranular Ni₃Nb phase. Two successive annealing at 720°C then 620°C were applied to obtain the STA condition yielding to the strengthening of the initial γ -fcc austenitic matrix by the quasi-coherent precipitation of γ'' (Ni₃Nb) disc-shaped particles evenly distributed in the fcc matrix.

During the welding process, severe thermo-mechanical loads are applied resulting in local microstructure and mechanical properties modifications. The characterization of the post-welded microstructures has been carried out by SEM and EBSD analysis on cross sectioned weld samples. Local inspection of the welding zone revealed the presence of a thermo-mechanical affected zone (TMAZ) that consists in a thick band of uniform dynamic recrystallized grains with an equiaxed grain morphology around the welding line, then a partially recrystallized zone composed of fine recrystallized grains distributed along the grain boundaries of elongated grains followed by a zone made of deformed grains remaining from the initial microstructure. Phase and microstructure changes across the joint were shown to have influence on hardness properties by micro-hardness tests across the welds.

As many nickel based alloys controlling the microstructure and the grain size and distribution after hot processing of IN718 is a key factor in the optimization of the mechanical properties of the material. To that end, post weld heat treatments were applied in an attempt to recover a homogeneous microstructure across the weld with interesting mechanical properties for in-use applications.

EXAFS and XANES techniques for studying the type of defects in Ti-zeolites and their role in catalysis

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Keywords: Zeolites, TS-1, XANES simulation, EXAFS analysis, HPPO reaction, NH₃ adsorption

Titanium Silicalite-1 (TS-1) is a synthetic zeolite where Ti atoms are introduced as isomorphous substituent of tetrahedral Si sites in a purely siliceous MFI zeolite.¹ TS-1 represents the prototype of a single catalyst due to its crystalline structure and its well-defined Ti sites. Whereas past investigation focused on perfect tetrahedral Ti (TS-1a), nowadays the focus is progressively moving toward the characterization of non-perfect Ti species (TS-1b) and their role in catalysis. With the exception of bulk TiO₂, the structure of these non-perfect species is still debated. In the present work, we discuss the capability of the various Ti sites to adsorb ammonia, a point that is relevant in some important reactions catalyzed by TS-1 such as cyclohexanone ammoximation. Furthermore, it has been followed also the HPPO (hydrogen-peroxide-to-propylene-oxide) reaction that is one of the most investigated processes as it is an economically and ecologically superior technology since potentially water is the only waste product. Here we report the extended x-ray absorption fine structure (EXAFS) spectroscopy and x-ray absorption near-edge spectroscopy (XANES) results at Ti K-edge of different samples of TS-1 after the contact with NH₃ feed. Moreover, we studied the Ti L_{2,3}-edge of the same compounds, more sensitive in elucidating the nature of the chemical bonds of the active metal site with the reactants, during the dehydration and the reaction with NH₃, H₂O₂ and C₃H₆. All the experimental data were supported by XANES simulation using the DFT-optimized clusters of the most stable Ti species in MFI (24 independent sites).² The EXAFS experiment was carried on *in situ* at Ti K-edge of TS-1a and TS-1b hydrated, activated at 500°C and after the NH₃ interaction at XAFS beamline (Elettra, Trieste). In addition, the XANES experiment at the L_{2,3}-edges of Ti was done in the new *operando* setup at APE-HE beamline at Elettra. The XANES spectra were collected for investigating the relation between the type of Ti species and their reactivity with some reagents (NH₃, H₂O₂ and C₃H₆). The data were analyzed with a theoretical support: DFT optimization and XANES simulation.

In summary we present a detailed analysis of EXAFS and XANES experiment conducted on the perfect tetrahedral and defective TS-1 with different treatments. These measurements

have permitted to identify the kind of Ti species presented in the TS-1b compared with TS-1a. Moreover, the adsorption geometry of NH_3 on Ti species was obtained. In addition, we also derived important information on the reactivity of Ti in TS-1 with H_2O_2 and C_3H_6 .

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Simulation of crystallographic texture evaluation during recrystallization by cellular automaton method

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After cold rolling of aluminum alloys subsequent heat treatment usually introduced into the process line to reduce the hardness, and bring back the deformability. During this process, the aluminum alloy recovers (if it can), then recrystallize. During the recrystallization, the texture components are changing. The cellular automata are one of the most frequently used method to simulate this recrystallization process, but many researches focus only the recrystallization's kinetics. The cellular automata give the complex nature of this process with a simple implementation. This study introduces a simple and fast one-dimension simulation method which makes possible to incorporate the texture components to the calculations.

Anisotropic slip activation in ZrB_2 ceramic grains during nanoindentation

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Anisotropic dislocation nucleation of differently oriented zirconium diboride (ZrB_2) grains was investigated during nanoindentation in a polycrystalline ZrB_2 -based ceramic composite. Nanoindentation was carried out on constituents of a ZrB_2 -SiC composite using a Berkovich tip at room temperature. Prior to testing, the crystallographic orientations of ZrB_2 grains were determined by electron-backscatter diffraction (EBSD). Based on the Hertzian stress analysis

of the measured strain bursts, the critical resolved shear stresses for slip systems of $\{101\bar{0}\}\langle112\bar{0}\rangle$, $\{101\bar{0}\}[0001]$ and $\{101\bar{0}\}\langle112\bar{3}\rangle$ types were determined. Homogeneous dislocation nucleation was revealed with near theoretical critical resolved shear stress (~ 35 GPa) for each slip system. The analysis of the orientation dependence of the calculated maximal resolved shear stress values revealed anisotropic slip activation in ZrB_2 grains. It is inferred that dislocations may be nucleated homogeneously only in the $\{101\bar{0}\}[0001]$ slip systems close to basal orientation (below tilt angle of $\Phi \sim 30^\circ$ from the basal orientation). In the region of $\Phi \sim 30^\circ - 70^\circ$, the simultaneous activation of $\{101\bar{0}\}[0001]$ and $\{101\bar{0}\}\langle112\bar{3}\rangle$ slip system families is found while close to prismatic orientation (over $\Phi \sim 70^\circ$), homogeneous dislocation nucleation is the most likely in the $\{101\bar{0}\}\langle112\bar{3}\rangle$ and $\{101\bar{0}\}\langle112\bar{0}\rangle$ slip systems.

Analysis of segregation effects during heat treatments and the influence on the formation of second phase particles in Titanium-834

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Keywords: Titanium alloy, heat treatment, micro-chemical analysis, SEM, APT

Titanium 834 is a near- α Ti-alloy and is used for applications in jet engine compressors, operating at temperatures up to 600°C . The alloy is often processed to have a complex bi-modal microstructure and is strengthened by the formation of two different types of second phase particles, intragranular, nano-scale Ti_3Al precipitation (α_2) and silicon rich particles forming at the interface between α and β -phase. The former is known to have a particular effect on strength, but also promotes slip planarity and therefore can affect the lifetime of engine components made from this material.

The present work focusses on detailed characterisation of the microstructure for different heat-treatment conditions using Electron Probe Micro Analysis (EPMA), Wavelength Dispersive Spectroscopy (WDS) and Atom Probe Tomography (APT).

Variations in the cooling rate after the solution heat treatment have been used to adjust the spacing of the secondary- α and β ligament width, while slower cooling rates also result in a slight growth of primary- α -grains. Detailed chemical mapping has shown significant segregation effects of alloying elements within primary- α for slow cooling rates, with the centre of the grain being enriched and the outer area having a lower concentration of α -stabilising elements. The variation of Al and Sn concentrations within primary- α is expected to have a strong effect on the formation and composition of α_2 -precipitates, which was studied by FIB-machining a cantilever beam through the cross-section of a primary- α -grain and subsequent site-specific APT. The results demonstrate precipitation reduced zones near primary- α grain boundaries for cooling rates typically used when processing Titanium 834.

Post-processing technique for macrozones definition in Ti834 alloy textures

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Keywords: Macrozone, Texture, Titanium, EBSD

A post-processing technique is in development for automatic detection of large regions with similar orientation to define macrozones. Within a bimodal titanium alloy, macrozones are generated during forging where a majority of primary alpha grains share a similar crystallographic orientation. While the performance properties provided by a bimodal microstructure are required for high-pressure parts applications, the unavoidable formation of strong HCP textured regions drastically reduces fatigue life. Texture characterization is carried out by the Electron Back Scattered Diffraction (EBSD) technique where the resulting orientation data is commonly displayed as an orientation map. Such map provides information about the spatial orientation distribution of a given crystal structure but further data analysis needs to be done to identify macrozones. The data was analysed using a code implemented in MatLab to identify macrozones from EBSD datasets. The code developed appears to be mostly successful and it will be a suitable tool for macrotexture characterization. It will allow an easier investigation on the effect of the thermomechanical processing parameters on the origin, size and density of macrozones, which are responsible for increasing the susceptibility of dwell fatigue. The computational routine created will be used as tool during the upcoming research as support of the EBSD analysis and the Channel5 software, widely used for texture analysis.

Correlation between substrate ion fluxes and properties of DLC films deposited by DOMS in Ar and Ar + Ne plasmas

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Keywords: DOMS, HiPIMS, DLC, Flat Probe

High Power Impulse Magnetron Sputtering (HiPIMS) has been under consideration for hard DLC thin films deposition in recent years [1-3]. In HiPIMS, a large fraction of sputtered atoms is ionized, thanks to 2–3 orders of magnitude higher plasma densities than in DCMS [4, 5]. The major driver to use HiPIMS for DLC deposition is the possibility of C ions formation in the plasma and subsequent use of these ions to bombard the substrate in a similar way to what is presently done in ARC deposition. The main strategy to achieve ionization of the sputtered species in HiPIMS is to promote electron impact ionization through increasing the plasma density. This route has been successfully implemented for many metals, i. e., for elements which exhibit ionization energies between 6 and 8 eV. However, this strategy is not effective for C which exhibit a significantly higher ionization energy (11.6 eV) and lower ionization cross-section. As a result, in HiPIMS discharges with a C target the C+/C ratio does not exceed 5 % [6]. An alternative strategy to increase the ionization reaction of the sputtered carbon species, proposed by Aijaz et al., consists in increasing the electron temperature of the discharge [6]. This can be achieved by using gases with higher ionization energy than Ar (15.6 eV) such as Ne (21.56 eV) in the plasma.

Several authors have reported that adding Ne to the plasma is beneficial for the mechanical and tribological properties of DLC films [7, 8]. In this work the effect of adding Ne to a pure Ar discharge on the ion fluxes measured at the substrate is investigated using a flat probe and correlated to the properties of DLC films deposited by DOMS.

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Characterising coherent Fe-Cu interfaces using density functional theory (DFT)

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Keywords: ferritic steels, embrittlement, precipitation, neutron irradiation, density functional theory (DFT)

Under neutron irradiation the alloying and impurity elements in a reactor pressure vessel (RPV) steel can precipitate out of solution forming solute clusters (commonly comprised of Cu, Mn and Ni). These clusters act to embrittle the RPV steel causing a shift in its ductile-to-brittle transition temperature (DBTT) over its lifetime which is not well accounted for by current regulatory models.

Cu is a particularly pernicious impurity element in RPV steels, coming out of solution relatively easily to form nanoscale coherent bcc precipitates which can lead to significant embrittlement and may also assist the precipitation of other solute elements. As such, it is important that the form of these Cu precipitate clusters and their role in the precipitation of other solute elements be well understood in order to inform reactor operation and RPV alloy design.

Density functional theory (DFT) methods have been used in this work to simulate coherent interfaces between a Cu precipitate and the ferrite matrix. These techniques allow the structures and interfacial energies of the different crystallographic orientations to be calculated, providing insights into the favourability of Cu precipitate geometries. Additionally, the interfacial segregation behaviour of other common precipitating alloying elements, such as Ni and Mn, has also been investigated.

Investigation of deformation behaviour and fatigue damage evolution in nickel 201 using X-ray Laue diffraction and a 2D energy dispersive detector

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Keywords: mechanical behaviour of materials, fatigue, dislocations, TEM, Laue Method, pn-CCD

The aim of this work is to expand the applicability of the energy dispersive Laue diffraction method [1] to study fatigue in polycrystalline metals. Using polychromatic X-rays and a special type of a charge-coupled device (pn-CCD) it is possible to measure structure and texture of a large ensemble of grains and to calculate the strain state and the dislocation densities of each separate grain in the beam path in a single shot experiment. In order to verify the applicability of the method to the characterization of fatigue damage, it is necessary to investigate characteristic materials with different fatigue behaviour. For this purpose Nickel 201, which exhibits wavy dislocation glide behaviour was examined. Different saturation fatigue states (obtained at different plastic strain amplitudes) were investigated. Each fatigue state is characterized by a stabilized stress amplitude, which corresponds for a given plastic strain amplitude to a particular dislocation structure and dislocation density [2], figure 1. The verification of the microstructural change (bundle/veins transformed into cells) in the material was done by means of TEM examination. The fatigued and characterized samples were subsequently investigated with ultra-hard X-ray Laue diffraction at EDDI beamline of BESSY II synchrotron facility using a pn-CCD. Laue patterns were collected at different sites of the samples. Streaking in the diffraction peaks was observed to change in response to dislocation and strain state. The analysis of the spectral profiles of the Laue spots (E vs Bragg angle) along the streak axis allows for the calculation of the response of a set of exposed grains to external stress providing new insights into the damage evolution and its interdependence with grain orientation and preferential lattice plane motion (slip). Energy dispersive X-ray Laue diffraction is a promising new method for the characterization of a material. This method is fast and non-destructive allowing for in situ observation of damage evolution during mechanical loading. It also provides information from a collective set of grains at once and serves as a tool to validate plasticity models from polycrystals. However, more developments and further improvements of the numerical evaluation of the data need to be done to fully benefit from the technique.

Figures

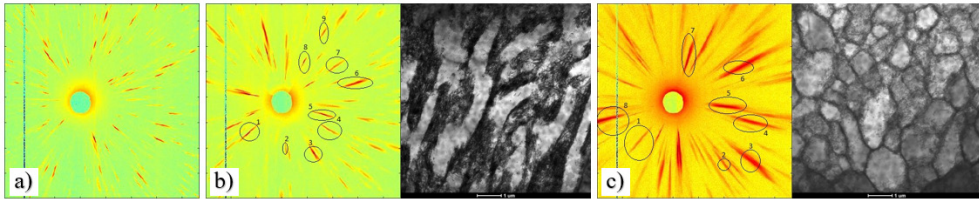


Fig. 1: Diffraction pattern and corresponding dislocation arrangement of a sample a) without prior fatigue loading, b) after fatigued at 0.15% elastic strain amplitude (~0.05% plastic strain) and c) at 0.3 % plastic strain amplitude

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Modelling of hot cracking phenomena of a laser welded Al-Cu-Li (2198) alloy

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Keywords: Al-Li alloy, Laser Beam Welding, Hot Cracking, Solidification, Microsegregation

The newly developed 2198 Al-Cu-Li alloy is a promising material for the aeronautical industry due to its unique properties. Li addition reduces density, improves damage tolerance, increases strength and Young's modulus making the material attractive to several industrial applications. It is thus of high significance the optimization of welding performance alongside with the minimization of problems encountered in fusion welding processes. The current work aims to contribute to the understanding of the mechanism of hot cracking phenomena (Fig. 1a) evolved during laser beam welding (LBW). The hot cracking is a limiting factor, towards the implementation of the LBW to the industrial applications of the alloy.

The investigation consists of the following steps:

- Determination of the solidification path by employing Thermocalc -Scheil model- in order to identify the type and the volume fraction of the phases formed during solidification^[1].
- Study of the dendrite's morphology adjacent to the crack path (Fig.1(b)) and verification of the Scheil model results. Field emission scanning electron microscopy (FESEM) equipped with EDS analysis was used. The results indicated a structure consists of equiaxial dendrites with severe segregation within the interdendritic region (Fig.1(c)).

- Study of the back-diffusion phenomena evolved during solidification via the Brody and Flemings model^{[2][3]}. The model provides quantitative predictions of micro-segregation in the vicinity of the cracks.

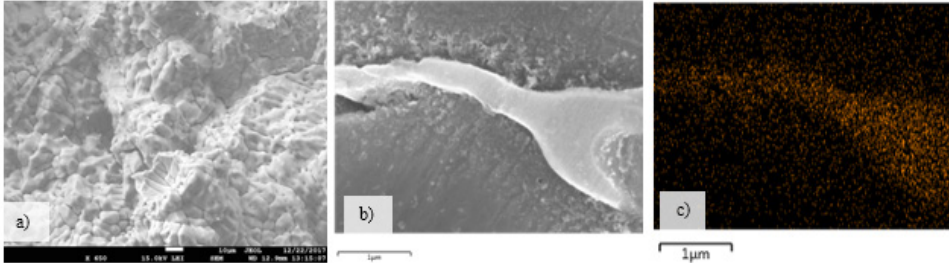


Fig. 1: a) Hot cracking along dendrites in the weld pool of LBW b) Interdendritic region within the crack propagation path c) Distribution of Cu at the grain boundaries.

The results allow the correlation of the experimental conditions to the tendency of hot cracking and thus contribute to the understanding of hot cracking phenomena and the optimization of the laser process.

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Evolution of precipitations in case hardening steel along the whole process chain

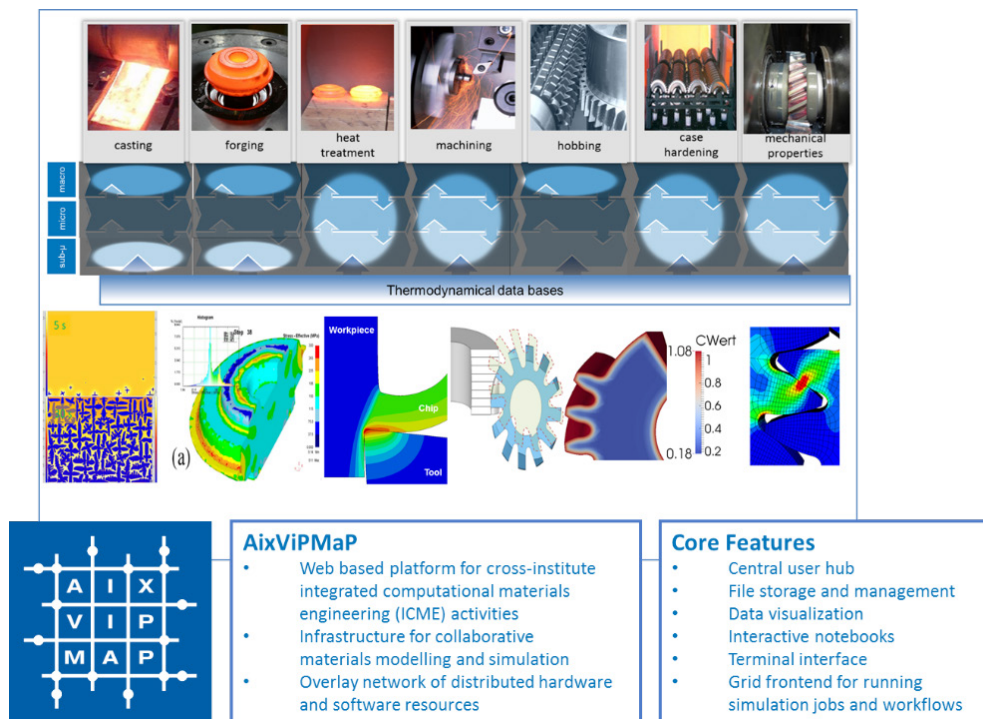
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As part of excellence cluster project, a simulation platform for integrative development of materials and process chains has been developed. This simulation platform is used for the numerical design of case hardened steel for large transmission gears. This study focuses on improving the understanding of influence of element segregation during casting, process chains and parameter on grain size stability during carburization process.

Solidification and phase evolutions are studied by using multi-component kinetic module of MactCalc. Multi-phase field modelling using MICRESS® is attempted to simulate the evolution of microstructure, spatially primary precipitates. The thermodynamic data and mobility data for the MICRESS simulation is assembled from ThermoCalc. Simulation in DEFORM analyze precipitation state during hot forging and heat treatment.

The combination simulations based on ICME (Integrated Computational Materials Engineering) platform and practical trials provides the understanding of steel characteristics on different process steps.



Influence of nickel substrate microstructure on diffusion processes in Ni/Al/Ni interconnection

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Keywords: diffusion soldering, intermetallic phases, microstructure, growth kinetics

Diffusion soldering (DS) is a promising technology for connecting components in many industries applications such as electronics, automotive or aerospace. This process bases on isothermal solidification when the reaction between substrates in solid state and a layer of liquid solder takes place. The diffusion soldering parameters such as temperature and time of annealing strongly affect the microstructure and phase composition of the interconnection. As

a result of DS the diffusion zone is composed of one or many intermetallic phases making it thermally stable up to the melting temperature of the intermetallics that totally fill the joined area. The literature data showed that due to Al-Ni reaction at the temperature not much above the aluminium melting temperature (660 °C) few phases can be formed. According to the equilibrium phase diagram these are in the order of their creation: Al_3Ni , Al_3Ni_2 , AlNi , AlNi_3 .

The subject of this study were Ni/Al/Ni assemblies annealed at 720 °C using different nickel substrates with varied grain size, and therefore, different amount of grain boundaries. At the starting point of the joining process the diffusion takes place in liquid-solid state, however, later on it proceeds through the formed intermetallic layers in the solid state. As a starting point the classical scanning electron microscopy observations and energy dispersive x-ray spectroscopy analysis were carried out for each diffusion-soldered joint, revealing its phase composition and thickness of particular intermetallics layers and also the chemical composition changes across them. As a next step, the electron backscattered diffraction technique was used to expose the amount and character of the grain boundaries and to correlate it with the interface composition and thickness. Figure 1 shows the comparison of the diffusion zone formed at the same experimental conditions (720 °C, 5h, vacuum) but using Ni substrates of different amount of grain boundaries.

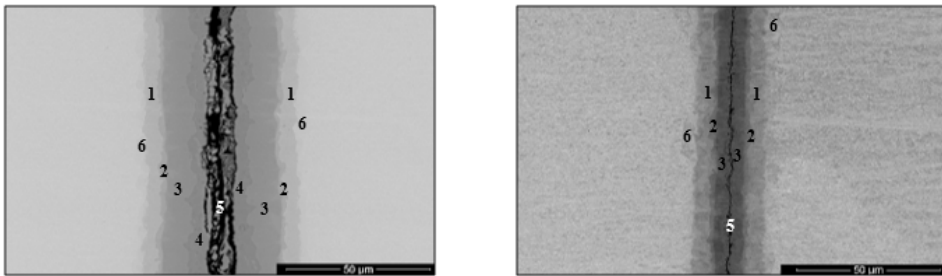


Fig.1. The backscattered electron SEM images of Ni/Al/Ni interconnection zone, where Ni substrates varied in amount of grain boundaries (more of them – left side or less – right side). Numbers 1-6 denote particular intermetallic phases: 1- Ni_3Al , 2- Ni_3Al_2 , 3- AlNi , 4- $\text{AlNi}(\text{Ni}-\text{needy})$, 5- $(\text{Al})+\text{Al}_3\text{Ni}$, 6-Ni solid solution.

Acknowledgments

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The effect of work hardening on the time and temperature dependent aging behaviour of a maraging steel

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Keywords: maraging steel, cold working, aging behaviour

The effect of work hardening on the aging behaviour has been investigated on the example of a maraging steel 12Cr-9Ni-4Mo-2Cu. After melting and casting, the steel was hot rolled to different thicknesses followed by cold rolling to a uniform thickness. The adjusted degrees of cold rolling reduction led to different levels of work hardening. Finally, these samples were aged at defined temperatures for different periods of time.

The mechanical properties of these specimens were investigated by means of hardness and tensile tests. The current literature suggests that work hardening increases the strength after aging. Therefore, our analysis focuses on the changes of these mechanical properties during aging depending on the degree of cold rolling reduction.

Special attention is given to the characteristic values of peak hardness and set-up time as a function of the degree of cold rolling reduction and aging temperature.

Novel method for accelerated biopolymer aging

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Keywords: biopolymer, poly(lactic-acid), weathering, degradation, accelerated aging

Today, there is an increasing social and industrial need for certain polymer products to be produced from annual renewable sources and bio-based & bio-degradable polymers. Biopolymers, such as polylactic acid, are possible marketable candidate to replace polyethylene, polypropylene, polystyrene and other petroleum-based polymers, providing solution for engineering aspect (processing conditions, final product properties) and customer needs (biodegradability).

The degradation processes of polymers have an outstanding importance, and therefore their extensive research is ongoing. In case of biopolymers the analysis of biodegradation processes by enzymes and microbes has great importance [1].

However, it should not be disregarded that the primary consideration in material engineering remains the calculation of various other (thermal, oxidative, photo) degradation processes during the shelf life of the product. As a result of these processes, unfavourable mechanical and optical changes strongly affect product shelf life and usability depending on exposure.

Well known methods include accelerated natural weathering and the use of environmental chambers. Their typical acceleration factor (accelerated aging: natural aging) is 2:1 or 5:1, with extreme and expensive methods it can be increased to 12: 1 [2]. Thus, a material and production process development process under the necessity to count with a significant additional development time. The test procedure that we used, irradiation with excimer laser: acceleration factor - based on literal data - is up to 3200:1 or greater [3].

During the research a new type of accelerated photodegradation procedure was applied to characterize biopolymers. We produced poly(lactic-acid) films (extruded, neat, high D-lactid percentage, amorphous), after that a managed to irradiate the films by an excimer laser at a given wavelength, simulating 200-hour exposure. In parallel, we also tested the 200-hour exposure of poly(lactic-acid) film samples using a standard environmental chamber (xenon lamp, daylight filter, based on standard conditions: ISO 4892-2:2013). The resulting two types of photodegraded film products were compared and broadly characterized with the usage of differential scanning calorimetry (DSC), thermogravimetic analysis (TGA), attenuated total reflectance (ATR), scanning electron microscopy (SEM) and optical microscopy to find out possible conformity between the two methods.

Acknowledgments

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Electrical conductive thermoplastic compounds for electromagnetic shielding applications

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Keywords: Thermoplastic Compounds, EMI/RFI Shielding, Electrical Conductivity, Injection Molding, Anisotropy

Engineering thermoplastics with electrical conductive properties have emerged significantly into the market of housings for electronic devices (e.g. computers, sensors or multimedia systems). An electrically conductive polymer can be used for electrostatic dissipation (ESD) and/or barrier to electromagnetic and radiofrequency interference (EMI/RFI), essential characteristics for electronic products safety. These plastic composites allow weight reduction, better handling, improved corrosion resistance, more design freedom, short production cycle and the adaptability to product requirements, which makes them an attractive alternative to traditional solutions using metallic shields and hybrid products based on coating technologies or embedded thin metal foils.

However, designing products with thermoplastic compounds can be an engineering challenge due their anisotropic and processing induced characteristics. The effect of processing conditions on the final properties of these materials is very important to provide valuable data for design optimization. Therefore, this paper exhibits an empirical analysis made for carbon fiber reinforced thermoplastics compounds. The anisotropic electrical conductivity and EMI shielding of such compounds were evaluated in injection molded rectangular plates as a function of processing conditions and relative location of the samples.

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Film thickness effect on the grain growth kinetics of nanocrystalline Cu

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Keywords: copper films, nanocrystalline, grain growth kinetics

The need for ever-increasing energy efficiency and resource optimization has raised expectations concerning the use of the latest technological developments in several industries. Sputtering is an extensively studied deposition process, in which through the optimization of the deposition parameters it allows obtaining tailor made materials with the appropriate set of properties and characteristics, such as grain size and preferential crystallographic orientations. The deposition process influences the materials propensity to develop twins within its matrix in low stacking fault energy materials, such as copper, as well as the density of dislocations inside the grains, for all metallic materials. The deposition process also affects the resulting internal stresses of the created films, with PVD processes being able to produce films with internal compressive stresses on the GPa order of magnitude [1-4].

The thickness of the films affects several materials' properties, its resistivity being the most relevant for electronic applications, as it is correlated with energy efficiency. A material's total resistivity is the result of distinct components, namely surface scattering, grain boundary scattering, impurity scattering and morphological defects, the latter component pertaining to the geometry effect of the grains themselves on the electron movement through the material [5, 6].

The present work compares different RF sputtering deposition parameters and their effect on film thickness, with grain size estimated by the Debye-Scherrer equation over XRD diffractograms and further corroborated by resort to transmission electron microscopy bright field imaging. Grain size estimations and bright field imaging of planar view sections indicate proximity to its electron mean-free path ($\lambda = 39.9$ nm), which should be the limit for the change in resistivity behavior due to electron scattering at the grain boundaries. Specimens were evaluated in the as-deposited, annealed and self-annealed condition, to assess the material's thermodynamic and kinetic stability. The texture was analyzed through selected area diffraction pattern analysis in conjunction with centered dark-field imaging, to assess the viability of the method for future developments [7].

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Modelling the effect of irradiation on statistical properties of plastic deformation

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Keywords: vacancies, dislocations, avalanche, plastic deformation, modelling

In a Zr alloy upon irradiation the concentration of impurities increases significantly. The strong local stress field of these impurities impedes the motion of dislocations (defects responsible for plastic deformation), thereby strongly influencing the sample's plastic behavior.

It is known that plastic strain accumulates in sudden avalanche-like events as clearly demonstrated by micropillar compression experiments [1]. Based on the statistical analysis of these bursts it has been suggested that plastic deformation can be described as a critical phenomenon [1,2,3].

In the talk we will primarily address how critical behavior associated with plastic flow is altered by the impurities. To this end, we apply discrete dislocation dynamics (DDD) simulations. Firstly, we developed a numerical algorithm that employs an implicit scheme in order to successfully cope with the extremely high computational cost of discrete dislocation dynamics simulations and makes the inclusion of impurities possible. Then results of load-controlled simulations are summarized where a varied number of impurities are added to the system. The statistical analysis is performed in terms of yield stress distributions [4] and the identification of extended soft deformation modes in the system [5]. In accordance with an earlier study [6], we find that impurities damp the critical behavior and extended modes leading to the significant weakening of strain bursts. This implies that the stress-strain curves get smoother upon irradiation.

Our results thus deliver a better understanding of the background of the strain bursts present in metals and how irradiation alters that. Since the numerical predictions can be explicitly checked experimentally by micropillar experiments, the results are expected to boost research in this direction.

Acknowledgments

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Acoustic emission aided fracture toughness testing of DCB composite beams with mechanical couplings

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Keywords: coupled laminate, DCB, delamination, acoustic emission

The Fiber-Reinforced Laminate Composites (FRLC) are commonly used in many fields of engineering for load-carrying structures. Delamination is particularly dangerous damage form in laminates [1]. The strain energy release rate (SERR) for different fracture modes can be determined in accordance with the ASTM Standards [2]. The authors have undertaken an attempt to modify and apply the standard procedure form mode I fracture toughness assessment to multidirectional coupled laminates. The relevant test specimen is the double cantilever beam (DCB), which consist in bending of two sublaminates, separated by delamination. As shown by York [3], bending of mechanically coupled laminates can result in other forms of deformation, such as twisting [1, 4]. In order to determine the mode I interlaminar fracture toughness (G_{Ic}) variability of coupled composites with various layups, a number of Finite Element (FE) simulations were performed by the first author. Next, experimental verification was done. During the DCB test the applied load was recorded, among other data. In particular, to determine the maximum load at crack propagation onset, the Acoustic Emission (AE) signal was registered as additional data. It allowed higher accuracy of determining the crack initiation moment. The obtained values of G_{Ic} for different ply layups are presented in Table 1.

Table

Table 1. Values of mode I strain energy release rate (G_{Ic})

Specimens interface	G_{Ic} [N/mm]
0°/30°	0,327
0°/45°	0,807
0°/60°	0,957
30°/30°	0,746
45°/45°	0,527
60°/60°	1,039

Acknowledgments

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Advanced non-destructive methods for residual stress and anisotropy determination by centreless X ray diffractometers

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Centreless X-ray diffractometers are developed for the sample-cutting free determination of residual stresses. The residual stresses strongly affect the lifetime of any object and is in a strong correlation with the production method and use of crystalline matters. The non-destructive feature makes it possible to apply them in such cases when sample cutting of the component is not allowed due to the unique or value feature of objects. Our investigation focused on the utilization of additional information on the crystal lattice of the examined object provided by the centreless diffractometers. This information is based on the properties of the X-ray interference function such as full width at half maximum (FWHM), integrated intensity of the reflections, Bragg angle, intensity distribution as a function of tilting. Our purpose is to introduce not only the stress measuring method based on centreless X-ray diffractometer

but also other possibilities to get information about the state of crystalline materials like anisotropy. The centreless diffractometers have never been used to describe anisotropy previously. The innovation and usability of the method stands in that it can be applied directly on the objects without the need of specimen cutting. The technique has literally no size limitations and it does no harm to the examined objects. Measurement and calculation methods for non-destructive texture tests were developed to determine the identical crystalline plane series distribution in centreless and in the conventional diffractometers. The methods were validated for OMEGA and modified PSI modes. In the presentation, examples of these related topics are presented in cases where destructive test of the target object is not allowed.

Functionally graded components for nuclear applications

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The application of functionally graded components is considered for the next generation nuclear reactors, where two dissimilar metals are needed to operate together in the framework of a single component. Currently, dissimilar metal welds are used in such joints, which are inherently weak spots in terms of mechanical properties, due to residual strains and stresses. In the present work, the materials studied are the austenitic 316L stainless steel and the ferritic low-alloyed SA508 steel grade 2. The materials were supplied in powder form and functional grading was realised by mixing the two metals in various compositions to create a transition zone from 316L to SA508. The powders were then consolidated into a single component via the Hot Isostatic Pressing (HIP) process and the microstructure development was studied for different stages of HIP processing. X-ray diffraction analysis was carried out to investigate ferrite/austenite phase fraction evolution during processing as a result of extensive diffusion of alloying elements from 316L to SA508. The mechanical properties along such a transition zone were also studied, in order to be linked to the resulting microstructure. The aim of the project is to optimise the initial composition profile of the two metals along the joint in order to achieve improved properties across the transition zone. If functional grading is found to result in superior properties across a dissimilar metal joint, compared to welding, this could lead to a significant performance increase. In addition, functionally graded components could be extended to other applications.

Effect of chromium content of steel on grain boundary penetration of liquid copper

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Keywords: Brazing, C45 Medium Carbon Steels, Austenitic and Martensitic SS, Copper, Chromium, Grain Boundary Penetration.

Joining in strictest sense is to bring two different or similar components together to make them into one. Joining is known to mankind for centuries and is one of the primitive processes/principles by which man could figure out how to build things with complex geometries. Brazing is one such joining process where a braze alloy is used to melt to form a bond between two substrates that are to be joined. Brazing traditionally was applied only between similar materials but advanced technologies have ensured its progression to dissimilar materials as well. Though many studies have been reported in regard to Steel brazing with Copper, most of them are focused on stainless steels or high temperature steels. Not much information is available on low-medium carbon steels and their brazing behavior with copper. This study focuses on braze alloy Penetration observed in C45 medium carbon steel besides observing penetration of copper in different grades of steels like Austenitic 1.4301, Martensitic 1.4034, Temper hardened 42Cr4Mo and Annealed 42Cr4Mo of varying elemental composition. Experiment was carried out with a thin copper foil of thickness 10-30µm placed between two steel samples to form a sandwich. The SEM results showed good penetration of copper into the steel grain boundaries at the interface. The penetration depth attained varied depending on the type of steel and its composition particularly in relation with chromium. In some cases the high chromium has hindered the penetration. We have found that materials like Cr have an effect on penetration of copper in steels. Thus, copper behavior can be seen as function of elemental composition.

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Modelling of the yarn pull-out process for the characterization of reinforcing woven fabrics

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Keywords: yarn pull-out, modelling, woven fabric

Woven fabric is one of the most commonly used fibrous structures for reinforcing polymer composite products. Depending on the shape of the manufactured product, the reinforcing fabric may have to assume twice-curved surfaces. Such great deformations significantly influence the endurance and properties of composite structures. The deformability of the reinforcing fabric mostly depends on the friction among yarns, so these are very important to examine and analyse. One possible measurement method for these is the yarn pull-out test. Knowing friction, we can describe the behaviour of the fabric with models as close to reality as possible, and provide the basis for design, calculation and simulation.

During the yarn pull-out test, the woven fabric has to be clamped on both sides, then parallel to the clamps we pull out one roving from the centre. During the pull-out process, the pull-out force and the displacement of the roving are measured. The yarn pull-out test can mainly be used to characterize the interaction among yarns.

The aim of the project was to test Vas's yarn pull-out model [1]. During the project eight glass woven fabrics from the same manufacturer were examined. Three pairs of glass fabrics had the same area density, but different weave patterns in each pair. The other two fabrics had approximately the same area density, but a special structure.

The presentation shows the examined materials, the measurement method applied and the results, Vas's yarn pull-out model and the results of the evaluations of the measurements.

On the one hand, we compared the values Vas's model provided with the actual measurement results, that is, we examined how precise predictions the model gives when certain attributes are known. On the other hand, based on the test results, we found connections between the length of the yarn in the woven fabric and the tensile force acting on the yarn.

All in all, we can conclude that the model describes the process of yarn pull-out well, so it can be applied for more complex woven fabric models and simulations.

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Consideration of the impact of residual stress on the short crack growth behavior in martensitic steels

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Keywords: microstructure-based fracture mechanics, martensitic steel, residual stress, high cycle fatigue

Martensitic steels with an ultimate tensile strength beyond 2000 MPa exhibit an excellent fatigue strength in the high cycle fatigue regime. The fatigue resistance can be even improved by compressive residual stresses introduced by shot peening. However, the influence of residual stresses on the mechanisms of short crack propagation in martensitic steels has not yet been sufficiently investigated. For this reason, further research work is required concerning the fracture-mechanical proof of fatigue strength of martensitic steels under the influence of residual stresses.

A new research project targets to reveal the micro-structure based short crack propagation in shot peened martensitic steels. For this purpose axial and torsional fatigue tests in the high cycle fatigue regime will be carried out and the fracture surface will be characterized by means of electron scanning microscopy. Subsequently, laser scanning microscopy and electron backscatter diffraction will be used to monitor the crack growth and to link the local crystallographic orientation of the crack initiation site with microstructural features. The residual stresses will be measured using x-ray diffraction analysis. Moreover, on the basis of an interferometric strain displacement gauge crack closure due to compressive residual stresses and its effect on the short crack growth will be investigated. To establish a better understanding of the impact of residual stress on short crack growth, a crack propagation model, which is based on a two-dimensional dual boundary element method, will be utilized to simulate short crack propagation in martensitic steels.

New aspects of relationship between fusion and solution enthalpies of aromatic compounds

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Keywords: fusion enthalpy, heat capacity, solution calorimetry, fast scanning calorimetry

Extensive data on the fusion enthalpies of compounds from various classes are available today. Nevertheless, the fusion enthalpy prediction remains a complicated task. To have an opportunity for the analysis of the fusion enthalpies of the different compounds melting at the different temperatures, one should have the way to adjust them to the same conditions. Solution calorimetry can be used as a source of information about the fusion enthalpy value

at 298.15 K. In the present work the relationship between the fusion enthalpies of aromatic compounds at the melting temperature and the solution enthalpies in benzene at 298.15 K is studied according to the following equation:

$$\Delta_{\text{soln}} H^{A_i/C_6H_6}(\text{cr}, 298.15 \text{ K}) - \Delta_{\text{soln}} H^{A_i/C_6H_6}(\text{l}, 298.15 \text{ K}) = \\ = \Delta_{\text{cr}}^{\text{l}} H^{A_i}(T_m) + \sum \Delta_{\text{tms}} H^{A_i}(T_{\text{tms}}) + \int_{T_m}^{298.15} [C_p^{A_i}(\text{l}, T) - C_p^{A_i}(\text{cr}, T)] dT$$

Where $\Delta_{\text{soln}} H^{A_i/C_6H_6}$ is the enthalpy of solution of compound A_i in benzene, $\Delta_{\text{cr}}^{\text{l}} H^{A_i}(T_m)$ is the enthalpy of fusion at the melting temperature, $\sum \Delta_{\text{tms}} H^{A_i}(T_{\text{tms}})$ is a sum of the enthalpies of solid-solid phase transitions occurring between 298.15 K and the melting temperature and

$$\int_{T_m}^{298.15} [C_p^{A_i}(\text{l}, T) - C_p^{A_i}(\text{cr}, T)] dT$$

is a thermal adjustment of the fusion enthalpy to 298.15 K calculated according to Kirchhoff's law.

For the most of aromatic compounds not capable of self-association due to intermolecular hydrogen bonding the $\Delta_{\text{soln}} H^{A_i/C_6H_6}(\text{cr}, 298.15 \text{ K})$ values are close to $\Delta_{\text{cr}}^{\text{l}} H^{A_i}(T_m)$, indicating weak temperature dependence of the fusion enthalpy, considering that the values of $\Delta_{\text{soln}} H^{A_i/C_6H_6}(\text{l}, 298.15 \text{ K})$ for such compounds are close to zero.

The analysis of Eq. (1) for the non-self-associated aromatic compounds having significant distinctions between $\Delta_{\text{soln}} H^{A_i/C_6H_6}$ and $\Delta_{\text{cr}}^{\text{l}} H^{A_i}(T_m)$ leads to a conclusion that the heat capacities of these compounds in hypothetical liquid state between 298.15 K and T_m appear to be linear extensions of the heat capacities of the melt. Preliminary studies of supercooled liquids heat capacities by fast scanning calorimetry support the latter statement.

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Poster

Comparative study between solar thermal and photovoltaic: modeling and characterization

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Keywords: solar thermal collector, photovoltaic cell, Matlab modeling

In our work, we used the Matlab software in the modeling of different parts of a photovoltaic and thermal solar collector. Our main goal is to make a comparative study between the two technologies by taking into account the cost, and by modeling the efficiency, the spectral response and the variability of the power delivered according to the illumination.

The influence of the phosphorous content on the nano-microstructure and microhardness of electroless Ni-P coatings

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Keywords: Ni-P coating, Nano-structure, Micro-hardness, Heat treatment, Inverse Hall-Petch rule

Electroless Ni-P coatings were deposited on W302 steel substrates using different bath compositions, which lead to different phosphorous contents of the coatings [1]. In our research, the effect of the P-content in the Ni-P coatings was experimentally studied on the different properties of the coatings: its nano-micro-structure, thickness and micro-hardness. The as-deposited samples were nano-crystalline (mostly amorphous according to XRD) and their micro-hardness was found to decrease with increasing the P-content. Upon heat treatment at 400°C a new Ni₃P intermetallic phase was formed while the nano-crystalline Ni-rich grains coarsened to micro-grains. The micro-hardness of the heat treated samples was found to increase with increasing the P-content.

In order to explain our experimental results, a complex model was built [2], supposing that the as-received Ni-P coating contains almost pure Ni nano-grains surrounded by segregated P atoms; as the grain grows, its surface is covered more and more by the P atoms. When the grain is fully covered by the P atoms, further grain growth is inhibited, the coating can grow

further only due to nucleation of a new grain. Thus, the size of the grains was found inversely proportional to the P-content of the Ni-P alloy. The need for a larger number of nucleation events with decreasing grain size explains why the coating has a smaller thickness for smaller grain size, i.e. higher P-content. The inverse Hall-Petch rule was found for the grain size dependence of micro-hardness of the as deposited coatings. Due to the grain boundary sliding of relatively hard Ni-rich nano-crystals along the soft P atoms, higher P-content lead to lower micro-hardness through smaller grain size. After heat treatment, the micro-hardness was found to increase with the P-content of the Ni-P coating i.e. with the volumetric phase fraction of the harder Ni_3P intermetallic phase within a relatively soft Ni matrix. The extrapolated value for the micro-hardness of the Ni_3P phase is found about $757 \pm 20 \text{ HV}_{0.01}$.

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Physico-chemical and mineralogical characterisation of marls deposits (Algiers)

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Keywords: marls, mineralogy, smectite, X-ray

Marls and marly clay deposits covers an extensive area to the south west of Algiers (Algeria), where the city and its suburbs are located. These deposits forms the bedrock on which the urban development take place and serve as foundation for most building and roads. Different alteration zones in marls deposits were determined depending on differences related to texture and colour. In order to determine mineralogical and physico-chemical characteristics of marls and their implications in stabilities problem, X-ray diffractometrie (XRD), scanning electron microscope (SEM), acid soluble carbonate, index swelling and identifications tests were performed on three samples collected at different depth. chemical analysis show a great proportion of silice compared to alumina values. this results are in agreement with XRD analysis, which revealed that the materials are composed of smectite and illite with minor

amounts of calcite and quartz. Based on the plasticity characteristics, marls are moderately plastic, while the altered materials are classified as moderate to high plasticity. Accordingly, it is evident that the altered materials reflect moderately to high swell potential due to the presence of expansive clays (smectite).

Characterisation and thermodynamic modelling of inconel-718 vacuum brazed joints with nickel-based filler metals

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Keywords: Brazing, Characterisation, Thermodynamic Modelling, Superalloys, Nickel Alloys

Brazing is a process for joining two or more metal or ceramic items using a filler material with a lower melting point than that of the items to be joined. Brazing commonly sees application in the aerospace industry as manufacture and repair of nickel superalloy parts such as jet engine components. The demands of such applications means that nickel-based brazing alloys are favoured, due to their high melting temperature, mechanical properties and corrosion resistance. Melting point depressants (MPDs) such as boron are often added to decrease the liquidus temperature to a suitable level, while also potentially resulting in the formation of brittle phases in the joint region. This study aims to characterise and identify phases formed in Inconel-718 joints vacuum brazed using nickel-based filler metals AWS BNi-2 and AWS BNi-9. Composition and microstructure of the brazed joints were investigated by optical microscopy, scanning electron microscopy (SEM) and electron probe micro-analysis (EPMA). Mechanical properties of the joint were examined by nano- and micro-hardness testing, and tensile testing in accordance with American Welding Society standards. Thermodynamic modelling of the systems was carried out using Thermo-Calc software. Phases predicted to form by the software are compared against experimental results.

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Detection of delamination in composite structures with DIC method.

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Keywords: composite, digital image correlation, non-destructive method

Nowadays, fiber-reinforced polymer composite structures are frequently used, due to their excellent specific mechanical properties. With their use, we are able to build lighter structures and more efficient machines. This is the trend in both the aerospace and the automotive industry. These industries are critical in personal safety therefore it is important to periodically estimate, monitor the condition of the high value, load bearing structures. The topic has been investigated by several research groups but the perfect, non-destructive method that effectively detects structural failure has not been found yet. The acoustic emission (AE) method can only compare the recorded data to previously acquired references, and it needs the cracks to propagate during testing. Thermography needs large energy input and a longer measurement time to show the internal damage in composite laminates. Computed tomography needs very expensive equipment and the sample sizes are very limited, often the investigated parts have to be removed from the vehicle. The evaluation of recorded ultrasound images is very time-consuming and the measurement is very sensitive to machine setup.

The problem can be probably solved by the application Digital Image Correlation (DIC) combined with small load, non-destructive mechanical testing, an old method that improved a lot with the developing technological industry. Its main advantages are that it is not a contact method and that it can be used on large surfaces. Furthermore, the method is simple and can be done quickly.

In our work, full field strain measurement with DIC technique is used. Our aim is to find the connection between the surface deformation field and the damage inside the structure using small loads. In this article, delamination, as one of the major damage modes of composites, is examined. The structural defects are artificially created in various ways and they are tested with DIC.

Acknowledgments

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Comparative analysis of real and virtual garments distance ease

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Keywords: 3D body scanning, virtual try-on, distance ease, simulation

In the past decade, virtual try-on gained considerable attention, due to rapid development of newest technologies in the fashion industry. Virtual try-on technologies provide significant benefits to the apparel manufacturers [1]. It helps to modify the model style and patterns through quick simulation of the fit. It provides accuracy, flexibility and suggests well fitted garment for individual body type.

Researchers try to combine 3D scanning systems and garment simulation technologies together in order to design the best fit garment for the individual figure. For effective practical use of virtual try-on technology, it needs to be investigated how practically garment fit is simulated by the particular technology and whether there is a gap between the real and virtual garment [2]. However the most of the scientists focuses into comparing the garment fit of real and virtual body model with parametric mannequin of the used 3D CAD system. There is another approach for evaluating garment fit to use the actual scanned 3D body model, which is not researched yet [3]. It is very important to prove virtual try-on matching with the real garments therefore the goal of this research was to compare distance ease between body and the garment using virtual try-on and scanning technologies.

In this study, 3D scanner VITUS Smart XXL was used. Women dummy was scanned with the real straight fit dress of the same size made from five different woven fabrics. Size of the dummy was changed uniformly from 88 to 95 cm (step 1 cm). Distribution of distance ease in tree main cross-sections (bust, waist and hip) between body and a garment was investigated. Later scanned dummies were imported into Modaris 3D software (CAD Lectra) and virtual try-on of the same dress was done to compare with the scanned results (Fig. 1).

Based on the results it can be concluded that generally appearance of the three main cross-sections of the simulated dresses was satisfactory in terms of the real scanned images, but there were some differences in garment shape fluency also in distance ease values between body and garment.

Figures

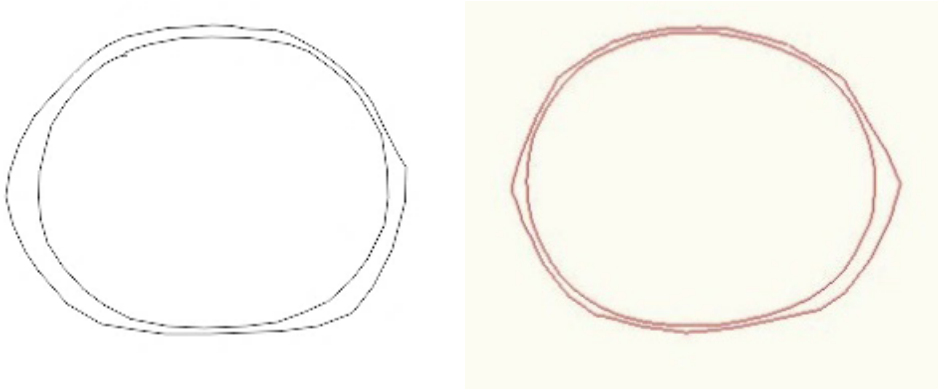


Figure 1. Cross-section of real (left) and virtual (right) dress at waist girth using fabric 02.

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Material characterisation of selectively laser melted haynes 282

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Keywords: Additive Manufacturing, Superalloy, Characterisation, Dendritic Growth, Laser Deposition

Additive Manufacturing is a fast growing manufacturing technique being used for a wider variety of applications and using more materials and alloys. Selective Laser Melting (SLM) is one of the core methods by which additively manufactured components are made. In this method, a single layer of powder is spread across a platform. A laser is then used to melt the powder in the cross section required. The platform is then lowered by one layer and the process is repeated. In this way, the part is built up [1].

SLM, in particular, has many benefits over traditional manufacturing methods that make it potentially applicable to the aerospace sector in particular. Material wastage is a major problem with high-precision parts for aeroplanes. In some cases, most of the material has to be discarded in order to produce the final part[2]. With SLM especially, this wastage can be vastly reduced as much of the unused powder can be reused. This dramatically reduces material usage as well as energy costs to produce[3]. In addition to this, the increased geometrical freedom allows for multiple components to be combined into a single part reducing weight[4]. This is a critical factor in aerospace design decisions.

Nickel superalloys are used in the combustion chamber of aircraft engines, their ability to retain their high strength at high temperatures make them suited to this application. Haynes 282 is a relatively new superalloy renowned for its weldability amongst nickel superalloys[5]. This makes it an ideal candidate for usage in SLM applications.

This study shows the results of the characterisation of, initially the powder then fully sintered test parts. Characterisation of the powder was done using both a particle size distribution and using SEM imaging. Once the test parts had been created they were sectioned and analysed for density as well as porosity. Micro-cracking was investigated using SEM images of the etched surface.

Although this study focused on the material characterisation it must be noted that with AM, the machine can have a large effect on the resulting parts. As a consequence, it was necessary to characterise the machine as well. This was done on a number of fronts that included examining the positional accuracy of the laser as well as the temporal power behaviour of the laser.

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Grain growth controlled by grain boundary and boundary junction migration

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Keywords: Grain growth, Triple junction mobility, grain boundary energy, high temperature laser scanning confocal microscopy.

The development of modern polycrystalline materials, including steels, ceramics and super-alloys depends on a fundamental understanding of the microstructural changes that occur during processing and operation. Since grain size plays an important role in terms of mechanical properties of polycrystalline materials, grain growth is one of the crucial phenomena e.g. occurring during heat treatment and welding processes. Theoretical models of grain growth are introduced that take into account boundary junction mobility. Each grain boundary can be characterized by its individual specific energy and mobility. The microstructure is approximated by a 2D setting consisting of a boundary junction and 3 adherent grain boundaries. It is discussed how boundary junction mobility influences grain boundary kinetics and therefore grain growth phenomena. Simulations of the kinetics of grain growth are performed by investigating the motion of individual grain boundaries meeting at triple junction lines of finite and infinite mobility. By means of these models grain growth in line-pipe steels during heat treatment is investigated. The kinetics of grain boundary regions near triple-junctions is analyzed numerically and compared to grain boundary motion in line-pipe steels observed by high temperature laser scanning confocal microscopy.

European Master Thesis Award

A digital microfluidics platform for loop-mediated isothermal amplification of DNA

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Keywords: Digital Microfluidics, Loop-mediated Isothermal Amplification, *c-Myc*, lab-on-chip, point-of-care diagnostics.

Molecular diagnostics is one of the fastest-evolving and most exciting fields in biotechnology, aiming to redefine the diagnostics paradigm from central laboratories to point-of-care (POC). Thus, the intervention of Materials Science is required, and Digital Microfluidics (DMF) promises to take the lead of this paradigm shift, presenting unmatched control of low-volume droplets (down to pico-liters), and excellent integration capabilities with external devices for any sort of droplet analysis. Furthermore, robustness and compatibility with standard microfabrication techniques also favour DMF as a viable option for commercial POC devices. Such advantages allow for fully automated low-volume reactions, namely isothermal nucleic acid amplification¹. With this work, we demonstrate for the first time the coupling of DMF and loop-mediated isothermal amplification (LAMP), one of the most specific and robust amplification technologies. The DMF devices are fabricated on glass substrates using low-temperature microfabrication techniques and include drilled ports for sample/reagent inlet/outlet, by means of a pipette, assuring a simpler, easier way to insert and withdraw samples (Figure 1). LAMP reactions for the detection of cancer biomarker *c-Myc* (overexpressed in about 20% of all human cancers), are performed on-chip with optimum temperature control, achieving merely 0.3°C of droplet temperature variation from the set-point (65 °C). With this setup, we achieved amplification of 0.5 ng/μL of target DNA in 45 minutes and 1.5 μL, a considerably lower volume than bench-top LAMP².

Figures

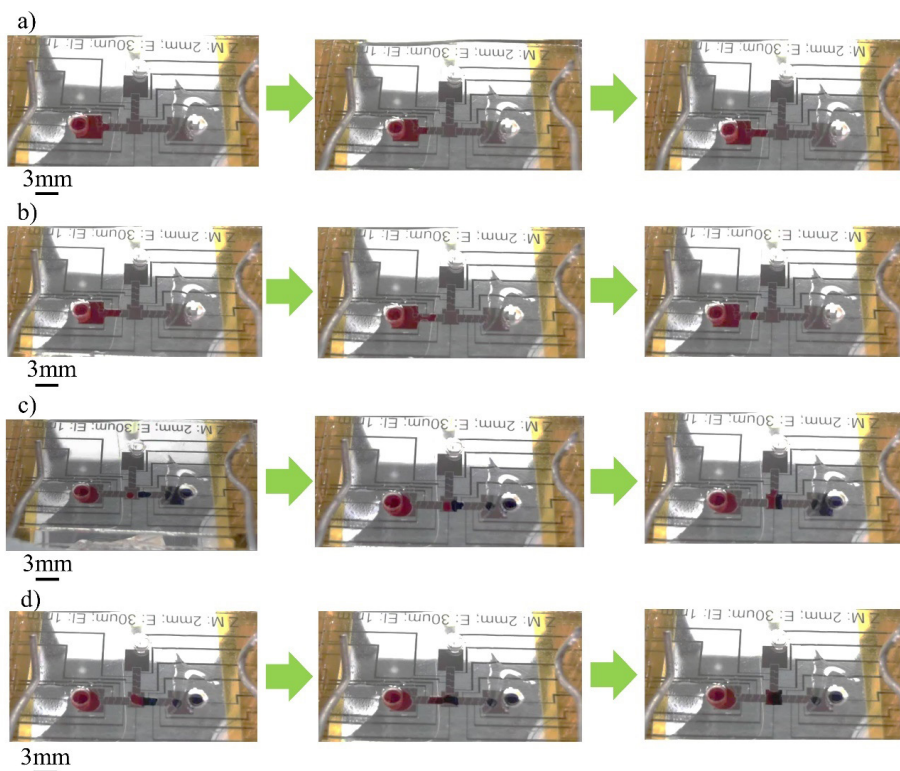


Figure 1: Video frames showing all the possible fluidic operations: dispensing (a), splitting (b), merging (c) and mixing (d).

Acknowledgments

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Experimental Determination of the Adhesion of Hard CAPVD Coatings

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Keywords: Hard film coatings, DIN 1.2999 hot work tool steel, scratch test, adhesion

Hard film coatings are extensively used in various applications, such as machining tools, die components, turbine blades etc. Along with other improvements, these coatings provide significant enhancement in the mechanical reliability of the components as well as in their wear resistance. A vital role to the substrate/coating system performance plays the adhesion of the coating which characterizes its capability to remain intact over all the substrate surface when the substrate/coating system is subjected to tensile or shear stresses during service.

The current work is aimed to determine the nitriding effect on the adhesion of single, duplex and multilayer coated DIN 1.2999 grade steel which has not been studied before as substrate material. For that purpose, various combinations of single (CrN), duplex (AlTiN) and multilayer (CrN/AlTiN) layer coatings were deposited on the surface of the DIN 1.2999 steel, either directly or after surface nitriding, by CAPVD process. Hence, nitriding prior to coating provided higher surface hardness values in the sequence of single, duplex and multilayer coatings. The experimental stage included the determination of the adhesion characteristics of coatings using the scratch test method and the failure modes were characterized by stereo – optical microscope, atomic force microscope (AFM) and scanning electron microscope (SEM/EDX).

According to the critical load (L_C) where the coating failure occurred, (i) nitrided samples had significantly increased values due to the existence of the diffusion layer and (ii) among all samples studied, the multilayer coatings having the highest L_C values, exhibited the better adhesion. SEM/EDX as well as AFM studies on the scratched surfaces showed that both adhesive and cohesive failure modes were present, while the coating failure pattern evolved during the scratch tests was also determined for each coating, providing useful data for the following tribological studies.

Synthesis and characterization of complex semiconducting oxide nanostructures

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Keywords: type your, keywords here, separated, by commas

β -Ga₂O₃ is a transparent conductive oxide (TCO) semiconductor with a wide band gap of ~4.9 eV and an exceptionally high breakdown voltage. These characteristics make it a very promising material for applications both in optics and in high power electronics. Doping with elements like Cr or Sn does not only improve its optical and electronic properties but also controls the dimensions and morphology of the obtained nanostructures [1]. SnO₂ is another TCO with applications developed in several areas, for example in lithium batteries [2]. In this work, complex nanostructures based on Ga₂O₃ and SnO₂ have been synthesized via a catalyst-free vapor-solid mechanism. Their morphology and physical properties have been studied by different analytical techniques with high spatial resolution based on electronic microscopy, confocal microscopy and photoelectron spectroscopy. Part of this master thesis was included in the following recent publications [3,4].

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PLMA-b-POEGMA amphiphilic block copolymers: Synthesis, characterization and properties in aqueous solutions

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Keywords: RAFT polymerization, amphiphilic block copolymers, micelles, indomethacin, magnetic nanoparticles

Amphiphilic block copolymers are intensively studied due to their ability to form self-assembled nanostructures in aqueous solutions of different morphologies, which can be utilized in nanomedicine, biotechnology and in bioimaging and theranostic protocols. RAFT

polymerization based synthetic strategies proved to be highly efficient in producing a wide variety of well-defined amphiphilic block copolymers with diverse chemical functionalities and physicochemical properties.

In this work, RAFT polymerization was utilized for the synthesis of amphiphilic block copolymers of the type poly(lauryl methacrylate-*b*-(oligoethylene glycol) methyl ether methacrylate (PLMA-*b*-POEGMA), with various hydrophilic/hydrophobic block ratios. These block copolymers self-assemble into compound micelles with large aggregation numbers and soft cores, when dissolved in aqueous media, as determined by light scattering and transmission electron microscopy. Critical micelle concentrations determined by fluorescence spectroscopy were found to be very low.

Encapsulation of indomethacin (IND) within the copolymer aggregates was successful. The maximum IND encapsulated was 30% w/w, relative to the PLMA block mass. The presence of IND altered the sizes of the mixed drug-copolymer nanoparticles, in a way that depended on the hydrophilic/hydrophobic ratio of the block copolymer. DLS measurements suggest that the size distributions of loaded aggregates become narrower after IND addition. In the case of magnetic NPs encapsulation the compound polymeric micelles can load up to 10% iron oxide nanoparticles in their PLMA cores, forming hybrid nanostructures. The size distribution of the mixed micelles is significantly broader after the loading of the magnetic NPs, as shown by DLS. The NPs maintain their magnetic properties after their encapsulation in the micelles, as it was proved by magnetophoresis experiments. The mixed solutions are colloiddally stable for copolymers where the hydrophobic ratio exceeds 30wt %. Experiments for the simultaneous encapsulation of both IND and magnetic NPs were also performed. In this case, only the copolymer with the lowest hydrophobic ratio was capable to ensure the colloiddal stability of the mixed three-component aggregates, resulting in magnetically active mixed nanostructures with encapsulated IND. The mixed aggregates seem to be affected by the presence of increased concentrations of salt and BSA but without loss of their nanosized dimensions and colloiddal stability.

Because of the colloiddal stability, drug encapsulation ability and magnetic properties of the nanosystems based on biocompatible PLMA-*b*-POEGMA amphiphilic block copolymers developed in this study, these hybrid nanostructures hold promising potential for utilization as drug delivery and triggered release nanosystems, as well as in bioimaging applications.

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2D grain growth modeling in ODS steel with different full field approaches

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Keywords: ODS, Grain growth, Post-dynamic recrystallization, Monte-Carlo method, Level Set method, Smith-Zener pinning.

Introduction: ODS (Oxide Dispersion Strengthening) steels are ferritic matrix steels hardened by many oxide nanoparticles $Y_2Ti_2O_7$ [1]. They are mostly used for applications in the nuclear industry. Each annealing step of the forming process must be perfectly controlled in order to obtain optimal microstructure and mechanical properties. Nanoparticles will slow down recrystallization and grain growth, by pinning grain boundaries (known at the mesoscale as Smith-Zener pinning [2]). Ideally, numerical simulations could be used to check mechanisms and optimize heat treatments without too many experiments. In this study, results from the traditional Monte-Carlo (MC) approach, used to model ODS recrystallization, are compared to those from the determinist front capturing Level-set (LS) method.

Numerical methods: MC methods are based on probability theory: the simulation domain is divided into 2D pixels or voxels in 3D, and an energy is assigned to each pixel. At each Monte Carlo Step (MCS) pixels are randomly picked and a reorientation probability, minimizing the system's energy and considering grain boundary mobility is calculated [3]. The dataset that describes the microstructure can be provided by an Electron BackScattered Diffraction (EBSD) analysis, which enables to consider crystallographic orientations.

In ODS steels, oxide particles are too small to fill one pixel. Another method using a Smith-Zener pressure was developed to incorporate the effect of these small oxides. This pinning energy is added to the global energy on each site for probability calculations [4].

Since the MC model is based on relatively simple probability calculation on regular grid, it is easy to implement and to parallelize. This leads to a reduced calculation time.

As drawback, the absence of direct link with physical time (simulations running in MC Steps) and the use of purely numerical parameters, constrains to calibrate the model with experimental results.

In contrast, LS method is based on solving partial differential equations (PDE) to describe grains boundaries migration, on a finite element mesh (linear P1 interpolation with unstructured triangular meshes in the considered framework) [5]–[7]. LS functions are used to describe the grains boundaries network by giving at any point the distance to the interfaces.

In this study, polycrystal built from an experimental EBSD map are used. Interface energy and grain boundary mobility are considered as isotropic. In this model, particles are represented by holes in the finite elements mesh. Their interactions with grains boundaries are

directly considered through their effect on the grain boundary mean curvature. In this work, the particles are assumed to be static in time, circular and incoherent with the matrix.

Results: First, simulations for grain growth without particles and without stored energy were performed with the MC and LS approaches. A linear relationship giving 171 MCS for 1 s was set by comparing the mean equivalent grain diameter at different time steps. Then, microstructures could be compared with grain size distributions. Distributions are very similar from a model to another independently of the considered time step.

In a second time, simulations for grain growth with different nanoparticles populations were done (see table 1). As particles are too small to appear on EBSD maps, they were randomly distributed on microstructure for LS simulations and a normal distribution was defined for their size. As a consequence of introducing these particles, grain growth is slow down and stops at an early stage of the heat treatment (figure 1). Results from LS simulations are not fully matching the Smith-Zener model predictions for nanometric particles sizes, despite that it was used to link simulations together and compare them to experimental results. For micrometric particles size, LS simulations show very good agreement with experimental values, but for particles sizes under 5 nm, experimental values are no longer on the fit from LS simulations. These observations suggest, as expected, that Smith-Zener pressure model is not suitable for such second phase particles where grain boundaries can no longer be considered as sharp interfaces comparatively to the particle sizes. It implies also that Smith-Zener pressure formulation used in the MC model is a strong hypothesis, which should be discussed.

The relation between MCS and time stay close to one established without particles, even if adding particles apparently slow down MC kinetics. A shift in the mean grain size is observed between MC and LS results at pinned state, which could be explained by an underestimation of the capillarity force in the MC model. Despite this, grain size distributions stay close one from each other.

Figure

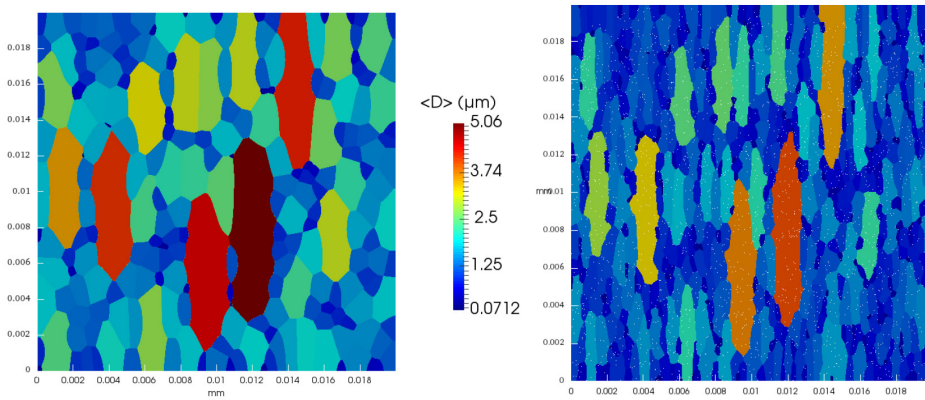


Figure 1: Comparison from level-set simulation at $t = 0.2$ s : left : no particles, right : with 10 nm particles and 0.5 % surface fraction (white points)

Table 1: particles populations studied with the Level-Set method

N°	1	2	3	4	5	6
Particles size (nm)	5	5	10	10	950	600
Particles surface fraction (%)	1	0.5	1	0.5	3.4	2.5

Acknowledgments

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FEMS Communication Award for Excellence in MSE for 2019

Toward the modeling of the surface treatment of composite components for satellites: the case study of the metallization of waveguides

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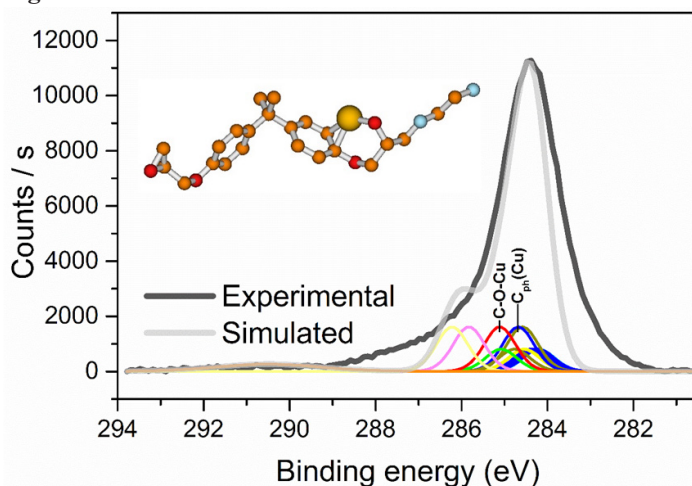
Keywords: Coatings, Epoxy surface, XPS, DFT

In the field of aeronautics and space, many metallic parts are subject to a substitution with composite materials with an objective of weight gain and durability. Carbon or glass fibers reinforced polymer composites are good candidates because of their excellent stiffness, low weight, and low chemical reactivity. Sometimes composite parts must be coated to recover the original properties of the metal or to provide additional properties such as optical absorbance, magnetic screening, or aesthetical aspect. However, the formation of a coating on materials that exhibit very low surface energy is challenging when one targets durability (adhesion, resistance to fatigue), and a tailored microstructure.

Whereas many processes have already been developed for the coating of composites in different fields such as microelectronics, we will briefly present new challenges posed by aerospace applications. How to adapt surface pretreatments? Why CVD is a processing method of choice? What properties can be expected?

Unfortunately, an engineering approach based on a rational screening of process parameters is not sufficient to gather a general knowledge about the treatment of composites surfaces. Therefore, in an attempt to develop an integrated model for the whole process, we present an original methodology that aims to link surface science on a model sample with the real world. We will focus on the study of the adsorption of Cu atoms on the surface of a poly-epoxy, and show how 'model' results can help solving technical issues.

Figures



Experimental (Black) and Simulated (Grey) XPS C1s spectra at a Cu thin film/epoxy interface.

Cellular architected materials obtained via additive manufacturing

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Keywords: architected materials, additive manufacturing, auxetics

Many industrial applications require materials with enhanced specific properties, i.e. performance per unit of mass, especially the transportation and biomedical sectors. Architected Materials are an emerging class of advanced materials that bring new possibilities in terms of functional properties, filling gaps in Ashby's material performance maps [1–3]. The term architected materials describes any heterogeneous material that exhibits improved specific properties due to a thoughtful and predetermined morphology and/or topology design. This usually induces characteristic length-scales comparable to the size of the final component being produced, i.e. the millimetre scale. Localised processing methods, such as additive manufacturing (AM), appear as natural candidates for developing such materials.

AM allows for the design and production of structures with geometrical features that would be unattainable with conventional manufacturing techniques such as forging or tooling. A striking example of innovative design enabled by AM are lattices or micro-truss structures, i.e. architected cellular materials, which have been extensively studied for their stiffness-to-weight performance. A special subset of lattices are auxetics, or negative Poisson's ratio materials, exhibiting non-classical behaviour which is of interest for structural and

damping applications [4–6]. Besides empirical design strategies, the advent of computational topology optimisation techniques enables new geometrical concepts for auxetics [7].

Nevertheless, from a materials science viewpoint these structures are more than an assembly of struts and can be considered as a material continuum with homogenised behaviour accounting for the underlying microstructure. On the other hand, the response of architected materials can be very sensitive to microstructural defects and geometrical imperfections. Different strategies have been studied in the literature for mitigating the surface defects of additively manufactured metallic lattices: chemical etching, electro-erosion, mechanical polishing. A new proposition is presented in this work: polymer coating or embedding of metal struts, by analogy with the soft-hard turtle-like strategy for mitigating crack propagation. Besides processing of such architected lattice structures, the present work brings experimental and numerical results concerning their mechanical behaviour. As a matter of fact, one engineering challenge is to predict the effective mechanical properties of architected materials; computational homogenisation using finite element analysis is a powerful tool to do so when considering quasi-static behaviour; difficulties arise when analysing the effective damping behaviour.

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