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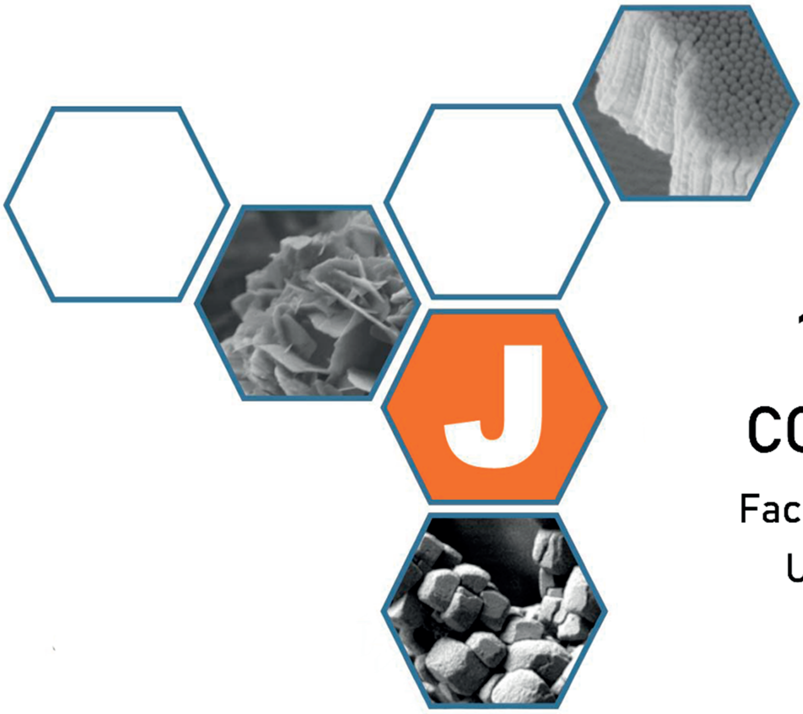
FEMS
FEDERATION OF EUROPEAN MATERIALS SOCIETIES

BOOK OF ABSTRACTS



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Plenary Talks

Plenary I - Flexible oxide electronics: where functionality meets sustainability

Pedro Barquinha, Associate Professor at NOVA School of Science and Technology, Portugal.

The recent demonstration of a flexible 32-bit microprocessor by PragmatIC and Arm using indium-gallium-zinc oxide (IGZO) thin-film transistor (TFT) technology clearly shows the interest of taking flexible electronics to applications beyond displays. Indeed, an anticipated vision would be to seamlessly integrate electronic functionalities into everyday objects, which would require researchers to conceive a high-performance multifunctional technological platform using sustainable processes and materials, tackling simultaneously More Moore and More than Moore concepts.

This presentation will focus precisely on the work being carried out at NOVA toward this direction. Topics to be covered include: replacement of IGZO by zinc-tin oxide (ZTO), a critical-element free semiconductor, without compromising low-temperature fabrication and TFT performance; oxide TFT integration in multiple analog and digital flexible circuits; use of oxide TFTs as ionizing radiation sensors; synthesis of ZTO nanostructures by hydrothermal synthesis and its combination with PDMS to create mechanical nanogenerators; integration of fibre-TFTs with fibre-QLEDs into a smart textile display system.

Plenary II - From nanoparticles to artificial tumors: A lifetime scientific journey

Luis M. Liz-Marzán, Ikerbasque Professor at CIC biomaGUNE, Spain.

Nanoplasmonics can be defined as the science studying the manipulation of light using materials of size much smaller than the radiation wavelength. This technology finds applications in various fields including sensing and diagnostics. An essential component of nanoplasmonics are nanostructured noble metals, which absorb and scatter light efficiently, through coherent oscillations of conduction electrons. Although the remarkable optical response of "finely divided" metals is well known since more than 150 years ago, the recent development of sophisticated characterization and modelling methods has dramatically reactivated the field.

This talk will provide an overview of the gradual shift in research approach, from colloidal synthesis methods, through self-assembly, optimization of plasmonic properties and ultimately performance in biosensing applications. Several examples will be presented in which nanostructured materials comprising gold nanoparticles have been used as substrates for ultrasensitive detection of biorelevant molecules.

Plenary III - Advancing Solid Interfaces and Lubricants by First Principles Materials Design

Maria Clelia Righi, Full Professor at Bologna University, Italy.

Friction and wear result in massive economic and environmental costs. By advancing tribological materials impressive energy savings, and consequent reduction of CO2 emissions, can be obtained. However, optimizing lubricant materials is challenging because their performances are ruled by molecular-level processes that occur at the buried interface, which are extremely difficult to monitor by experiments. Simulations can play a decisive role here, in particular those based on quantum mechanics, which is essential to accurately describe materials in conditions of enhanced reactivity as those imposed by the mechanical stresses applied.

In this talk I will describe the computational tools we developed to i) perform multiscale simulations and design materials to reduce friction and provide fundamental understanding on chemical reactions activated by mechanical forces. ii) perform high throughput screening of solid interfaces. A database for interfacial properties, such as adhesion and shear strength will be presented and discussed.

Keynote Talks

Keynote I - Electrochemistry for energy: conversion, storage and integration for a self-powered Society

Federico Bella, Professor at Politecnico di Torino, Italy.

Efficiency, stability, sustainability and integration of energy devices are milestone targets towards world electrification and renewable sources-dependent society.

In this talk, new strategies for materials design, use of biosourced components, unconventional device architectures and structure-propriety-performance correlation will be shown for the design of different energy devices for the storage and conversion of energy.

In particular, the lecture will cover these intriguing aspects in the current energy scenario:

- Solar fuels from emerging conversion technologies.
- The design of stable cell components for post-Li batteries;
- The transition towards aqueous photovoltaics.
- The use new materials for allowing the design of integrated conversion/storage systems for cities and industries.

The importance of considering sustainability, stability and industrial scalability as main targets in the research work will be stressed, along with a real consideration of efficiency results presented in the literature.

This project has received funding from the European Union’s Horizon 2020 Research and Innovation programme (grant agreement No. 952169, project title: SYNERGY).

Keynote II - Innovative designs in (bio)sensors with plastic antibodies

Goreti Sales, Associate Professor at University of Coimbra, Portugal.

(Bio)sensors combine in a single device a (bio)recognition element and a transducing element. The biorecognition element is the one responsible for the selectivity of the biosensor, holding the ability to discriminate a target compound among several others. Many (bio)recognition elements may be employed, including materials from natural or synthetic origin. Plastic antibodies are synthetic biomimetic materials that have become attractive alternative to natural antibodies in (bio)sensors. They are tailored by imprinting a given target molecule on a polymeric network and then extracting this molecule from it. The vacant sites so generated display great affinity for the specific target molecule used at the imprinting stage, due to complementary spatial and electrostatic interactions. Combining (bio)sensors and plastic antibodies is a long and successful scientific story that has also been extended to the integration of renewable forms of energy to generate self-powered devices. Several approaches have been developed for this purpose. Some include photovoltaic and fuel cells, which will be discussed in this presentation.

Acknowledgements: European Research Council, through the Starting Grant 3P's/GA311086, and the European Commission, through Symbiotic/FET-Open/H2020/GA665046 and MindGAP/FET-Open/H2020/GA829040.

Keynote III - Molecular doping of metal catalysts for improving the electrochemical conversion of CO2 to multicarbon products

Damien Voiry, CNRS researcher at University of Montpellier, France.

The conversion of CO_2 via electrochemical processes is a relevant technology to close the carbon cycle; especially when combined with renewable energy sources. Because of their high market value and their high energy density, research has aimed at developing catalysts for the electrochemical conversion of CO_2 into multicarbon molecules. Copper (Cu) is one of the few transition metals that can efficiently catalyze the electrolysis of CO_2 to multicarbon products such as ethylene, ethanol, acetate, propanol. The design of Cu-based catalysts by adapting some of the concept of molecular catalysts in order to finely tailor the behavior of the active sites of metallic surfaces is currently regarded as the long-standing interest for the controlled design of novel electrocatalytic materials. Increasing the oxidation state of copper has been suggested to improve the CO_2RR performance and notably the formation of C_{2+} species.

In this context, we have proposed a new strategy to improve the conversion of CO_2 into hydrocarbon molecules with two or more carbon atoms (C_{2+}) *via* molecular doping of a metal catalyst. Specifically, we identified electrophilic functional groups that allow to direct the electrochemical reactions towards the production of C_{2+} species such as ethanol and ethylene and improve the reaction rates at the surface of the catalyst. In my presentation, I will review our recent findings on how the control of the oxidation state of Cu for controlling the selectivity of the CO_2 reduction reaction.

ORALS

A.1. Nanomaterials 0D to 3D

ID 44 - Investigating the mechanical behaviour of MoS_2 thin films under stress

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Due to its impressive mechanical, optical and electrical properties, molybdenum disulfide (MoS_2), among transition metals dichalcogenides (TMDCs), is a good candidate for applications in flexible electronics, optoelectronic devices and catalysis [1]. Characterising its mechanical response is thus a key-point to exploit its potential. It is the goal of our current research.

Transferring MoS_2 films from the growth substrate to other substrates of interest is a major challenge. Existing procedures lead to the destruction of the growth substrate, sample degradation and/or presence of sacrificial coating residue on the free surface. Therefore, we developed a new transfer method which guarantees a quick, reliable, chemical and impurity-free transfer to desired substrates while preserving the morphology and quality of the film for various physical analyses and applications.

To understand their mechanical behaviour, 2D films of MoS_2 transferred to polyether-ether-ketone (PEEK) substrates were subjected to uniaxial compression. The surfaces *in situ* evolution under increasing/decreasing strains was investigated by scanning probe microscopy. Straight-sided buckles are evidenced above a specific strain, lying perpendicularly to the (Ox) compression axis (Fig. 1). Some of them are observed to interact together, as described in the case of high elastic contrast between the film and its substrate [2]. The results are presented and discussed in the framework of the elastic theory describing the buckling phenomenon.

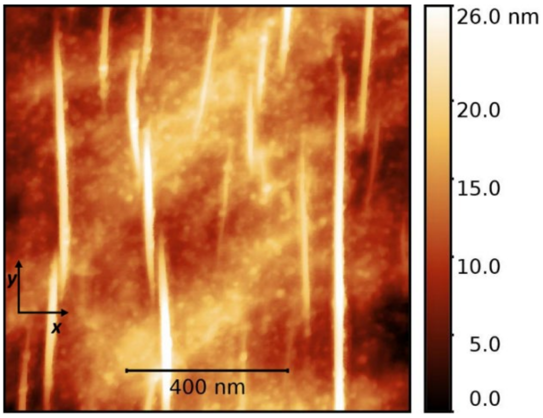

Fig. 1. MoS_2 monolayer buckles on strained PEEK substrate (ε= 6%)

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[2] Coupeau *et al.,* Journal of the Mechanics and Physics of Solids 124 (2019) 526.

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ID 100 - Dense and Mesoporous FePd Nanowires as Excellent SERS-active Substrates

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Dense and mesoporous FePd nanowires (NWs) of different diameters and compositions (ranging from $Fe_{29}Pd_{71}$ to $Fe_{60}Pd_{40}$) have been successfully fabricated by employing template- and micelle-assisted pulsed potentiostatic electrodeposition method. The NWs were electrodeposited into nanoporous anodic alumina (AAO) and polycarbonate (PC) templates of varying pore sizes. An FePd electrolyte was utilized for obtaining dense NWs while a block copolymer, P123, was added to this electrolyte as the micelle-forming surfactant to produce mesoporous NWs. The as-prepared NWs have a face-centered cubic structure and exhibit a range of length, from 3.1 μm to 7.1 μm. PC-derived dense NWs are longer and more continuous compared to their AAO-derived counterparts. The mesoporous NWs have a Pd-rich composition and reveal a core-shell structure where the porosity is only witnessed in the internal volume of the nanowire while the outer surface remains solid and non-porous. Fe incorporation was favoured at more negative potentials and some dependence of composition on pore size was also observed. Then, the NWs partially embedded in the AAO template were examined as active substrates for Surface-enhanced Raman Spectroscopy (SERS). The NWs display strong SERS effect for 4,4'-bipyridine probe molecule credited to the presence of multiple hotspots. Impressively low detection limit of $<10^{-12}$ M has also been recorded. Hence, the excellent SERS performance of the reported NWs holds remarkable promise for their application in the field of life science and ultrasensitive instrumentation.

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ID 125 - Modification and Characterization of Ag and CuO Nanofluids for Stability Control and Thermal Conductivity Enhancement

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An innovative method of improving heat transfer capability in modern cooling and heating systems is the use of nanofluids. Enriched fluids with nanoparticles, compared to conventional ones, increase the efficiency of these systems, due to their improved thermophysical properties. Experimental measurements carried out when heating buildings, using nanofluids showed that their application can result in up to a 38% reduction in energy needs [1]. However, instability (formation of aggregates, sedimentation) has been observed in the behaviour of nanofluids, which reduces their performance.

In the present work, silver and copper oxide nanoparticles suspended in deionized water (Ag/ DIW, CuO/ DIW) were studied under different pH and temperature conditions in order to find the region of maximum stability. Experiments were performed to characterize Ag/ DIW and CuO/ DIW nanofluids by obtaining UV-Vis spectra, changing the concentration, pH and dispersion medium from deionized water to deionized water/ ethylene glycol mixtures (DIW / EG) and then their stability under different temperature conditions was studied. In addition, the enhancement of thermal conductivity of water and ethylene glycol in the presence of Ag and CuO nanoparticles was investigated.

Experimental results have shown that Ag/ DIW, Ag /DIW /EG, CuO / DIW and CuO/ DIW/ EG nanofluids under distinct acidity (pH) and temperature conditions exhibit increased stability, strengthening the need for further research.

Keywords: Ag and CuO Nanofluids, Stability, Heating-cooling systems.

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ID 169 - MgO nanoparticles obtained by Pulsed Laser Ablation in Liquid - a study on fabrication versatility aiming different applications

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Nano and micro-sized metal particles are used in diverse fields, from electronics to biomedical. Among them, Magnesia, also known as magnesium oxide (MgO), is one of the most interesting metal oxides due to its unique properties, such as a large electrochemically active surface area and chemical stability.

Although chemical routes such as chemical reactions, thermal evaporation,sol-gel, chemical vapor deposition and hydrothermal treatments are mainly used to fabricate metal oxide nanoparticles, they require the use of toxic reagents and long processing times. Thus, developing a simple green synthetic process for preparing MgO nanoparticles remains a challenging topic of investigation. Pulsed laser ablation in liquid (PLAL) has emerged as a potential alternative to chemical methods because it does not require chemicals, generates no waste, and produces high-purity particles. By changing some parameters such as the laser wavelength, laser fluence, and liquid media the size and shape of the particles produced can be tailored. The obtained particles can be added to act as anti-biofilm agents on coatings.

Both influence of the liquid medium and the laser energy on nanoparticle composition and morphology are investigated in this work, as well as the effects of laser energy and different liquid media on the properties and characteristics of these nanoparticles.

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A.2. Flexible and porous materials

ID 91 - Nanoporous Gold synthesis from metastable Au33Fe67 alloy for Surface Enhanced Raman Spectroscopy applications

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Nanoporous gold (NPG) can be produced by dealloying, a process in which the less noble elements in an alloy are chemically or electrochemically dissolved into an electrolyte, leaving on the surface the noble element to form ligaments and pores with size of tens or hundreds of nanometers, characterised by excellent optical and electrochemical properties.

In this work, a low-cost NPG has been prepared by chemical dealloying of a $Au_{33}Fe_{67}$ supersaturated solid solution, whose ribbons were obtained by rapid solidification technique. The dealloying procedures were carried out in 1 M HNO_3 at 70 °C for varying durations. As-quenched ribbon and dealloyed samples have been structurally and compositionally investigated using XRD, FESEM and EDS techniques. The obtained NPG is homogeneous with tunable ligament size and shape (Fig. 1), easy-to-handle and free-standing. Surface-Enhanced Raman Scattering (SERS) was explored using 4,4′-bi-pyridine as probe molecule. Strong SERS effect has been observed with low detection limit of 10^{-15} M. The NPG sample performs excellently as an economical and highly sensitive SERS-active substrate for potential applications in life science and ultrasensitive instrumentation.

Most notably, a metastable precursor has been favourably obtained from an immiscible Au-Fe system. Pairing Au with cheap and abundant Fe and fabricating an Fe-rich precursor gives an exceedingly cost-effective starting material. No usage of critical raw materials is involved. Then, employing a straight-forward and rapid dealloying procedure to obtain the NPG sample, makes for an overall inexpensive and sustainable production.

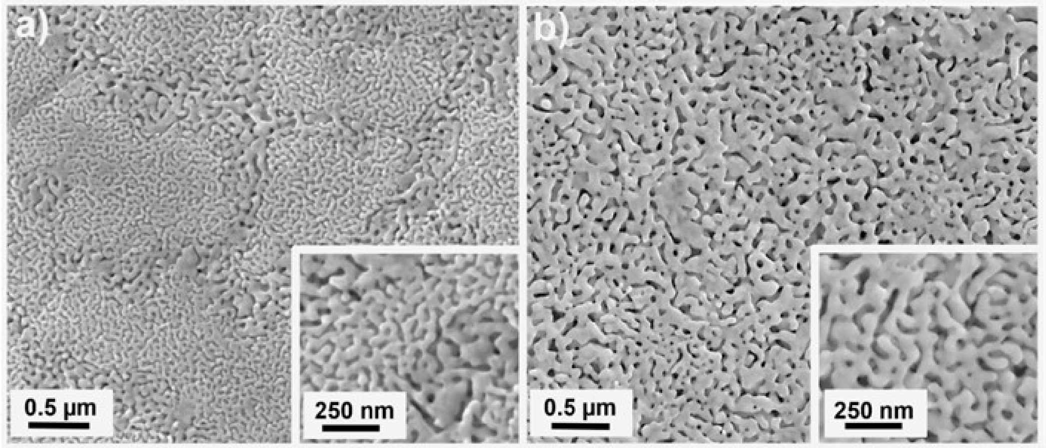

Fig 1: SEM-SE images of (a) NPG_1h; (b) NPG_2h

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ID 127 - Tunable wettability of laser-induced graphene through processing environment or processing parameters

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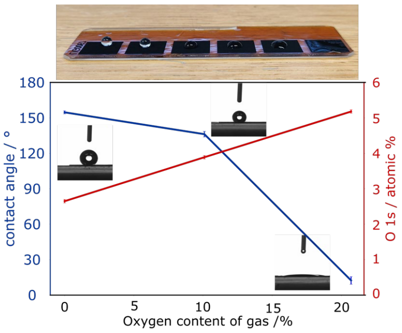
Laser-Induced Graphene (LIG) created by laser scribing on a polymer precursor like polyimide exhibits many interesting properties. Along with creating conductive patterns embedded in flexible substrates the wettability of these materials can be finely tuned by simply changing the IR laser processing parameters. The water contact angle (CA) of LIG could be tuned from a superhydrophilic state -with a CA $\varphi = 0°$ - to a superhydrophobic state with a CA $\varphi = 150°$. Two different strategies were investigated: 1) tuning of the scribing pattern, 2) change of the O_2 content of processing atmosphere through local air/N_2 purging. The resulting CA of the different LIG types were investigated together with the chemical surface composition, measured by XPS (X-ray Photoelectron Spectroscopy). A correlation between the oxygen content of the surface and the CA could be observed. This opens the possibility to manipulate and direct fluids on the LIG surface; demonstrators like a fluidic device were fabricated based on these findings. Inspired by the Namib desert beetle a biomimetic application in fog basking was demonstrated. Sheets of polyimide patterned with various types of LIG were used to collect water from fog, with a collection rate similar to other recent but more complicated approaches. An environmental scanning electron microscope (ESEM) was used to investigate the condensation and wetting of water on LIG at the microscale.



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A.3. Smart and self-healing materials

ID 74 - Advances in isocyanate microencapsulation for new ecological and mono-component adhesives

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Adhesives used in the footwear industries typically result from two-component formulations, consisting of isocyanate species (cross-linkers) and a polyol-based component (OH prepolymer). The present work aims to develop a new mono-component adhesive in which microencapsulated isocyanate is added to the OH prepolymer, preventing health hazards associated with the direct contact to isocyanate. The microcapsules (MCs) are designed to respond to the external stimuli of pressure and/or temperature applied during the footwear manufacture, at the same time offering enough storage stability and chemical resistance.

The solvent evaporation technique combined with a double microemulsion system was used to obtain MCs with a polycaprolactone (PCL) shell by a purely physical process, containing monomeric but also more reactive oligomeric isocyanate species. The newly developed MCs contrast with the Polyurethane/Polyurea (PU/PUa) ones, synthetized using the interfacial polymerization technique, typically used for the isocyanate microencapsulation: (i) biodegradability of PCL, which makes these MCs not a microplastic, by definition, (ii) a high hydrophobicity, which contributes to a longer shelf-life, (iii) and a low melting temperature and low melting viscosity which enables the melting process of the MCs and promotes the homogeneous distribution of the isocyanate within the adhesive joint material.

Optimization studies using the MODDE® software enabled to obtain MCs with a core-shell morphology, a high encapsulation rate of 60 wt%, and a narrow size distribution.

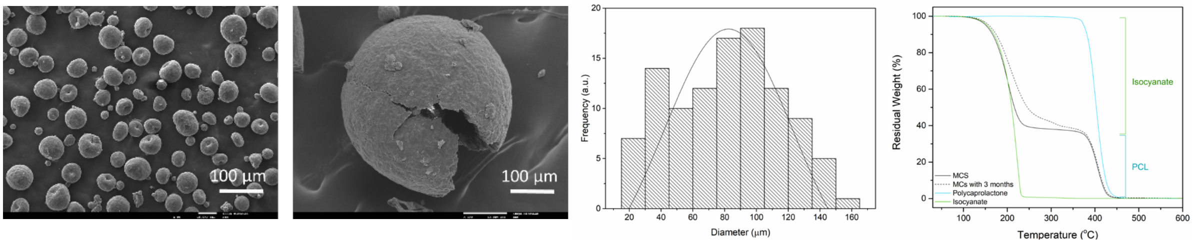

Figure 1. A) and B) SEM images of the PCL MCs containing encapsulated isocyanate C) PCL MCs' size distribution D) TGA of the PCL MCs, PCL MCs afther 3 months and respective components.

The present work brings an advance to the state of the art, in what regards the encapsulation of reactive isocyanate species to increase their shelf-life and response to external stimuli.

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A.4. Surfaces & Interfaces

ID 131 - Anti-slipping Winter Shoe-soles: A Nature Inspired Solution

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Walking on dry/wet ice during winters is troublesome, as Slips and Falls (SFs) appear to be a major challenge. SFs result in millions of severe injuries every year in Nordic regions of Europe, USA, Canada, and UK. These injuries cost billions of euros on economic fronts (healthcare, insurance burdens), loss of skilled workers for industries (the workers who face SF during their travel to work or else), permanent injuries, and deaths. SFs rank second among the causes of human injuries. The existing solutions to avoid or reduce SF injuries on icy surfaces composed of the use of spikes/crampons/studs, anti-slipping shoes with changed tread patterns or modified sole materials. However, prolonged and indoor use of spikes/crampons is not possible. Elderly people cannot use them and putting on/off is another challenge. Modified anti-slipping shoes lose their effectiveness quickly and once the ice sticks or fills up the patterns, they become inefficient. Some better modified anti-slipping shoes are very expensive. We propose a nature inspired laser-based tailoring of the shoe-sole to reduce SFs on icy surfaces. The prominent cause SF on ice is a micro-layer of water (wet ice) on the surface. The inspiration from gecko and frog provides a solution as these creatures have excellent surface adhesiveness on wet or dry surface. Shoe-sole material (silicone rubber) is laser textured combining hierarchical structures inspired from gecko and frog foot. The textures offer adhesion because of the van der Waal and capillary forces. The solution offers better protection against SFs on icy surfaces.

ID 207 - Surface treatment of stainless steel 316L towards poly(L- lactic) acid polymer adhesion

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In the current work we provide an approach for efficient functionalization of the surface of medical grade Stainless Steel 316L (SS316L) for the successful application of a thin film of the polymer poly(L- lactic) acid towards pronounced bioactivity. Two methods, thermal action and UV irradiation, we confirmed suited well for the treatment procedures due to the formation of OH groups on the metal surface being of great importance for the silanization process. The use of conventional thermal treatment procedure causes toxic chromium oxide formation while the UV-irradiated SS316L substrates were found identical to those of pristine ones without prominent surface changes. The effect of charge trapping on the surface after both treatments was observed playing a significant role in drastic changes in the work function regarding pristine SS316L. The UV treatment was solely found allowing control of the electrocatalytic activity of SS316L substrate. The effect of clusterization was observed in thermally treated substrates due to the low value of contact angle (28.07 ±3) and increased value of work function (φ= 5.2 eV). This causes the better results of the cross-hatch test demonstrated for the PLLA film deposited on the UV treated SS316L substrates due to significantly increased adhesion forces.

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A.5. Energy and environment materials

ID 28 - Sodium and Potassium ion rich ferroelectric solid electrolytes for electrode-less structural batteries

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The transition to a sustainable society is vital and requires electrification. In paving the way for a more ecological energy world, sodium and potassium ion-based electrolytes will likely play an important role in energy storage as these elements are very abundant. The latter cations and chlorides are especially interesting since life on the planet is somehow based on biological transfers of these ions through cell membranes. K+ is the key charge carrier in plants.

Here, we characterize electrochemically, and electrostatically, novel electrolytes, K2.99Ba0.005ClO, and compare their performance with Na2.99Ba0.005ClO in asymmetric structural electrode-less cells, such as Al/K2.99Ba0.005ClO composite/Cu, Al/Na2.99Ba0.005ClO composite/Cu, Zn/Na2.99Ba0.005ClO composite/Cu, and Zn/K2.99Ba0.005ClO composite/Cu at temperatures that range from 0 to 40°C. As in Na+-based electrolytes, the ferroelectric character of the K+-based electrolytes is well recognizable, especially at 40ºC in Al/K2.99Ba0.005ClO composite/Cu asymmetric structural electrode-less cells and Zn/K2.99Ba0.005ClO composite/Cu asymmetric. As in Na+-based electrodes-less structural battery cells, self-charge and self-cycling phenomena are also demonstrated reinforcing the ferroelectric nature of the A3ClO (A = Li, Na, and K) family of electrolytes. These studies may contribute to understanding K+ and Na+ transfer behavior in energy harvesting and storage as well as the biologic world.

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ID 148 - Chitosan modified silica aerogels for the adsorption of pollutants

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Wastewaters can feature several types of pollutants. Of great relevance are heavy metals and antibiotics, the presence of the latter in wastewaters is not regulated in the EU. Antibiotics have been giving increasing attention, due to their effect on marine life and on the surge of antibiotic resistant bacteria. In this work, chitosan and silica are used to create hybrid aerogels for the removal of pollutants belonging to the aforementioned groups from wastewaters.
Chitosan is widely studied for its antimicrobial activity, biodegradability and biocompatibility. It also features amine functional groups that are important for the sorption of several pollutants. Silica aerogels are versatile, extremely light and porous materials, which is why they were previously employed as adsorbents. The aerogels are obtained by reticulating the polymeric chitosan chains with silane moieties to form cohesive gels, followed by freeze-drying. Their chemical composition and structural properties are assessed. The impact of the addition of the silica phase is evaluated, in comparison to chitosan reticulated gels and the pristine chitosan powder. The adsorption performance of the different gels towards antibiotics (tetracycline) and metal ions (e.g., copper and nickel) is evaluated through sorption isotherms and kinetics.

Acknowledgments:
Work developed under the scope of the project "BIOSHELL", Ref. "BLUEBIO/0003/2019 – Recycling Crustaceans Shell Wastes for Developing Biodegradable Wastewater Cleaning Composites"), financed by FCT within program ERA-NET Cofund on Blue Bioeconomy (BlueBio) – Unlocking the Potential of Aquatic Bioresources.

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ID 167 - A New Supramolecular Liquid for Energy-Storage Applications

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Aqueous non-lithium based rechargeable metal-ion batteries are emerging as promising energy storage devices thanks to their attractive rate capability, long-cycle life, high safety, low cost, environmental-friendliness, and easy assembly conditions. Nonetheless, the narrow electrochemical stability voltage window of aqueous batteries (∼1.23 V), restricts the optimal choice of cathode and anode materials. This excludes most electrochemical couples that occur above the output voltage of 1.5V, which limits the enhancement in energy density of full devices. Highly concentrated "water-in-salt" (WIS) electrolytes can efficiently expand the stable operation window, which brings up a series of aqueous high-voltage rechargeable batteries. In the WIS electrolytes, all water molecules participate in the ion solvation shells, and no "free" water remainders can be found. This has been extensively used in lithium-ion batteries (LIBs). However, the economic and environmental concerns, as well as the rarity and increasing consumption of Li resources, restrain the scalable applications of lithium-based electrochemical devices. Recently, aqueous zinc-ion batteries, due to their remarkable thermal stability, high theoretical specific capacity, intrinsic safety, and low cost of the Zn metal, are considered to be the most promising alternative to LIBs. However, their extensive applications are still limited by suitable aqueous electrolytes with excellent thermal properties and safety. We have recently reported that hydrophobic solvation of small organic molecules triggers the formation of a unique, highly incompressible supramolecular liquid, involving essentially all water molecules of the solvent. And this translates to a broader electrochemical stability voltage window due to the corrosion resistant properties of this new fluid when tested as electrolyte. More importantly, this new fluid has recently allowed the exploitation of fast electron-transfer kinetics in a zinc-ion battery showing its potential in the energy-storage.

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ID 191 - Sol-gel derived di-ureasil based ormolytes for electrochromic devices

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In the context of the United Nations Sustainable Development Goals for 2030 one of the major tasks in the world is to improve energy efficiency and energy savings (goal 7) and to promote more sustainable cities and communities (goal 11) [1]. Modern buildings have large façade of windows and rooftops, that provide good daytime illumination, but reduce the energy efficiency of the buildings.

To overcome this problem new technologies that use electrochromism have been proposed to control the amount of sunlight (visible (VIS) radiation) and solar heat (near-infrared (NIR) radiation). This fine control will allow reducing energy consumption, while increasing the occupant's thermal and visual comfort. Electrochromic devices (ECDs) undergo a reversible color change upon application of a small voltage (1-3 V). They present a multilayer structure: an EC film of the cathodically coloring tungsten trioxide (WO_3), an electrolyte film, an EC film of the anodically coloring nickel oxide (NiO_x), and two outermost transparent conductive oxide (TCO) layers [2].

In the present work the sol-gel method [3] was employed to prepare a series of di-urea cross-linked poly(oxyethylene)/siloxane (di-ureasil) hybrid electrolytes doped with a lithium salt, an ionic liquid (IL), or a mixture of both. The structure, morphology, thermal behaviour, ionic conductivity and wettability of the resulting films was characterized. The electrolytes with optimized formulations were used as ormolytes in the assembly of ECDs whose electro-optical performance was evaluated by means of chronoamperometry, cyclic voltammetry, UV-VIS spectroscopy, and colour coordinates calculation.

Acknowledgments
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A.6. Biomaterials and healthcare

ID 8 - Effect of bactericidal agents Ga and Cu on structural and mechanical properties of β-TiNb alloys

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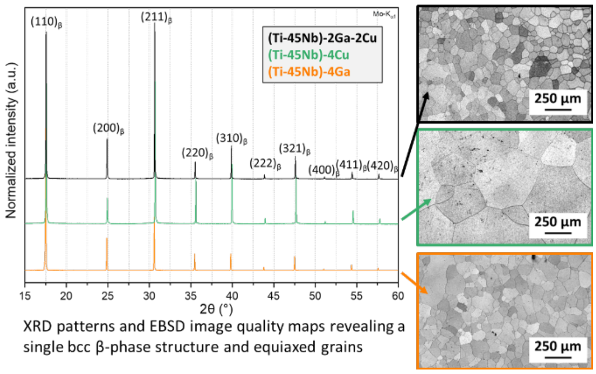
Titanium is the material of choice for the manufacture of bone-related implants because of excellent corrosion resistance and good biocompatibility. The occurrence of premature implant failure due to implant-associated infections, however, remains a prominent concern for clinicians. One strategy of creating functional antibacterial materials is adding a bactericidal alloying element which may prevent bacterial adhesion and colonization. Among metals with antibacterial properties, copper and gallium have shown superior *in vitro* antibacterial performance [1].

With the aim of developing new antibacterial Ti-alloys, we produced by two steps casting (arc melting + cold crucible casting) novel beta-TiNb alloys containing minor additions of Cu and Ga.

In the present study we investigated the structure and mechanical properties of three novel Ti alloys: (Ti-45Nb)-4Ga, (Ti-45Nb)-4Cu and (Ti-45Nb)-2Ga-2Cu (wt.%). Microstructural investigations revealed a beta (bcc) single-phase structure with coarse equiaxed grains in all alloys. The measured Young's moduli are lower (E = 69 ÷ 100 GPa) than those of clinically used metallic biomaterials. Microhardness and strength are observed to be higher in the quaternary alloy. Their elastic admissible strain (stress-to-modulus ratio; ε = 0.50 ÷ 0.90), makes them potentially suitable for load-bearing implant applications if compared to CP-Ti (ε = 0.46) or SUS 316L steel (ε = 0.09) [2].



XRD patterns and EBSD image quality maps revealing a single bcc β-phase structure and equiaxed grains

This project received funding from the EU's Horizon 2020 research and innovation programme under the MSCA grant agreement No. 861046 (BIOREMIA ETN).

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ID 18 - Multifunctional copper and strontium co-doped mesoporous bioactive glass: Preparation, characterization and bioactivity

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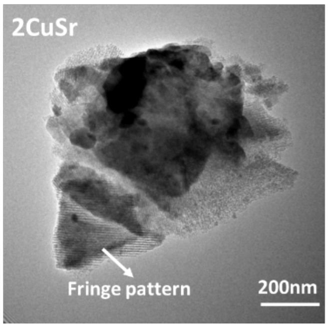
Mesoporous bioactive glass (MBG) is an extensively studied material for the healing of bone defects due to its controlled mesoporous texture, large surface area and ability to bond with living bone tissues provides a smart platform for bone regenerative medicines. Its properties can be tailored by introducing metallic ions such as strontium(Sr) and copper(Cu) which can offer osteogenesis, angiogenesis and antibacterial effects on bone formation [1]. In the present study Cu and Sr ions are co-doped (ratio1:1) upto 2 mol% each in $80SiO_2$-(15-x)CaO-$5P_2O_5$ MBG structure and synthesized using sol-gel and evaporation induced self-assembly technique. XRD confirmed the amorphous nature of the glass while Raman confirmed the presence of Si-O-Si and P-O bands. Compositional analysis were done by ICP-OES, confirmed the presence of dopant ions in added amounts. However, the amount of phosphorus was less compared to the nominal compositions. Surface area of co-doped powders was relatively high upto 378 m^2/g in comparison to base glass (upto 254 m^2/g). All the powders were scanned by TEM showing a fringe pattern that represents the formation of a highly ordered mesoporous structure. The CuSr doped MBG showed a positive effect on apatite formation when immersed in SBF, although the release rate of Cu and Sr ions was relatively slow for 1mol.% of co-doping which signifies stable net structure of the glass. The apatite layer was further confirmed by XRD and SEM. The results suggested that CuSr doped MBG can be used for multifunctional applications such as bioactive fillers and drug delivery system.



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ID 79 - Synthesis and Characterization of Silicate and Zinc Co-Substituted Hydroxyapatite

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The use of bioactive materials in bone repair and regeneration has been of interest for several years. Ideally, an implant would assist in the tissue repair and then be resorbed by the body. One way to accomplish this is to use composites comprising a biodegradable polymer matrix reinforced with bioactive ceramic filler particles. At the same time, implant-related infections are a significant problem, therefore it would be ideal if the composites could offer antibacterial properties.

The objective of this work is to explore the production and incorporation of silicon and zinc ions into hydroxyapatite (HAp), to produce a ceramic filler material with a combination of antibacterial and osteoconductive properties. However, to understand the effects of the material on the biological response, it is important to characterise fully the materials that have been produced.

To that end phase pure powders of HAp, Si-HAp (1.5 wt% Si), Zn-HAp (0.65 wt% Zn), and Si-Zn-HAp (1.5 wt% Si, 0.65 wt% Zn) were synthesised using a wet precipitation method. All materials were thoroughly characterised using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and X-ray fluorescence (XRF) to confirm phase purity, the presence of the substituting species, and quantify trace elements. Scanning electron microscopy (SEM) and laser diffraction were used to investigate morphology and particle size. The powders were also processed into dense ceramic discs for further testing.

Phase pure, single and co-substituted apatites have been successfully prepared in both powder and sintered forms and will be investigated regarding their antibacterial potential.

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ID 80 - Improving the interface between orthopedic implants and bone – a comparison between different surface treatments

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Orthopedic implants for load-bearing applications are usually composed of titanium-based materials. However, insufficient bioactivity of metallic materials impairs the bonding with bone, compromising osseointegration at an early stage. The implant-bone interface may be improved by regulating some surface properties of the biomaterials, including surface chemical composition, surface energy, roughness and topography, which influence the behavior of bone cells.
In the present study, commercially pure titanium and Ti6Al4V alloy were used to investigate the effect of three surface treatments, after performing two different chemical pre-treatments, on the characteristics of the obtained oxide films. Regarding the pre-treatments, no major differences were observed between performing alcohol cleaning or acidic pre-treatment, considering the surface crystallinity, roughness and wettability. However, the TiO2 layer formed upon anodic oxidation, hydrothermal treatment and anodic oxidation followed by hydrothermal treatment presented different characteristics regarding its crystallinity, roughness, thickness and wettability.
This study compared specific surface treatments and the hydrothermal treatment is proposed as a simple treatment capable of improving the characteristics of the implant surface, thereby promoting osteoconductivity. In fact, the culture of human Mesenchymal Stem Cells on Ti-based materials subjected to hydrothermal treatment and consequent induction of osteogenic differentiation confirm the improved surface characteristics.

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ID 95 - ATR-FTIR/Micro-Raman Spectroscopy are powerful techniques to disclose chemistry and polymerization of novel smart self-adhesive composites

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Understanding physico-chemical properties of dental composites is pivotal to predict their behaviour in the oral environment. However, it is often required to employ several different techniques to characterize materials. To overcome this, vibrational spectroscopy can be used to simultaneously assess chemical composition, filler type/distribution, filler/polymer ratios, and free-radical addition polymerization kinetics of composites. Three commercial self-adhesive materials (Activa^{TM} Kids, AK; Constic, CC and Vertise^{TM} Flow, VF) and one experimental (SMART_1) were tested. SMART_1 was prepared by combining urethane dimethacrylate, poly(propylene glycol) dimethacrylate and adhesive monomer 4-META, with a hybrid glass filler phase containing remineralising particles of monocalcium phosphate and antibacterial poly-l-lysine (powder/liquid ratio=3:1). An ATR-FTIR was used to determine real-time polymerisation kinetics of 2x10 mm composite discs (37ºC). A semi-quantitative model was constructed based on the Beer-Lambert Law to analyze composition. Polymerized discs were polished and assessed using micro-Raman, by mapping a 40x40 µm area to characterize filler chemistry/dispersion. ANOVA with Tukey's HSD were employed to compare means (α=0.05). Modelling was consistent with CC/VF having 35% Bis-GMA and AK 45% UDMA. Degree of conversion was largely material-dependent (ANOVA, $p<0.001$), with Constic showing the highest (82±2%), while ACTIVA the lowest (67±2%). Rates showed a similar trend. Shrinkage was greater in Constic (5.8±0.1 vol%) and VF (5.3±0.1) when compared to SMART_1 (4.6±0.1; $p<0.05$). ACTIVA showed the largest particles (>12 µm), with a high filler/polymer ratio, while CC/VF had similar microstructural features. It can be concluded that ATR-FTIR/Raman techniques are especially valuable to characterize novel dental polymer formulations and compare them to commercial competitors.

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ID 96 - Engineered porous FeMn alloys with addition of Ag as antibacterial element for biodegradable implant applications

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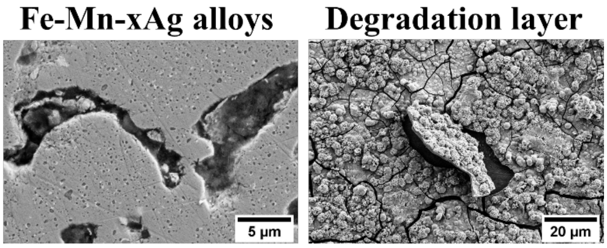
Biodegradable implants constitute a new generation of biomedical materials that are being developed to assist the tissue healing processes and gradually corrode and degrade in-vivo after their function is being fulfilled. Among others, Fe-based alloys show promising mechanical properties such as ductility and high ultimate strength. However, the corrosion rate of those implants is relatively slow, therefore alloying with other elements such as Mn, is studied to accelerate the biodegradation rate.



In this study, porous Fe-Mn alloys with additions of Ag have been fabricated via powder metallurgy route and characterized in terms of their microstructure, mechanical and magnetic properties, degradation behavior, biocompatibility and antimicrobial performance. With this purpose in mind, four different compositions have been designed – equiatomic FeMn alloys, and FeMn alloys with 1,3 and 5 wt.% of silver additions. Antibacterial Ag, aimed at limiting the infection rates of implants, is added to the $Fe_{50}Mn_{50}$ base alloy using mechanical alloying of metallic powders. The high content of Mn is proposed to ensure the non-magnetic character of the investigated alloys, even during degradation. A long-term biodegradability test has been performed in terms of ion release and degradation surface analysis (see figure) as well as the evolution of magnetic properties upon immersion.

Our results reveal that porous, non-cytotoxic, metallic FeMn-based biomaterials with higher degradation rates can be fabricated by the powder metallurgy route. The addition of Ag is believed not only to accelerate the corrosion rate but also to limit the biofilm formation on the surface of implants.

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ID 111 - Dealloying a Ti-Cu based Amorphous Alloy for Enhancing its Antimicrobial Properties for Biomedical Implant Application

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Ti-based biomaterials have been widely used for decades in the medical industry for hard tissue implant applications. This is possible due to their superior properties like high load-bearing capacity, corrosion resistance, and relatively low young's modulus. However, due to both the high stress-shielding effect and biofilm formation, there are several cases of implant rejection in the patient's body. To overcome this problem, a novel composition of $Ti_{40}Cu_{40}Zr_{11}Fe_3Sn_3Ag_3$ at% was developed containing good glass formers and biocompatible elements. Metallic glass ribbons of 50 µm thickness and 10 mm width was developed using melt spinning technique. In order to produce antimicrobial implant materials for inhibition of biofilm formation, the surface of the ribbon sample was chemically modified using chemical dealloying to selectively remove Cu atoms from the surface of the ribbon samples. In this study, we investigated the effect of several electrolytes ($FeCl_3$, HNO_3, HF, $(NH_4)_2S_2O_8$, H_2SO_4, piranha solution, aqua regia, NaOH, artificial seawater) on dealloying of Cu and optimized dealloying parameters like temperature, electrolyte concentration, and time for producing nanoporous structure on ribbon samples. The surface morphology and composition of dealloyed samples was investigated using XRD, SEM and EDS analysis. Further, the wettability, hemocompatibility, and antibacterial activity of the dealloyed samples were investigated to understand the surface chemistry and antimicrobial property for potential application as hard tissue implant material.

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ID 143 - Laser-assisted surface modification of zirconia-based materials to guide osteoblast response for dental applications

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The surface topography of dental implants is a key parameter to achieve a strong and direct integration with the surrounding bone, however, the optimal topography that maximizes the osseointegration still remains unclear. Considering that, laser surface micro-patterning of dental-grade zirconia (3Y-TZP) was explored with the objective of providing a topography able to guide bone-cell response. A nanosecond laser was employed to fabricate micro-groove patterns on the surface of 3Y-TZP discs, yielding three different topographies according to the groove periodicity (30, 50 and 100µm). The resulting topography, surface damage and hydrothermal degradation behaviour was assessed by means of advanced characterization techniques. A cellular study was conducted to evaluate the behaviour of human mesenchymal stem cells (MSCs) on the patterned samples in terms of adhesion and cell morphology. The topographical analysis of the three patterns showed grooves of approximately 1.7µm height that exhibited surface damage in the form of pile-up, microcracks and cavities due to the melting and re-solidification associated to the nanosecond regime. The accelerated aging test revealed a slight decrease in the hydrothermal degradation resistance after the laser patterning, and interestingly, a non-homogeneous monoclinic phase distribution was observed along the laser textured surfaces. Finally, the three micropatterns enabled MSC attachment and increased cell area compared to polished zirconia. Moreover, cell morphology and alignment were influenced by the periodicity of the patterns. Among all the microtopographies, only the 50µm periodicity, which better mimics the size of the cells, significantly favoured cell elongation and alignment along the grooves, which is associated with a higher cell migration, thus paving the way to further explore this topographical pattern to tune the response of MSCs on dental zirconia.

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ID 174 - Tackling implant infections: chitosan layers loaded with gold nanoparticle conjugated with gentamicin for orthopedic applications

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The trends in development of novel biomaterials put a heavy emphasis on their antibacterial properties, as implant infections lead to substantial amount of revision surgeries today. Early and precise treatment is crucial due to change in the state of opportunistic bacteria colonizing implant surface, from planktonic to sessile biofilm. This transformation makes conventional antibiotic treatment difficult, leading to persistent and chronic infections. Several solutions are being described in recent literature, involving both the bacteriostatic and contact killing mechanisms. The latter involves antibacterial nanoparticles, antibacterial peptides or antibiotics conjugated with nanocarriers. Antibiotic conjugates gained considerable scientific attention, since they utilize existing drugs that are amplified or modified by specific carrier, surpassing their limitations.

Our work presents a novel approach to handling the threat of bacterial infections in orthopedic applications by modifying Ti6Al7Nb alloy with chitosan layers loaded with gold nanoparticle (AuNP) – gentamicin (GE) conjugates. Mildly bacteriostatic chitosan provides biocompatible matrix for modified AuNPs. On the other hand, the usage of AuNP-GE enables localized antibiotic delivery, using the ability of AuNPs to adhere to the bacterial cell walls. Size of AuNPs (TEM, ZetaSizer) and subsequent chemical stages during conjugation (ATR-IR) were monitored. Layers deposited on chemically etched titanium alloy, were thoroughly analyzed in terms of atomic and chemical composition by means of EDS and IR spectroscopy. The topographical surface state after chemical treatment and subsequent deposition were measured by SEM and optical profilometry. Finally, functional parameters, such as mechanical parameters and corrosion resistance, as well as biological and antibacterial properties were examined.

Acknowledgements

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ID 180 - Near-infrared light-responsive nanoparticles as an efficient carrier to cross the blood-brain barrier

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Currently, there are no effective therapies available to completely reboot the brain function after occurence of neurodegenerative diseases. Blood-brain barrier (BBB) is a bottleneck in development of drugs or drug carriers for the treatment of brain diseases. The transient opening of the BBB using the heat generating nanoparticles (NPs) upon near-infrared (NIR) light exposure has been proposed to target the brain. However, it remains elusive if the local heat can activate pro-inflammatory conditions, which affect the integrity of BBB. Here, we demonstrate that cytokines and chemokines released by macrophages after exposure of PDA NPs and NIR light do not compromise the integrity of BBB model.

We have synthesized 63±3 nm PDA NPs, which is conjugated with 180 transferrin (Tf) peptides. Results obtained from in vitro BBB model showed that 50 μg/mL of Tf180-PDA NPs crossed the BBB more efficiently than the PDA NPs after the exposure of NIR light (wavelength 785 nm, power density 2W/cm2) for 5 min. Importantly, 50 μg/mL Tf180-PDA NPs and PDA NPs do not affect the integrity of the BBB after the exposure of NIR light. There is no significant change in the integrity of in vitro BBB model after incubation with the conditional media containing cytokines/chemokines secreted by mouse macrophages.

We show that light responsive Tf180-PDA NPs can transiently open the BBB upon the exposure of NIR light and thereby promoting greater crossing of NPs.

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ID 206 - Photo-degradable, tough and highly stretchable hydrogels

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This work present for the first time highly stretchable and tough hydrogels with controlled light-triggered photodegradation. A double-network (DN) of alginate/polyacrylamide (PAAm) is formed by using covalently and ionically crosslinked subnetworks. The ionic Ca^{2+} alginate interpenetrates a PAAm network covalently crosslinked by a bifunctional acrylic crosslinker containing the photodegradable o-nitrobenzyl (ONB) core instead of the commonly used methylene bisacrylamide (MBAA). Remarkably, due to the developed protocol, the change of the crosslinker did not affect the hydrogels' mechanical properties. The incorporation of photosensitive components in hydrogels allows external temporal control of their properties and tuneable degradation. Rheological and mechanical analysis demonstrated the loss of properties of ONB based DN hydrogel after irradiation, confirming the efficient destruction of the covalently crosslinked network, resulting in the loss of mechanical properties. Scanning electron microscopy (SEM) analyses also visually confirmed the network photodegradation. Cell viability and cell proliferation assays revealed that hydrogels and their photodegradation products are not cytotoxic to the NIH3T3 cell line. In one example of application, these hydrogels were used for bio-potential acquisition in wearable electrocardiography. Surprisingly, these hydrogels showed a lower skin-electrode impedance, compared to the common medical grade Ag/AgCl electrodes. This work lays the foundation for the next generation of environmentally friendly though and highly stretchable hydrogels in a variety of fields such as health, electronics, and energy, as they combine excellent mechanical properties with controlled degradation.

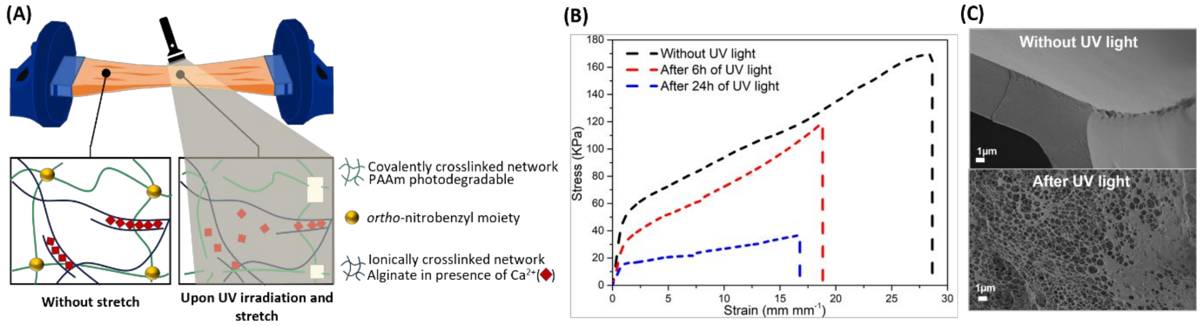

Figure 1. Overview of the approach. (A) Structure of ONB based hydrogel. After UV irradiation, the ONB crosslinkers undergo an irreversible photocleavage, breaking the PAAm hydrogel crosslinks. When stretched the PAAm network cannot stabilize the deformation, while the alginate network unzips progressively, resulting in a high loss of mechanical properties. (B) Stress-strain curves curves of photo-degradable hydrogel without and after irradiation (C) Representative SEM images of the cross-section morphologies of photo-degradable hydrogels before and after 24 h of irradiation.

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ID 210 - Antimicrobial porous Ta2O5 surfaces with Zinc nanoparticles deposited by magnetron sputtering

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The rejection of dental implants can be minimized by modifying implant surface properties.
This research is focused on the surface modification of metallic tantalum (Ta), by plasma electrolytic oxidation (PEO), to promote surface bioactivity and accelerate the osseointegration by mimicking the bone's morphology and chemical, with the formation of micro/nano-porous structures and the incorporation of osteoconductive elements. Over these bioactive surfaces, zinc (Zn) nanoparticles (NPs) with and without a thin carbon (C) layer are deposited by magnetron sputtering to endow them with antimicrobial activity.
Different NPs quantities were tested. Increasing the deposition time and/or decreasing the working pressure, a higher Zn amount was deposited. The zinc ions release increased with the Zn quantity and decreased with the carbon layer addition. The chosen optimized surfaces, the ones with lower and higher ionic release, were studied regarding the antimicrobial activity and corrosion resistance. The Zn NPs significantly improved the antimicrobial activity, as the higher quantity of Zn NPs showed the highest C. albicans growth inhibition. The corrosion results revealed that the formation of Ta_2O_5 sharply improved the corrosion behavior, compared to untreated Ta surfaces. The incorporation of Zn NPs promoted the degradation of corrosion resistance, which improved as a function of immersion time and became closer to the porous Ta_2O_5 surface corrosion behavior.
The results prove the NPs ability to avoid biofilm formation and did not compromise the corrosion resistance of the porous Ta_2O_5 surface. Thus, PEO and magnetron sputtering are promising surface modification techniques for functionalize Ta surfaces for dental implants.

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A.7. Sensors & Actuators

ID 11 - Development and testing of a SMA-based proof of concept bending micro-actuator

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Shape memory alloys (SMAs) represent a highly attractive technological solution for the development of simple and reliable smart thermally driven actuators with sensing capability. The SMA functional thermomechanical behaviors derive from diffusionless martensitic phase transformation. Thermally driven SMA actuators (via Joule heating and/or environmental heating/cooling) display reversible strain changes. Slow cooling in the temperature range 20-90 °C of represents a drawback of SMA actuators while a very high power/volume ratio is the greatest advantage.
This study aims at the development of a smart micro sensor/actuator for ventilation systems consisting in bent NiTi alloy ribbon embedded into a soft 3D-printed polymeric case. Since the NiTi element works against the bending stiffness of the host polymer matrix, reversible bending deformation is obtained upon thermal cycling. The operation of the smart actuator relies on the optimized recoverable bending deformation of the NiTi ribbon in the desired temperature range and on the elasticity of the polymer. The micro sensor/actuator was designed, fabricated and tested in a purpose-built test bench, where the heating cooling was achieved via forced airflows as common in ventilation systems. Thermomechanical characteristics of the NiTi ribbon in bending mode (necessary for the design) were evaluated by dynamic mechanical analysis. Various NiTi ribbon geometries, transformation temperatures, shape setting parameters and geometries of the 3D-printed polymeric case were considered in the design of the actuator. The obtained results demonstrate that a smart SMA micro sensor/actuator with optimized functionality can be designed and built via combining bent NiTi ribbon in 3D printed polymer case.

ID 163 - Shape memory soft actuation exploiting multimaterial functionalization: design and control

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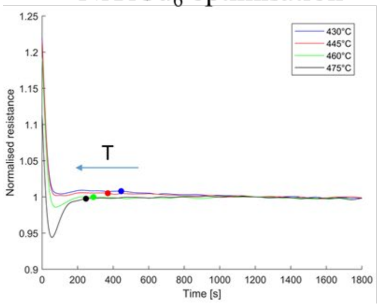
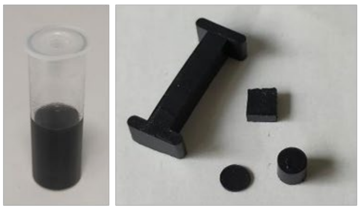
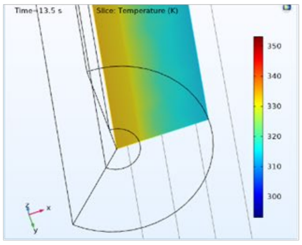
Shape memory alloys (SMA) are often employed in the fabrication of soft actuators, thanks to their inherent flexibility and high energy density [1]. However, their bandwidth is limited by the rather slow cooling rates, unless active cooling is employed. This work explores opportunities offered by the embedding of the SMA element in a functionalized matrix able to address not only external mechanical interfacing, but also thermal exchange and controllability, without the need for active cooling.

We shall present modelling and experimental results showing that an elastomeric matrix functionalized with carbon nanoparticles (CNP), particularly when coupled with a tailored switched controller that manages system nonlinearities, is able to improve the operation characteristics of multimaterial soft actuators based on SMA-based technology. Ternary NiTiCu has been selected thanks to its low hysteresis, providing faster response. The optimization of the SMA is carried out with an innovative technique that ensures high cyclic stability and mechanical properties [2]. The matrix characteristics, and the effect of variable CNP additions are described from the mechanical and calorimetric points of view. Furthermore, important aspects relating to the design of multimaterial interaction will be discussed. The methods employed in this work are suitable for the fabrication of soft miniactuators for application in different industrial and biomedical fields.


$NiTiCu_6$ optimisation


Matrix functionalisation


Thermal design of multimaterial matrix

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A.10. Magnetic, electric and electronic materials

ID 113 - Advanced waste-based magnetic activated carbon towards the removal of ubiquitous emerging contaminants from wastewater

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Powdered activated carbon (PAC) has proven to be highly efficient in the removal of persistent microcontaminants, such as pharmaceuticals, from water. A generalized incorporation of PAC into conventional wastewater treatment plants – not designed to remove pharmaceuticals – is still limited due to its small particle size, which hampers the recuperation of the spent material from the treated effluent. This work aims at producing magnetic activated carbon (MAC) through microwave pyrolysis and chemical activation of primary sludge from the paper mill industry, with subsequent magnetization using magnetic iron oxides. Hence, a waste-based MAC was produced via an ex-situ synthesis route by simply combining PAC with synthesized magnetic iron oxide particles in a water suspension at a controlled pH. The physicochemical characterization of the produced MAC revealed remarkable properties regarding not only specific surface area (S_{BET} = 795 m^2 g^{-1}), but also saturation magnetization (M_{S} = 19 emu g^{-1}), while the XRD and XPS analyses confirmed the presence of magnetite/maghemite. The antiepileptic carbamazepine (CBZ), the anti-inflammatory ibuprofen (IBU), and the antibiotic sulfamethoxazole (SMX) – listed in the last EU surface water watch-list (Decision 2020/1161) – were used to perform adsorption tests with MAC in wastewater. The very fast kinetics of MAC proved the versatility and broad applicability of this material in the adsorption of pharmaceuticals. The maximum adsorption capacities (qm), determined by the Langmuir adsorption model, were noteworthy: 468 ± 20 µmol g^{-1} for CBZ, 145 ± 10 µmol g^{-1} for SMX, and 273 ± 8 µmol g^{-1} for IBU. These results demonstrated the promising performance of a waste-based ex-situ MAC synthetized by microwave pyrolysis, which is rapidly retrievable from water, as an alternative tertiary wastewater treatment for pharmaceutical removal.

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ID 211 - Crystal growth and electrochemical intercalation of a frustrated magnet α-$RuCl_3$

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Frustrated magnets are quantum materials with localized magnetic moments, whose competing interactions cannot be simultaneously satisfied. α-$RuCl_3$ is a frustrated magnet with a honeycomb structure of Ru^{3+} ($J_{eff} = ½$) magnetic moments. Thanks to the strong spin-orbit coupling, it is close to the realization of a quantum spin liquid ground state as captured by the Kitaev model [1]. In experiment, α-$RuCl_3$ orders antiferromagnetically at 7 K (*ABC* stacking) or 14 K (*AB* stacking). This transition can be suppressed by applying high magnetic fields [2], which signals the possible formation of a quantum spin liquid. This exotic state gains attention thanks to a potential application in quantum computing [3]. We explore chemical routes to tune the magnetic and electronic properties of α-$RuCl_3$, namely to suppress the antiferromagnetic transition.

We conduct electrochemical intercalation of the alkali-metal ions (Li^+, Na^+, K^+) in polycrystalline powders and single crystals of α-$RuCl_3$ in organic and aqueous electrolytes. α-$RuCl_3$ has a layered structure, so intercalation is expected into the van der Waals gaps. Temperature- and field-dependent magnetization measurements are performed on a SQUID magnetometer.

The formation of stable intercalates is evidenced by X-ray and electron diffraction methods and by energy-dispersive X-ray spectroscopy. The crystal structure of K_xRuCl_3 (x=0.5-0.8) is determined via the 3D electron diffraction technique. The crystal structure of $K_{0.5}RuCl_3$ (space group $P\bar{3}m1$, a = 6.058(11) Å, c = 7.057(17) Å, R_1 = 0.1434) is determined on nm-sized domains. Bulk crystals of $K_{0.5}RuCl_3$ undergo an antiferromagnetic transition at ~2.1 K. For Li_xRuCl_3 and Na_xRuCl_3, *operando* x-ray diffraction studies are performed during Li- and Na-intercalation (x=0-0.7). The unit cell parameter c of Li_xRuCl_3 and Na_xRuCl_3 powders is 3 times larger than in pristine $RuCl_3$. Li_xRuCl_3 and Na_xRuCl_3 powders show paramagnetic behavior without any magnetic ordering down to 2 K.

To conclude, we find that alkali metal ions can be intercalated into the van der Waals gaps of α-$RuCl_3$ using an electrochemical route, and this intercalation modifies the crystal structure and the magnetic properties of the α-$RuCl_3$ parent compound.

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B.2. Advanced steels and cast irons

ID 61 - Fatigue Crack Initiation And Propagation Mechanisms In Quenching And Partitioning (Q&P) Treated Stainless Steels

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In the last decade, the development of the quenching and partitioning (Q&P) heat treatment has led to new grades of carbon steels that display a better-than-average balance of strength and ductility [1]. This Q&P treatment is also suitable for certain stainless steels, combining this improved strength-ductility ratio with the inherent corrosion resistance. However, this subject is still to be thoroughly studied given the scarce number of papers in the literature about Q&P processing of stainless steels and even scarcer for their mechanical or functional properties. This work aims to study the fatigue performance of Q&P treated martensitic stainless steel.
Three different stainless steels were subjected to Q&P treatment. Their microstructure was thoroughly analyzed using electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) techniques. Special attention was paid to the volume fraction and morphology of retained austenite. High cycle fatigue tests were carried out, and a fatigue limit was determined for each material. Analysis of fatigue fracture surfaces was performed using scanning electron microscopy (SEM) to identify the mechanisms of fatigue crack initiation and growth. It is shown that the fatigue limit of the Q&P processed stainless steels is similar to their conventional counterparts. The effect of Q&P microstructure on the fatigue behavior of stainless steels is discussed.

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ID 120 - Development of Advanced High Modulus Steels for Automotive Applications

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By introducing a new element or changing the weight percent (wt.%) of an element within steel, improvements can be observed in ultimate tensile strength, a key mechanical property in the automotive industry. Some examples which highlight the use of steels in a traditionally made automobile are car body panels, chassis, engines, and exhaust components. From these examples, it can be determined that steel is an integral structural material and now has the potential to be become lower in density whilst retaining/improving strength (commonly referred to as advanced high strength steels, AHSS). This is achieved by synthesis of metal matrix composites through introduction of ceramic particles such as titanium diboride (TiB_2), niobium diboride (NbB_2) and vanadium diboride (VB_2) into steel matrix. Another aspect to consider when manufacturing steel is the thermomechanical processing route used for grain refinement, as this greatly affects microstructure and hence, mechanical properties. This project aims to conduct research and testing on steel composites via two routes; which are named ‘vacuum-induction melting’ (VIM) and ‘field-assisted sintering technology’ (FAST). VIM is the melting of a metal within a vacuum whilst utilising electric current whereas FAST uses a direct current (DC) coupled with compressive force to consolidate powders together. Both methods will allow the production of steel ingots with different compositions, which can then be put through hot-rolling. From this, mechanical testing and microstructural analysis can be performed and suitable steel-matrix composites will be finalised as for production at an industrial scale. This project acknowledges Volkswagen as the industrial sponsor for this PhD.

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ID 159 - Computational design of alumina-forming martensitic stainless steels

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Alumina-forming austenitic stainless steels (AFA) are designed for applications where corrosion and creep resistance are of paramount importance. However, they suffer from stress corrosion cracking and, due to their FCC crystalline structure, show poor resistance to volume swelling under irradiation. The motivation of our work is to take advantage of the knowledge acquired in the last 40 years of service of heat-resistance ferritic-martensitic steels, combined with the exceptional corrosion resistance properties offered by a protective layer of alumina. For this reason, in this research, alumina-forming martensitic steels have been designed. For the design of these steels, a modified Schaeffler diagram combined with massive thermodynamic calculations using Thermo-Calc© software have been used to estimate the phase balance present in the microstructure. Parameters such as the optimum austenisation temperatures and the stacking fault energy, through the Olson and Cohen model, have been used to maximize the martensite volume fraction in the microstructure. Using this methodology, several alloys in the following compositional range have been designed and subsequently manufactured and characterised: 10 - 17.5 Cr, 8 - 14 Ni, 0.08 - 0.15 C and 3.5 Al (wt.%). The transformation from austenite to martensite has been evaluated using high resolution dilatometry (Figure 1a), while the obtained microstructures have been characterised using X-ray diffraction, optical (Figure 1b) and scanning electron microscopy. The results presented indicate that the design methodology described gives good results.

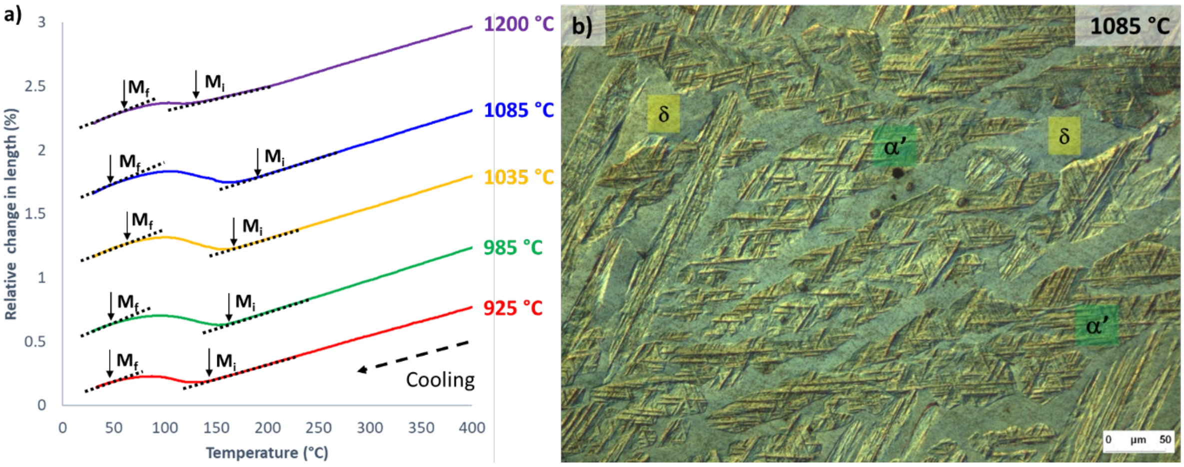

Figure 1. a) Dilatometric curves during cooling at different austenitisation temperatures b) Thermal etching micrograph with an austenitising temperature of 1085 °C

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B.2. Advanced steels and cast irons

ID 164 - Feasibility study on quenching and partitioning treatment on 33MnCrB5 boron steel

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Quenching and partitioning is a heat treatment which has been reported to enhance both mechanical properties and ductility of low carbon steels. The treatment is composed by a quenching between Ms and Mf, and a partitioning step, and it is designed to introduce some retained austenite in the microstructure. This phase is responsible for the increase in the ductility of the material, and moreover, when subjected to a stress, it transforms into martensite, increasing the mechanical properties of the material itself.

In this work, multiple quenching and partitioning treatments were designed and performed on a 33MnCrB5 boron steel to investigate the feasibility of the application of this treatment on the chosen material. Different quenching and partitioning temperatures (evaluated through Thermocalc simulations) and times were tested to investigate their effect on the final properties of the material. The heat treated material was characterized both regarding the microstructure, and the mechanical properties. Specifically, SEM and EBSD were used to observe the generated microstructure, XRD analyses were conducted to evaluate the presence and the amount of retained austenite, while tensile tests were performed to investigate the induced mechanical properties. The designed quenching and partitioning treatments were reported to introduce retained austenite in the final microstructure, and enhance both the yield strength, the ultimate tensile strength and the elongation at break with respect to a conventional quenching and tempering treatment. Therefore, it can be concluded that the suggested treatment appears promising for the material selected.

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B.3. High-performance alloys and intermetallics

ID 57 - The influence of molybdenum content on phase transformations in binary Ti-Mo alloys

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Aerospace industry, medical implants, sports equipment or jewellery: all these fields make use of titanium alloys. Titanium alloys are materials with varied properties, thanks to a wide range of possible microstructures which can be achieved by additions of alloying elements and by thermo-mechanical treatment.

Titanium is an allotropic element, as it crystalizes in several crystallographic structures, depending on temperature. The low temperature structure, called α phase, is HCP, and the high temperature structure, β phase, is BCC. When certain elements (Mo, Fe, V,…) are added to titanium, the β phase can be retained at room temperature after fast cooling. These alloys are called metastable β-titanium alloys. Several metastable phases can be observed in β-titanium alloys; the one important for this study is the ω phase with a hexagonal structure.

We studied the effect of molybdenum content on phase transformations in β-titanium alloys. Three alloy compositions (Ti-12Mo, Ti-15Mo, Ti-18Mo, in wt.%) and one functionally graded alloy Ti-xMo, where x ∈ <9, 12, 15, 18, 21> wt.% Mo, were investigated. We utilized in-situ methods during linear heating: dilatometry, differential scanning calorimetry (DSC), resistometry and ex-situ methods after linear heating or isothermal annealing: SEM and microhardness measurement. Each of these methods is sensitive to different features and processes, but being complementary they provide comprehensive information about phase transformations and microstructure changes.

β-stabilizing element, molybdenum, supresses both the α phase precipitation and the ω phase formation. Higher molybdenum content causes the temperature interval of ω phase evolution to shrink, leading to lower microhardness.

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ID 86 - An in-situ study of grain boundary migration and sliding in AZ31 magnesium at elevated temperatures

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While it is known that grain boundaries play an important role in accommodating plastic deformation in magnesium (Mg) alloys at elevated temperatures, our understanding of the process by which they do so is limited. The accommodation of deformation incompatibility in an AZ31 Mg alloy at 200 °C has been captured at the sub-grain scale by in situ high resolution digital image correlation (HRDIC) within a scanning electron microscope. Complementary electron backscattered diffraction (EBSD) revealed the grain boundaries before and after deformation. The migration and sliding of these grain boundaries has been qualitatively and quantitatively revealed by analyzing the displacement fields at several incremental strain steps up to 50% plastic strain. Other plastic deformation mechanisms, including dislocation slip, grain rotation and extension have also been captured. Grain boundary movement led to the formation, annihilation and break up of grains, contributing to the superplastic behaviour of the alloy at temperature. Further, the active deformation mechanisms (e.g., slip bands and grain rotation) varied significantly depending on the initial grain orientation, size, and morphology, as confirmed using the post deformation EBSD map. For instance, slip band formation was dominant for the grains well aligned for <a> type basal slip, contributing to the grain boundary movement. The 3D surface topographies of the deformed samples showed significant surface textures, revealing significant out of plane deformation. The present study shed light on the role of grain boundary movement on the superplastic behaviour of AZ31 Mg alloy during incremental straining.

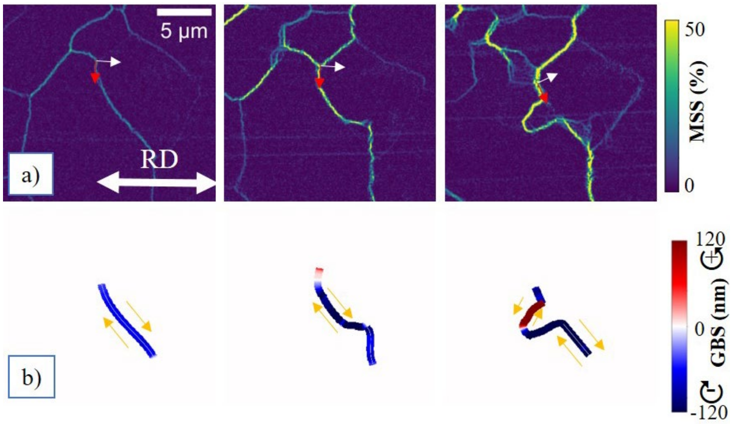

Fig.1 (a) Incremental maximum shear stress (MSS) maps between 0.55-1.95%, 4.81-6.39%, and 10.77-12.62% strain and (b) their corresponding GBS quantification for a single grain boundary, respectively. The loading direction is parallel to the rolling direction (RD)

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ID 90 - Microstructure and mechanical properties of zirconium alloys

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Zirconium alloys generally possess high strength, ductility and corrosion resistance. Furthermore, Zr has a very low absorption cross-section for thermal neutrons, which makes solute-lean zirconium alloys particularly useful in nuclear technology. Pure Zr is found in two crystallographic modifications: the low-temperature alpha phase (hexagonal close-packed) and the high-temperature beta phase (body-centered cubic) with the transition temperature of 864 °C. In Zr alloys, the transition temperature and the range of thermodynamic stability of the two phases can be modified depending on the type and amount of alloying elements. Elements that increase the transition temperature are called alpha stabilizers, these are usually non-transition metals or interstitial elements. On the other hand, elements that decrease the transition temperature are called beta stabilizers and usually belong to transition metals.

In this work, we designed several Zr alloys with a higher content of alloying elements (Sn, Nb, Al, Cr and Fe) with the aim to enhance their mechanical properties. These materials belong to the so-called alpha+beta Zr alloys, which are composed of a mixture of alpha and beta phase at room temperature. This work focuses on studying phase transformations, microstructure evolution and the resulting mechanical properties of the new Zr alloys.

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ID 112 - The role of boron on the microstructure and properties of a Ni-Si-B cast alloy

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Ni-Si-B alloys are used in many applications for aerospace, aeronautical, nuclear, chemical, automotive, as well as tools and mould industries, due to their high resistance to wear, impact, corrosion and oxidation at high temperatures. Typically, the microstructure of Ni-Si-B alloys processed by casting is composed of austenite and hard phases such as borides (Ni_3B) and silicides (Ni_3Si). The high-temperature properties are achieved with the proper balance of hard phases and austenite, ensuring a good compromise between toughness and hardness. However, there is a large microstructural variation due to chemical composition fluctuations among the melts, and variations in the cooling rate imposed by the wall thickness of the cast components. The aim of this work was therefore to study the boron influence on the microstructure and hardness of a Ni-Si-B cast alloy. Specimens with different contents of boron were fabricated by the lost-wax casting process and characterized by differential scanning calorimetry coupled with thermogravimetric analysis (DSC-TGA), scanning electron microscopy (SEM-BSE) with energy-dispersive X-ray spectroscopy microanalysis (EDS), X-ray diffraction (XRD) and hardness tests. Furthermore, the interpretation of experimental results on the kinetics of phases formation under casting conditions was compared and complemented with thermodynamic simulations using the Thermo-Calc software. The results reveal that there is an increase in the percentage of nickel borides (Ni_3B) as the boron content increases, which is accompanied by a considerable increase in the hardness of the alloy.

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B.3. High-performance alloys and intermetallics

ID 149 - Characterisation of Zirconium alloys with Copper and Vanadium additions for nuclear fuel assembly materials

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Zirconium alloys are utilised in nuclear reactors as fuel assembly materials due to their beneficial material properties and low neutron cross sections (1). Zirconium alloys do have limitations – the environment of nuclear reactors affect their material properties, degrading the cladding (2). Developing alloys that show improved resistance to degradation effects can improve in-reactor efficiency. Recently, Westinghouse Electric AB developed Zirconium alloys with Copper and Vanadium additions, which showed improved performance compared to previous cladding generations (3).

The aim of this PhD project is to understand Copper and Vanadium's formation in Zirconium and the roles of these elements during irradiation. Copper and Vanadium are uncommon alloying additions in Zirconium, consequently there are gaps in the mechanistic understanding of their roles. As such, this project is focused on investigating new alloy compositions that have not been experimented on in detail before now, the W10 (Zr-1Nb-0.3Sn-0.2V-0.05Fe) and W13 (Zr-1Nb-0.3Sn-0.2Cu-0.05Fe) alloys developed by Westinghouse Electric AB, which were neutron irradiated in the BOR-60 research reactor and will be proton irradiated at the Dalton Cumbria Facility.

Characterisation of the unirradiated alloys is important to understand how Copper and Vanadium influence the microstructure of the alloys, such as their precipitate compositions, and to act as a reference for microstructural evolution when examining the irradiated samples. A range of techniques will be employed to examine grain morphology and orientation, material texture, the dispersion of elements, precipitate structure and the dislocation structure of these materials, including XRD, SEM and TEM, the findings of which will be presented at the conference.

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ID193 - Response to Different Artificial Aging Conditions of the As-built and Rapid Solubilized AlSi10Mg Alloy Produced by Laser-Based Powder Bed Fusion

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The Laser-based Powder Bed Fusion (L-PBF) technology meets the growing market demand for high-performance and custom-made components. In this regard, the AlSi10Mg alloy matches both the technological requirements (excellent thermal conductivity, fluidity, and weldability) and, after adequate heat treatment, the mechanical requirements (excellent strength/weight ratio, good fatigue resistance, and impact toughness) necessary in the production of high-tech mechanical parts.

However, the improvement of the tribological behavior of the L-PBF AlSi10Mg alloy necessarily requires the deposition of a multilayer coating system, in which, for example, diamond-like carbon (DLC) by physical vapor deposition (PVD) can form the top-coating. Prolonged exposure (2 ÷ 6 hours) of the components to temperatures between 160 °C and 180 °C, therefore, requires an appropriate study to evaluate the effect of thermal exposure on the mechanical behavior of the alloy.

Against this background, the present work is aimed at studying the effect of different thermal exposure conditions, simulating the DLC deposition treatment (4 ÷ 6 hours at 160 °C and 2 ÷ 4 hours at 180 °C), on the tensile properties of the alloy in the as-built condition and after short solubilization (10 minutes at 510 °C).

The results showed that the mechanical properties of the L-PBF AlSi10Mg alloy heat treated at the same temperature and time conditions of the deposition treatment were comparable to those of the T5 and T6 heat-treated alloys reported in the literature. Hence, a suitably optimized PVD process could replace the aging phase in both heat treatments, reducing costs and production times.

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ID 49 - The effect of vanadium on the high-temperature oxidation of complex concentrated alloys FeAlCrNixVy

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An ongoing call for materials capable of operating at high temperatures has led to the development of numerous structural alloys such as Ni-based superalloys or austenitic steels. In recent years, much hope was also put into Complex concentrated alloys – alloys consisting of multiple elements in near-equiatomic ratios. Their systematic research is complicated by the vastness of their compositions and problematic properties predictions based on element content. Our department developed equimolar alloy FeAlCrV possessing promising mechanical properties up to 800 °C. However, its oxidation resistance is unsatisfactory, probably due to the high vanadium content. Therefore, we focused on the possible improvement of this alloy and further characterization of its oxidation behavior. A series of $\mathrm{FeAlCrNixV_y}$ was made to examine in detail the effect of vanadium which seems to play a crucial role in the kinetics of the oxidation. Further characterization of the oxide scale was performed and compared with the theoretical model.

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ID 71 - Cu-Mn cosegregation in $MnCrFe_2Ni_2$ base HEAs produced by different manufacturing routes

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The segregation behavior of MnCrFe2Ni2 High Entropy Alloys (HEAs) with different additions of Cu and Mo is analyzed in this work. The replacement of Co in many HEAs due to its elevated cost and scarcity motivates the use of other FCC-stabilizers, as Mn and Cu. To analyze the possible elemental segregation, both as-cast and additive manufacturing processing routes are considered.

The samples produced by casting+hot-forging (C+HF) where later heat treated above 1100 ºC and showed a strong co-segregation of Cu-Mn at the grain boundaries (see Figure 1(a)-(b)). The formation of Cr/Mo-rich phases have also been observed above 700 ºC for alloys with high content of Mo. These phases and segregation would deteriorate the mechanical behavior of these alloys and, thus, they should be minimized or avoided. The most promising composition has been gas-atomized and processed by means of LPBF (Laser Powder Bed Fusion) using different printing conditions. The microstructural characterization show that crack formation has been difficult to elude in as-printed condition and generally associated to grain boundary co-segregation of Cu-Mn (see Figure 1(c)-(d)). Even though the segregation of Cu-Mn in this HEA family might be unavoidable, with the corresponding detriment in mechanical properties, it is worth exploring further the relationship between crack formation and segregation observed in this type of HEAs during this investigation.

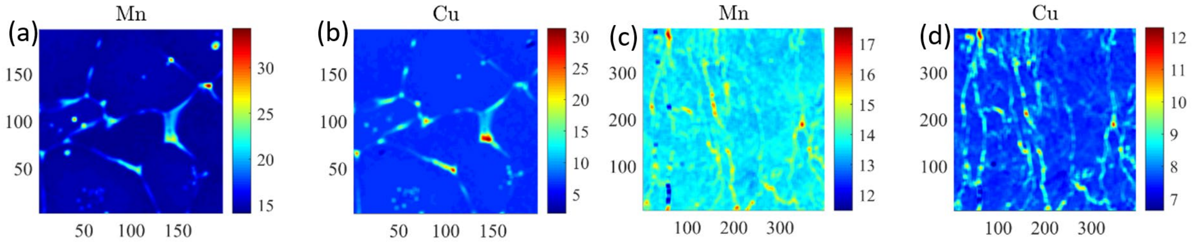

Figure 1. EMPA analysis. (a)-(b) C+HF sample (1100 ºC heat treatment); Cu-Mn co-segregation at the grain boundary (c)-(d) LPBF as-printed samples; Crack formation at Cu-Mn segregated grain boundaries.

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B.4. High entropy alloys

ID 94 - Improving the oxidation resistance of high-entropy alloys via addition of reactive elements

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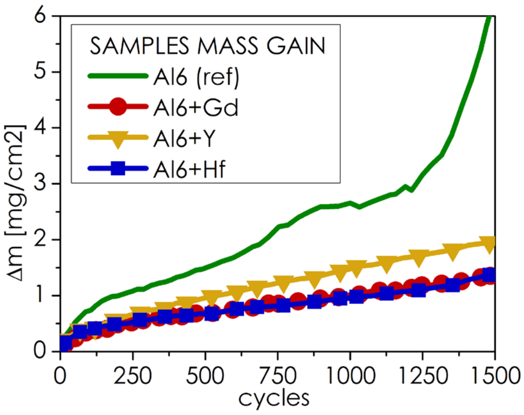
High-entropy alloys (HEAs) are among the most promising materials for high-temperature structural applications. Unfortunately, their high-temperature corrosion resistance, especially under thermal cyclic conditions, remains worse than the state-of-the-art heat-resistant materials, making the search for a feasible solution to this problem of special importance.

In the presented study, the application of reactive elements effect (REE) is shown to drastically improve the oxidation behavior of HEAs. The selected chromia-forming $Al_6(CoCrFeNi)_{91}$ high-entropy alloy is investigated under thermal cyclic conditions at 900°C in air. In order to examine the influence of different reactive elements, the samples are surface-modified with thin, discontinuous layers of Gd_2O_3, Y_2O_3, and HfO_2, respectively. The oxidation kinetics are determined over 1500 cycles, each of 1 h duration, followed by thermal shock and a 20-min cooling period. The post-exposure analyses including both surface and cross-section studies are carried out with the use of XRD, SEM-EDS, Raman spectroscopy, and EPMA mapping techniques.



The results indicate a spectacular improvement in oxidation behavior after the addition of Gd, Y, and Hf, with almost total suppression of scale spallation, much slower overall oxidation kinetics, and much simpler scale structure when compared with the reference, unmodified sample. As a consequence, it can be stated that the effect of reactive elements can be a very effective solution leading to the improvement of the oxidation behavior of typical high-entropy alloys, and its incorporation may allow for obtaining of next-generation of high-temperature-dedicated HEAs.

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ID 103 - Solid solution strengthening in single-phase HEAs based on Au-Cu-Ni-Pd-Pt

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High entropy alloys possess a good combination of high strength and ductility. It is widely accepted that the solid solution strengthening is the dominant strengthening mechanism due to the large amount of different elements. In contrast to dilute alloys, solid solution strengthening of concentrated multicomponent alloys is not well understood yet. To enable usability by engineers, it is necessary to develop reliable models for the prediction of the mechanical properties. To verify such models experimental work is needed that illustrates the concentration-dependent solid solution strengthening of different alloys over a preferably large composition range.

The high entropy alloy system Au-Cu-Ni-Pd-Pt is unique as it crystallizes in face-centered cubic crystal structure and is single-phase presumably within the entire concentration range. Hence, it serves a suitable benchmark system to investigate composition-dependent effects on the strengthening mechanisms. In this study, it has been examined whether the model of Varvenne [1] is suitable to predict the solid solution strengthening in this alloy system. For that purpose, the concentrations of individual elements were successively varied and the strength and hardness have been obtained by compression tests and microhardness measurements. The results give insights about the impact of different types of elements on the strength and can help to further develop strengthening models. The theory of Varvenne is misguiding the development of the strength in a large compositional range.

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ID 146 - The occurrence of atypical serrated flow in entropy alloys

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Entropy metallic alloys consisting of two to four principal elements (medium-entropy alloys) or more than four principal elements (high- entropy alloys), as first proposed in 2000s, have recently attracted an extensive attention of materials scientists. This contribution presents a systematic study of serrated flow of the FeAlCrMo and FeAlCrV medium-entropy alloys in compression, first observed at 400 °C and a strain rate of 10-4 s-1. These parameters were further varied in order to evaluate how they affect the deformation behavior. Compression curves and strain rate jump tests suggest that dynamic strain aging (i.e. the Portevin-Le Châtelier (PLC)) effect is responsible for the jerky flow. During compression, three most common types of the PLC effect are observed at different conditions. Results of the conventional compression tests are also correlated with the concurrently recorded acoustic emission (AE) data. The AE data suggest that the serrations most likely result from a sequence of dislocation avalanches rather than from individual ones with very high magnitude. Furthermore, at certain conditions, peculiar periodic serrations appeared throughout plastic loading, which possessed bimodal distribution of drop magnitudes. In this multicomponent material, this effect likely originates in complex dislocation-solutes interactions at the atomic scale.

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ID 147 - Study of Casting and Selected Thermal Processes in a Copper Based Multicomponent Alloy

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Copper-zinc alloys, widely known as brass alloys, are among the most important categories of copper-based alloys, due to their extensive field of application and range of properties. In recent years, the emerging field of high-entropy alloys (HEAs) has paved the way towards the discovery of new materials through traditionally unconventional means of alloy design. Such design principles could be employed towards the development of novel brass compositions aiming to achieve enhcanced mechanical and physical properties. This work concerns the design, casting and heat-treatment process of a novel CuAlZnSn Multicomponent Alloy (MCA),utilizing alloying elements that are used as base components in commercial brass and bronze alloys. The investigated material was macroscopically etched to reveal the solidification zones. Microhardness measurements were conducted in the as-cast condition, to reveal the effects of segregation on the mechanical properties along the cast, and heat-treated conditions to pinpoint changes in the mechanical properties during the progression of the heat-treatment. In addition, various heat-treatments were conducted for 8, 16 and 24 hours at 550°C, followed by different cooling paths. The aim was to investigate the formation and stability of the phases present in the as-cast microstructure. Both as-cast and heat-treated samples were analyzed through means of Optical (OM) and Scanning Electron Microscopy (SEM) to unveil the various microstructural features and highlight possible segregation phenomena. Alterations in the cooling rate would either suppress or promote the formation of the occurring phases resulting in changes in their morphology and volume fraction. Through this analysis a primary $Cu_{55}Zn_{25}Al_{20}$ at. % phase could be identified, which shows high thermodynamic stability regardless of the applied heat-treatments and is characterised by an enhanced hardness compared to conventional brass alloys.

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ID 183 - Microstructure and mechanical properties of light-weight refractory high-entropy alloys fabricated by powder metallurgy

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High-entropy alloys correspond to an emerging field of metallic materials, composed of five or more elements in near-to-equiatomic proportions. Among them, refractory high-entropy alloys (RHEAS) have exhibited interesting mechanical properties at high-temperature as well as excellent oxidation resistance. In this study, the microstructure and mechanical properties of a series of novel Al-Cr-Nb-Mo-Ti-V-Fe RHEAs prepared using a powder metallurgy approach was studied. Elemental powders were mechanical alloyed for 50 h in a planetary ball mill under argon atmosphere. Then, the milled powder was sintered by spark plasma sintering. Dilatometric studies indicated that at 1150 °C for 10 min and 50 MPa under vacuum, sample achieved the maximum density. Dense disks sintered under these conditions were then subjected to a secondary heat treatment at 1350 °C for 16 h. The SEM-EDS analysis of the heat-treated sample indicated a matrix with the presence of Mo,V-rich and Nb,Fe-rich phases , while Al_2O_3 and Ti,C-rich particles were observed as secondary phases. The XRD pattern suggests the presence of a main bcc phase with lattice parameter of 3.06 Å. Additionally, peaks associated with TiC and Al_2O_3 also confirmed their presence. In terms of mechanical properties, the heat-treated sample exhibited a hardness of 1007 HV0.3 and a compressive fracture stress of 2234 MPa. At the same time, the as-sintered sample exhibited a higher hardness value, corresponding to 1124 HV0.3, but a lower compressive fracture stress of 1712 MPa; notwithstanding, none of the samples exhibited plastic deformation.

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B.5. Advanced ceramics

ID 92 - Hydrothermal Ageing Behavior of Bioinspired Material with Piezoelectric Functions for Implant Applications

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Implant-bone fixation loss can occur 10-15 years after surgery. This is related to bone resorption and consecutive biofilm formation in the generated gap, which could lead to tissues inflammation and implant loss [1]. Current solutions are focused on primary osseointegration for implant early survival rate. In long term, they lose effectiveness due to implant foreign body reaction once bone metabolism is challenged by a non-part of the body. Bone is composed of piezoelectric materials with production of biological electricity due to collagen deformation and displacement of the local electric field [2]. Barium titanate is a lead-free piezoelectric bioceramic without toxicological risk and absence of foreign body reactions that can mimic natural bone piezoelectricity and inhibit bacterial adhesion [3]. The focus of this work is design, produce, and characterize a smart, bio-inspired, and multi-functional composite material to improve implant long-term bone regeneration and antibacterial effect. In this sense, a composite with BaTiO3 particles and bioinert ZrO2 was produced. 5%BaTiO3-ZrO2 composites were mixed in isopropanol alcohol and ultrasonicated for total dispersion, followed by press (200MPa) and sintering technic (1300-1500ºC). The different samples were characterized with SEM (Fig1.(a)), that present two distinct phases, XRD analysis, and subjected to hydrothermal ageing (Fig1.(b)), to evaluate the stability of the tetragonal zirconia phase. After 5h of accelerated ageing (≈10 years), monoclinic phase is more evident with the sintering temperature increment, but always lower than 25%, staying in according with ISO 13356:200, promising to be promising a potential replacement material for implant applications.

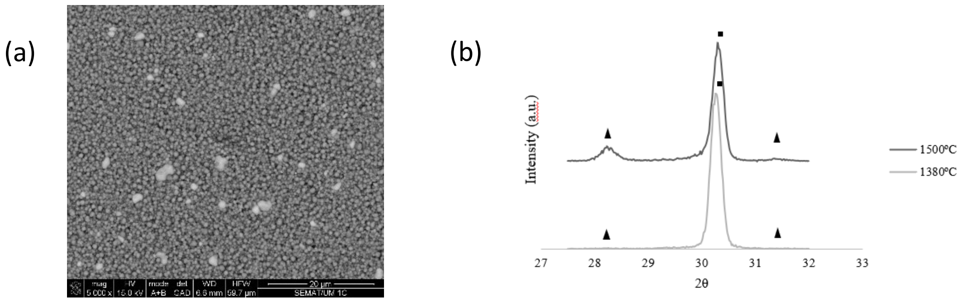

Figure 2 (a) Top view recorded on as sintered 5wt% BaTiO3 – ZrO2 composite sintered at 1500ºC; (b) XRD X-ray diffractograms spectra for the composites sintered at 1380ºC (1.7% monoclinic phase) and 1500ºC after 6h hydrothermal ageing with monoclinic phase percentage of 1.7 and 14.3, respectively.

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B.6. Carbon based materials

ID 68 - Sustainable process to produce waste-based activated carbon by microwave pyrolysis for the removal of antibiotics from water

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Biomass wastes from brewery industry are produced in large amounts is a valid option to be used as precursors of carbon adsorbents for further application in water treatment systems. Adsorption by activated carbon (AC) has been proven to be an effective technology for the removal of antibiotics from water. The utilization of industrial wastes as AC precursors contributes to the environmental sustainability of this process, avoiding the use of non-renewable raw materials. Typically, conventional pyrolysis has been applied to produce AC from biomass wastes, however, recently, microwave-assisted pyrolysis has attracted considerable attention due to the lower energy consumption, faster heating rate and higher production yields. The present study aimed to produce AC from brewery wastes by microwave pyrolysis to be used for the removal of the antibiotics sulfamethoxazole (SMX), trimethoprim (TMP) and ciprofloxacin (CIP) from water. To optimize the AC production process, a fractional factorial design was applied. Three factors were studied at three levels: activating agent:precursor ratio (1:1, 1:2, 1:5 w:w), temperature of pyrolysis (600, 700 and 800 ºC) and residence time (10, 20 and 30 min). A fourth factor was also evaluated at two levels: activating agent (KOH and K2CO3). Eighteen different AC were produced, and the effect of the assessed production parameters on the SBET, yield of production (%), total organic carbon and adsorptive removal (%) of SMX, TMP and CIP was evaluated. One AC was selected, further characterized and its performance evaluated by kinetic and equilibrium adsorption studies from ultrapure and wastewater systems. The obtained results demonstrate the adequacy of brewery wastes as precursor of highly efficient carbon adsorbents synthetized by microwave pyrolysis, so minimizing the amount of activating agent and the pyrolysis time (several minutes instead of several hours in conventional furnaces) with the consequent lower energetic cost.

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B.7. Composites and nanocomposites

ID 14 - Characterisation of Advanced Metal Matrix Composite Materials for Application to High Energy Physics Detectors

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The Outer Tracker of the Compact Muon Solenoid (CMS), one of the large experiments at the CERN Large Hadron Collider, will consist of about 13200 modules, each built up of two silicon sensors. The modules and support structures include thousands of parts that contribute to position and cool the sensors during operation at -30°C. These parts should be low-mass while featuring high thermal conductivity, stiffness and strength. Their thermal expansion coefficient should match that of silicon to avoid deformations during cooling cycles.

Due to their unique thermal and mechanical properties, aluminium-carbon fibre (Al/CF) Metal Matrix Composites are the material of choice to produce such light and stable thermal management components for High Energy Physics detectors. For the CMS Outer Tracker, about 50000 cm^3 of Al/CF raw material will be required, to be produced through a reliable process to guarantee consistent properties all along part manufacturing.

Two Al/CF production routes are currently considered: liquid casting and a powder metallurgy process based on continuous semi-liquid phase sintering. Dimensional stability of the resulting material is of paramount importance. Irreversible change of shape may be induced by moisture adsorption and onset of galvanic corrosion at the discontinuous interfaces between CF and Al.

This paper presents the results of an extensive investigation through Computed Microtomography, direct microscopical investigations, electrochemical studies of the interface reactivity and metrology measurements, aimed at comparing and interpreting the response to different environments of the respective products. The effect of the application of noble metal coatings is also discussed.

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ID 17 - Combining non-linearities to affect the damping behaviour of fibre-reinforced polymer composites

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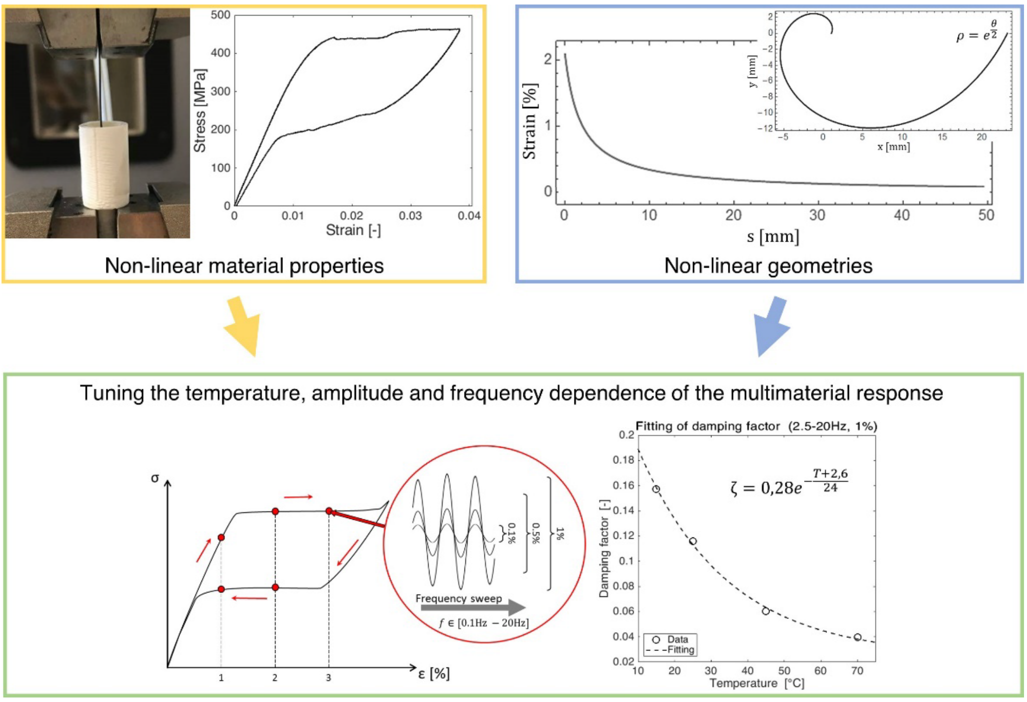
In several state-of-the-art applications, shape memory alloy (SMA) wires have been successfully added to polymer composite materials in order to improve the damping response, impact resistance and crack closure [1,2]. The tuneable non-linear, pseudoelastic behaviour of the alloy is often exploited for these aims, yet the use of non-linear geometries of the wires remains unexplored. A number of examples in literature present fibres with non-linear geometries used in composites such as Variable Stiffness laminates to optimise material properties like stress distribution, buckling and frequency response [3].

In this work, we present a multimaterial construct, fabricated with non-linear wires of pseudoelastc NiTi embedded in an epoxy matrix. This construct combines material and geometrical non-linearities in order to affect its viscoelastic behavior (Fig. 1). In particular, we discuss how to tune the damping response of SMA wire [4], and characterise the effects that non-linear geometries, combined with this tuning, have on the overall damping factor of the multimaterial.



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ID 75 - Luminescent YAG: Er microspheres used for preparation of phosphor in glass materials

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The work deals with the preparation of luminescent YAG glass microspheres doped with Er^{3+} and embedded in bulk yttrium aluminum silicate (YAS) $17Y_2O_3$-$19AL_2O_3$-$64SiO_2$ by viscous flow sintering.
First, luminescent YAG powder precursors were prepared by sol-gel Pechini method. As a source of erbium ions, Er_2O_3 was added in following concentrations: 0.25; 0.5 and 2.2 mol. %. Flame synthesis was used for spheroidisation of powder precursors. Amorphous nature of prepared of microspheres was confirmed by X-Ray diffraction (XRD) analysis.
Yttrium aluminum silicate prepared by melting and quenching technique was crushed and sieved through 40 µm mesh sieve and this brash was used to embed luminescent microspheres. Densification of samples was provided by viscous flow sintering in hot press at temperature 900°C.
Morphology of materials was investigated by Scanning electron microscope (SEM).
Luminescent properties of all materials were investigated.

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ID 101 - High-performance multifaceted materials: structural approaches for Li-ion batteries

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The worldwide development depends on a key factor: energy. In this context, energy storage systems (ESS) are seen as crucial factor [1]. At the end of 20th century, and due to the emerging market for wireless technologies, the demand for rechargeable batteries has increased. Along with the increase of scientific research in this area, it was possible to ally the electrical storage potential of batteries with the mechanical resistance obtained by polymer-based composites, creating a novel class of multifunctional materials known as structural power composites [2]. In this field, lithium-ion (Li-ion) structural batteries are faced as one of the most promising chemistries, delivering high-energy and high-power density. Common drawbacks of this technology are related with dendrite and irregular solid-electrolyte interface (SEI) formation, unregular ion transport and parasitic reactions [3]. Since the interface engineering is crucial to the safety of these devices, we report preliminary studies on the functionalization of pitch- and polyacrylonitrile (PAN)-based carbon-fibres (CFs) with nanomaterials with the intuit of optimization of the electrode//electrolyte interface. A hybrid CFs system was prepared and used to produce multifunctional and multiscale composites. A laminated structure was developed and tested as positive electrode against Li metal anode (in a coin-cell assembly), to understand if this type of electrode is able to intercalate Li ions. The final assessment of this study is to apply the functionalized fibres in a structural battery.

Keywords: Li-ion, structural batteries, multifunctional materials

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ID 150 - Deformation behavior of CNT reinforced metal matrix nanocomposites

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The interest in advanced lightweight materials, able to achieve high mechanical properties, such as reinforced metal matrix nanocomposites (MMNCs), has recently increased mainly for the transport industry. The nanoscale reinforcements showed to be significantly more efficient than the micrometric in reinforcing these types of matrices. The carbon nanotubes (CNTs) are one of the most promising reinforcements to produce lightweight nanocomposites with matrices such as aluminium due to their unique structure and properties [1]. So far, few works have addressed the influence that CNTs can have on the deformation behaviour and strain hardening of MMNCs, a crucial issue for their processing and possible service application [2,3]. In that sense, the main objective of the present work is to understand the microstructural changes induced by deformation on MMNCs and how the presence of CNTs can affect them. MMNCs reinforced with 1.0% vol. of CNTs were produced by powder metallurgy route, dispersed/mixed in a single step, cold-pressed, and sintered under vacuum. Pure metals and respective nanocomposites were then cold-rolled at different rates to understand the deformation behaviour of the MMNCs. Microstructural analysis was carried on through advanced microstructural characterization techniques such as scanning electron microscopy (SEM), focusing on electron backscatter diffraction (EBSD), transmission electron microscopy (TEM), and high-resolution TEM (HRTEM). The results show that the presence of CNTs can have a significant impact on the deformation behaviour of the metallic matrix, especially when lower deformation rates are applied [4].

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B.8. Fatigue wear and corrosion

ID 78 - Solid particle erosion resistance of a high-chromium Fe-Cr-C hardfacing alloy: the role of microstructure and particle size

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High chromium cast irons (HCCIs) are broadly employed in high-demanding applications thanks to their excellent abrasive resistance, which stems from their peculiar microstructure, made up of dispersed primary hard carbides in the surrounding soft matrix. The progressive surface material removal due to erosion phenomena affects the performance, reliability and service life of many engineering components. Many efforts and research have been devoted to studying and enhancing the erosion resistance of materials, considering the main influencing factors related to the properties of erodent particles and substrate. So far, however, the influence of microstructure and particle size has not been closely investigated. Hence, this work seeks to analyze the erosive wear resistance of a HCCI tested with Arizona dust quartz as the erodent with two different mean diameters of the particles. Experiments were performed in a purpose-built air blast test rig based on the ASTM G76 standard. The preliminary optical and scanning electron microscopic investigations enable the evaluation of the metallographic phases, which were then related to the particles' diameters. The tribological performance was assessed through weight loss measurements and microstructural analyses of the worn surfaces to describe the role of both carbides and matrix characteristics in the wear damage. The experimental findings suggest a strong correlation between the microstructural features and the erosion resistance of the alloy. Particle size is a critical parameter that affects the resistance, as regards both the mass loss from the surface for the unit of mass of erodent and the wear mechanisms of material removal.

ID 105 - Examining the role of environmental factors and grain orientation in fatigue life of aluminum alloy 7075

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Nanofluids are solid-liquid mixtures composed of solid nanofibers or nanoparticles, that suspend in a fluid with sizes that range from 1 to 100 nm.

Their applications span from cooling engines and electronics, heat exchange in solar collectors, heat storage systems and coolants in manufacturing. The use of nanofluids has been proved to offer a positive effect on the corrosion resistance in some cases. Aluminum is a light material that has remarkable fatigue resistance even in harsh environmental conditions such as air, water, and sea, as well as chemical resistance.

The aim of this study was to evaluate the fatigue performance of a cold-rolled AA 7075-T6 plate both in normal and corrosive environments such as Cu nanofluid. A three-point bending fatigue device was specially designed and manufactured for this case. The tests were performed at frequencies of 25 Hz, R=1 and ambient temperature. Fatigue Wohler-like curves along the long transverse (T), short-transverse (S) and rolling (L) directions of the plate were extracted. Also, 10^6 cycles were considered as the fatigue life limit (endurance limit).

The results were interpreted against the forming direction and revealed significant differences in fatigue and corrosion-fatigue life. The nanofluid impact on aluminum fatigue life was evaluated as a parameter with high potential which deserves further investigation.

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C.1. Coatings and surface nanomodification

ID 4 - Scale Development and Inhibition on Sugar Cane Heat Exchangers

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Brazil is one of the top producers of sugar for the global sugar production.Recently, Brazil has increased its production of ethanol from sugar cane and BP-Bunge Brazil contributes by producing over 1.5 billion litres of ethanol. The process to produce ethanol and sugar is to first break down the sugar cane which is then clarified using addition of lime. The addition of lime increases the concentration of calcium ions that are present in the sugar juice produced. When the juice is heated in the evaporator system the calcium ions form scales on the surfaces. This reduces the heat transfer coefficient of the steel walls, efficiency of the evaporator system and the rate of ethanol and sugar production. A scale inhibitor applied to the surface of the steel to reduce or prevent scaling is envisioned for this project. The preliminary study is to develop a film of calcium phosphate scaling on stainless steel 304L in order to develop coatings to prevent this film from being developed. By creating a worst-case scenario of scaling, tailored coatings of polydopamine with super hydrophobic silica nanoparticles is investigated in regard to: its strength of binding to the stainless steel 304 L surface, the prevention/reduction of nucleation sites on the 304 L surface and accumulation of scale onto the coating. This will be compared to coatings of polydimethylsiloxane with epoxy resin and super hydrophobic silica nanoparticles further along in the research.

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ID 16 - Incorporation of carbon nanotubes in alumina layers grown by plasma electrolytic oxidation

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Plasma electrolytic oxidation (PEO) is a plasma-assisted electrochemical surface treatment particularly suited to converting aluminum into a protective alumina layer. PEO differs significantly from acid anodizing processes since it is conducted under higher voltages/currents and oxidation continues because of the apparition of micro-discharges issued from the electric breakdown of the growing oxide layer1.

Few studies investigated the possibility to conduct PEO on aluminum in an electrolyte wherein carbon nanotubes (CNTs) were dispersed2. Although the tribological properties and corrosion behavior of the formed oxide layers were evaluated, little efforts went into characterizing the incorporation of CNTs within the alumina layer. It can be explained by the fact that the porous nature of the PEO alumina, combined with limitations regarding reliably detecting carbon, pose major obstacles to finely characterize this incorporation.

In the present work, complementary characterization techniques such as high resolution SEM, Raman spectroscopy and glow discharge optical emission spectroscopy, definitely show the presence of CNTs within the PEO alumina layer. Moreover, the effect of the concentration of CNTs dispersed in the electrolyte on the formation of the PEO oxide layer was also investigated. The potential degradation/alteration of the incorporated CNTs was also evaluated (and constitutes an additional concern throughout the process). Finally, the collected results allow proposing a mechanism for the incorporation of CNTs in alumina layer during PEO of aluminum.

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ID 30 - Ion-exchanged borosilicate glass vials for pharmaceutical packaging

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Abstract: Pharmaceutical containers for parenterals like vials, ampoules, prefilled syringes and cartridges have been predominantly manufactured using glass as a packaging material of choice, especially type Type-I glass or borosilicate glass [1, 2]. Borosilicate glass is worldwide use at this scope, but it may have some issues related to breakage, corrosion and delamination that might compromise the drug quality, safety and efficacy [3]. Various techniques have been proposed to improve the mechanical properties of glass containers surfaces like chemical ion-exchange strengthening, during the ion-exchange process, small alkali ions, such as lithium and sodium, representing typical “modifiers” in the glass, are replaced by the larger potassium ions in a molten salt, where the glass is held for a certain time. The aim of this work is to develop innovative or modified inner glass vials surfaces and incorporation of antbacterial element into the surface by ion-exchange treatment by using different alkali molten salts to control the interactions between the surfaces of glass vials and the recently developed drugs and to improve the mechanical, chemical durability and corrosion resistance properties of glass vials in order to respond to the demand of packaging of new drugs. Many analytical techniques will be used to characterize the inner surface of different type I glass vials received from Nuova OMPI company, Italy. All vials were investigated topographically by Scanning Electron Microscopy (SEM). And the glass composition of the surface was studied by energy dispersion X-ray spectroscopy (EDS). The hydrophobicity/hydrophilicity of the inner surface was assessed by dye tests and measuring of contact angle [4].

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ID 33 - Stainless steels patterning by selective etching in ICP chlorine-based plasmas

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Surface structuring is a huge thematic of research to answer problematics such as friction reduction or wettability management. This work is dedicated to the stainless steels patterning by dry etching, a mature technic in microelectronic industry needing to be adapted for this new application. The development of selective masks and a well-controlled etching through a complex material (polycrystalline with several chemical elements) are challenges to address.

This work is based on both modelling and experimental approaches. An ICP/RIE SENTECH device has been used, delivering 800 W RF power and owning a heating (up to 220°C) and biased substrate-holder. To understand the etching mechanisms of stainless steel, etching of the main components of such materials, namely Fe, Cr and Ni were studied separately. Under Cl_2/Ar plasmas, observations by SEM and chemical analyzes by EDX/XPS showed the formation of species rich in chlorine. It was demonstrated that the adsorbed chloride compounds $FeCl_2$, $CrCl_2$ and $CrCl_3$ can be etched spontaneously or by ion assisted etching. However, the very low desorption rate of $NiCl_2$ constitutes a barrier for an efficient etching of steels rich in nickel.

A surface model, based on a cellular approach to represent the etched surface, has been developed. Monte-Carlo method is used to describe the main etching mechanisms. This model has been validated for binary Fe/Cr alloys etching by comparing the simulation results with the experiments [1].

With very selective sol-gel alumina masks, long etching results of stainless steels whose patterns reach 2.5 µm in depth will be presented.

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ID 70 - The influence of Cu^{2+} on SaOS-2 and human mesenchymal stromal cells

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Cu2+ is necessary for physiological processes like angiogenesis and osteogenesis. However, Cu2+ does also show cytotoxic effects at particular concentrations. Ti6Al4V alloy disks covered with pure metallic Cu were used in this study to closely determine its concentration-dependent effects on human cells in an in vitro approach. Samples were submerged, up to 2 weeks, in α-MEM containing 10% fetal calf serum (FCS). Medium was renewed, and collected at 1, 10, 20, 40 mins, 1, 2, 4, 8, 24 h, and daily until day 14. The concentration of Cu2+ in the extracts was quantified via ICP-OES, with values ranging between 0.3 µM and 1432 µM. Human MSC and SaOS-2 osteoblast-like cells were cultured in the medium containing Cu2+ and cell viability was assessed in a luminescence-based assay. hMSC exhibited higher tolerance to Cu2+ compared to SaOS-2 cells. Results suggest that Cu2+ demonstrates cytotoxicity on SaOS-2, potentially due to the high proliferative rate of this immortalized cell line. Primary hMSC, conversely, exhibited comparatively high resistance to Cu2+. Work is ongoing to address the effect of direct cell seeding of hMSC on Cu-coated Ti6Al4V and further investigate the effects of Cu2+ on hMSC and SaOS-2 cells.

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ID 106 - UV absorbing transparent coatings in glass matrices

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Ultraviolet (UV) light is an electromagnetic radiation with three fractions: UVC (100–280 nm/4.43-12.4 eV), UVB (280–315 nm/ 3.94-4.43 eV) and UVA (315–400 nm/ 3.10-3.94 eV). The wavelength of the UV radiation is slightly shorter than the visible light (400-780 nm) but their photons carry much more energy (3.1-12.4 eV). UV light can negatively affect drug products'/medicines' quality. For instance, UV and visible light can accelerate the oxidation of fats and oils[1]. It also affects vitamins (A, B2, B12, D, E and K). Since the main source of UV radiation is the sun, it is paramount to find appropriate solutions to protect goods. Typically, light sensitive products are packaged into opaque or dark coloured packaging to avoid photodegradation. However, consumers like to inspect the food products before buying it. They feel an increased sense of security seeing the product in its unaltered form[2]. Consequently UV-shielding and transparent packaging is of increasing interest. In this work, it is explored the development of coatings with carbon quantum dots made with two different approaches. The first one uses microwave-assisted synthesis, the other is performed into an autoclave using a conventional oven. It is investigated the size and dispersion of carbon quantum dots in the liquid matrix, and the way as these two parameters they relate to the UV light absorption capacity. Also, it is followed the impact of withdrawn rate applied in the dip-coating process on the colour variation (CIELAB) and UV-shielding. The trade-off between colour and UV-shielding is discussed.

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ID 156 - Antibacterial hydroxyapatite/silver composite coatings onto mechanically improved TiMoNbTa alloy: evaluation and optimization of electrodeposition processes

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Over the last years, the design and improvement of medical implants has been of great interest. For this, hydroxyapatite (HA), being a ceramic material very similar to human bone, in combination with silver nanoparticles (Ag NPs), which are regarded as an antibacterial agent, represent a suitable alternative to enhance the performance (including anti-biofouling properties) of orthopaedic implants. The aim of this study was to explore and determine the most suitable way to develop a composite material consisting of a HA matrix with Ag NPs to coat, via electrodeposition, a previously fabricated β-type TiMoNbTa alloy with improved mechanical properties and provide it with antibacterial properties to avoid infections caused when implanted. Two different deposition methods have been evaluated: (1) a one-step method where Ag nitrate is added to an electrolyte containing the HA precursors ($Ca(NO_3)_2$ and NH_6PO_4) and a simultaneous co-deposition of metallic Ag and the HA matrix takes place; and (2) a two-step method consisting in the decoration of previously electrodeposited HA coating with Ag from Ag nitrate electrolyte. The parameters affecting the correct incorporation, distribution and content of Ag into the HA matrix were studied and optimized, aimed at having finely dispersed Ag NPs. In a similar way, initial attempts to introduce Zn(O) into the HA matrix are also shown. Thus, a non-toxic and biocompatible alloy with a functionalized surface with potential antibacterial properties was produced in a controlled, reproducible and facile manner.

This work has been done within the Biofilm-resistant materials for hard tissue implant applications (BIOREMIA) European Training Network (ETN) funded by the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No. 861046.

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ID 162 - Hydrophobic silver coated copper foams suitable for oil/water separation applications

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Marine environments are vulnerable to oil leakage causing irreparable harm to the ecosystem. Oil sorbent materials are often favored for oil-splits cleanup methods due to their low cost, ease of usage and high efficiency.

In this work a simple two-stage chemical solution process is reported, to deposite a superhydrophobic silver film on copper foams with a view to be employed in oil absorption or filtration procedures. The first stage includes the growth of a silver layer to increase micro roughness and the second evolves the modification of the film using stearic acid. The whole process has the advantage of being time-saving low cost and versatile. UV-Vis spectroscopy was employed to determine optimum deposition durations and detect potential film detachments during the synthesis process. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) were used to examine the film structure. Surface functional groups were detected by IR-FTIR spectrophotometer. An adherent superhydrophobic silver coating was achieved under optimum deposition durations. A feather-like structured morphology appeared from silver deposition and spherical, microflower morphologies from stearic acid deposition. The influence of process conditions on wettability and the obtained silver film morphology and topography were examined and clarified. Thermal and chemical stability was examined for acidic, alkaline, and salty environments under several temperatures. Filtration capacity under free and forced flow along with oil absorption capacity were also evaluated for the optimum superhydrophobic copper foams. The results show that the produced superhydrophobic foams can potentially be used to oil/water separation applications.

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ID 168 - A machine learning solution to the texture optimization problem

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Our world is overwhelmed by the environmental impact of human activity and there is imperative need to reduce pollution and mitigate its effects to help avoid irreversible global warming. In the transportation industry, among the largest contributors to polluting emissions, a significant part of fuel and energy is wasted in overcoming friction forces between moving parts in contact, thus, friction reduction methods represent a simple and rapid way to reduce our carbon footprint.

Thanks to advances in laser texturing techniques, allowing for the rapid and efficient generation of textured surfaces, the control of friction in an environmentally friendly manner is possible. Conventional means for designing and testing these textures are based on trial-and-error methods, the optimal design of a texture is impossible to achieve with traditional experimental and numerical methods.

We present a solution to this problem, a deep neural network (DNN) trained to predict the Stribeck curve of a system based only on its micro-texturing pattern. Capable of effortlessly predicting all possible cases of a total of around 100 million possibilities in seconds, trained with just 0.05% of them. We solved the texture optimization problem by taking advantage of the incredible performance of our DNN, predicting the relevant optimal textures in the process. Properties of our network such as accuracy, extrapolation, and interpolation capabilities were also determined, demonstrating its robustness and reliability.

This work paves the way for the use of machine learning as a tool to realize careful friction control of surfaces through optimally designed textures.

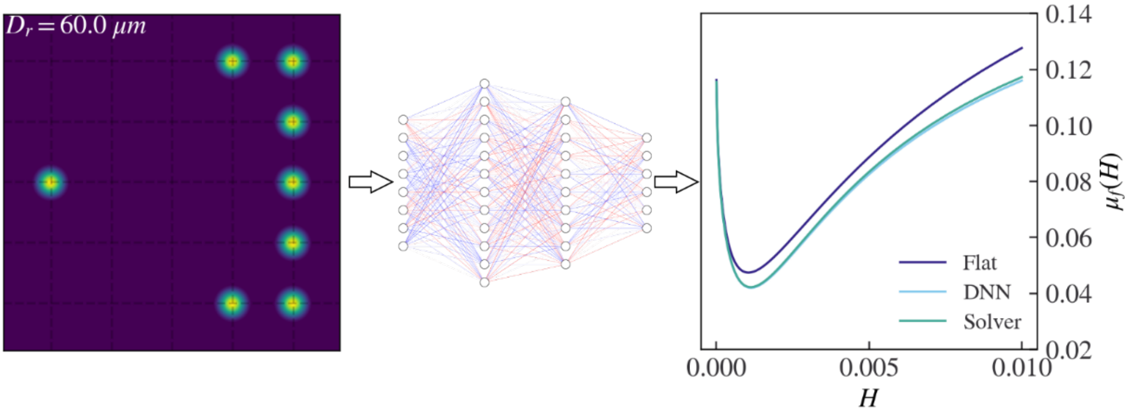

Figure 1: optimal pattern in the context of the smallest overall coefficient of friction in the hydrodynamic range solved by a traditional solver and the deep neural network.

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C.2. Advanced metallurgical processing

ID 15 - Dispersoid Evolution During Homogenization in 5XXX Alloys. Effects of Composition, Micro-segregation, and Modelling of Strengthening

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[Oral] - Non-heat treatable 5XXX Aluminium alloys have attractive properties such as weldability and corrosion resistance, but achieve only moderate strength and can suffer from sensitization. A potential route to improve both strength and reduce sensitization is to form an additional population of dispersoids by introducing new dispersoid forming elements. In this study, novel 5XXX alloys with L12 dispersoid forming additions have been produced and analyzed. To maximize the L12 dispersoid volume fraction as well as minimize cost, a combination of different dispersoid formers have been explored, namely zirconium, scandium, erbium, and yttrium. The effect of homogenization treatment on the precipitation of L12 dispersoids was studied to optimize the dispersoid size, volume fraction, and distribution. Two-step homogenization regimes, guided by a dispersoid precipitation model, were shown to precipitate many fine dispersoid particles, whose size and distribution through grains was studied using high resolution scanning electron microscopy (SEM). Electron Probe Micro-analysis (EPMA) was used to quantify the micro-segregation of elements after casting and the subsequent effect this had on precipitation in the dendrite cores and edges. The results show precipitation is highly dependent on position within grains and the local elemental concentration. The dispersoid precipitation model was coupled to a simple strengthening and work hardening model to understand the effect of L12 dispersoids on mechanical properties. The model predictions were compared with microstructural and tensile property measurements. It was demonstrated that a notable increase in both strength and work hardening was obtained in alloys with an optimized dispersoid distribution.

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ID 26 - Physical and computational simulation of Functionally Graded Materials

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Additive manufacturing (AM) of metallic materials, in particular direct energy deposition (DED) technique, has evolved up to the point of enabling manufacturing of functionally graded materials (FGMs) that show a gradual change in composition within a part or component, thus coming as a great option when dealing with dissimilar joints or create location-specific properties.

Here, we investigate material compatibility and explore processing and grading parameters for FGMs, specifically focusing on a material graded from Ni-based superalloy (Inconel 718) to Stainless Steel (316L). We combine three complementary approaches. (1) Using advanced characterization of DED-manufactured graded materials, we assess the quality (porosity), microstructures (phases) and properties (tensile, compression, nanohardness, thermal expansion) of DED-printed samples. (2) Using computational thermodynamics, we calculate thermal properties and phase fractions to assess their expected presence along the FGM gradient. (3) Finally, we design and perform original physical simulations using a Gleeble 3800 apparatus involving partially melting of the bi-material samples to investigate material compatibility and diffusion at the interface.

This study will shed light onto fundamental mechanisms of microstructure selection and properties in metallic FGMs. It will result in advanced simulation-based (computational & physical) design methods for innovative graded alloys, as well as for the optimization of their processing parameters.

This investigation was supported by the Spanish Ministry of Science under the Retos-Colaboración project MultiFAM (Ref. RTC2019-007129-5) in collaboration with AIMEN Technological Centre (Spain) and ArcelorMittal (Spain). D.T. also gratefully acknowledges support from the Spanish Ministry of Science through a Ramón y Cajal Fellowship (Ref. RYC2019-028233-I).

C.3. Thermomechanical processing and severe plastic deformation

ID 25 - Towards electrically assisted manufacturing of light metal and steel parts on a semi-industrial scale

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Efforts to reduce CO2-emissions of cars and aircrafts by lightweighting require materials with high strength-to-weight ratio. However, these materials are often inherently hard to form due to limited ductility and necessitate high forming forces at room temperature. A possible approach to reduce the forming force and improve the formability is the use of electric current pulses during forming, leveraging the electroplastic effect (EPE). The EPE in metals is known to reduce yield stress and springback, and to allow significantly higher degrees of deformation before fracture [1]. Overall energy input is significantly reduced. However, Joule heating occurs, the magnitude of which depends on the electric energy input. It is difficult to distinguish between the EPE and softening due to Joule heating. Besides mandating special tooling and lubricants, excessive heating can deteriorate the microstructure of materials, for example by coarsening of hardening precipitates in Al alloys. If Joule heating can be reduced to a minimum, temperature-sensitive materials can be deformed.

To study the influence of electric pulse length, height, and frequency on the formability of various light metal alloys and steels, we equipped a semi-industrial-scale hydraulic press with an electric system, while controlling for temperature to decouple the EPE from Joule heating. First experimental results show a reduction of flow stress in compression tests for samples pulsed with current. Subsequently, tensile testing and deep-drawing experiments (electrically assisted manufacturing) are planned. TEM will be used to elucidate the mechanisms underlying the EPE in Al, Mg, and Ti alloys as well as steels.

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ID 51 - Magnesium alloys containing yttrium, gadolinium and calcium processed by Equal Channel Angular Pressing (ECAP)

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Magnesium alloys with low density and high specific strength are suitable candidates for the use in weight-saving industries, such as the aerospace industry. The use of magnesium alloys in the aerospace industry is limited because of their low flammability resistance. Flammability resistance of magnesium alloys can be affected by alloying elements. It has been proven that rare earth metals and calcium increase resistance to ignition.

Magnesium alloy containing yttrium, gadolinium and calcium (Mg-2Y-2Gd-1Ca, wt. %) was designed for use in the aerospace industry. In order to obtain a fine-grained microstructure, alloy was processed by extrusion and also eight passes of Equal Channel Angular Pressing (ECAP). The microstructure after extrusion was bimodal. The grain size and the degree of recrystallization depended on the parameters of extrusion. ECAP processing led to homogenous microstructure with mean grain size less than 1 μm, which meets the parameters of ultrafine-grained alloys. The mechanical properties corresponded to the observed microstructure. The alloy is also characterized by a high ignition temperature and is therefore promising for use in the aerospace industry.

The microstructure was observed using transmission and scanning electron microscopy, including electron backscatter diffraction (EBSD). Mechanical properties were studied using tensile/compressive deformation tests and microhardness measurements.

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ID 64 - Analysis of the texture influence on deformation behavior of a rolled AZ31 magnesium alloy

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A rolled sheet of the magnesium alloy AZ31 exhibiting a strong basal texture was analyzed in order to reveal the influence of this texture on the activation of individual deformation mechanisms. Sets of samples with three distinct orientations – rolling direction (RD), normal direction (ND) and 45° between RD and ND (45) were subjected to compressive loading. During this, a set of mutually complementary advanced *in-situ* experimental techniques was employed to reveal the active deformation mechanisms. Neutron diffraction spectra were collected in pre-selected points along the deformation curves together with a concurrent measurement of the acoustic emission (AE) signal. The samples were further compressed inside the specimen chamber of a scanning electron microscope, allowing for the collection of electron backscattered diffraction (EBSD) patterns in corresponding stages of deformation. These measurements were complemented by a high-speed camera imaging of the deformed sample surface providing an insight in the macroscopic inhomogeneities of strain distribution, further highlighted by application of a digital image correlation (DIC) analysis on these datasets. The key influence of sample orientation with respect to the detected texture and direction of loading on the activation of individual deformation mechanisms mediating its plasticity was confirmed due to the mutually complementary information obtained from each experimental technique. Namely the activity of the $\{10\bar{1}2\}$ extension twinning throughout the straining of the samples has proved to play a crucial role in the overall mechanical properties of the deformed material, mediating yielding as well as providing potential for further strain accommodation in the favorably oriented samples.

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ID 153 - Deformation Mechanisms in Pure Heat-treated Polycrystalline Cobalt with Different High Temperature FCC Phase Content

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Systematic two-step thermal treatment was employed to control the microstructure of the pure cobalt samples and the residual high temperature fcc phase content. First isothermal annealing was applied, annealing the samples at different temperatures between 600°C and 1100°C, causing recrystallization and grain growth. Next thermal cycling around the phase transition temperature was used. This thermal preparation resulted in controlled grain size and fcc phase content. Compressive deformation testing with constant speed of deformation combined with acoustic emission (AE) technique was carried out on thus-prepared different sets of cobalt samples. The results shows that increasing grain size increases fracture strain while decreasing yield strength. As the fcc phase content decreases both yield strength and fracture strain decrease. Analysis of AE data shows further differences in the deformation dynamics in terms of basic AE parameters as well as power-law distributions of energies of the AE events. This confirms the collective nature of twinning and dislocation motion in cobalt, suggesting that interactions between dislocations result in self-organized scale invariant avalanche-like motion of dislocation ensembles.

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ID 199 - Structure-dynamics relationships in cryogenically deformed bulk metallic glass

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The atomistic mechanisms occurring during the processes of aging and rejuvenation in glassy materials involve very small structural rearrangements that are extremely difficult to capture experimentally. Here we use in-situ X-ray diffraction to investigate the structural rearrangements during annealing from 77 K up to the crystallization temperature in Cu44Zr44Al8Hf2Co2 bulk metallic glass rejuvenated by high pressure torsion performed at cryogenic temperatures and at room temperature. Using a measure of the configurational entropy calculated from the X-ray pair correlation function, the structural footprint of the deformation-induced rejuvenation in bulk metallic glass is revealed. With synchrotron radiation, temperature and time resolutions comparable to calorimetric experiments are possible. This opens hitherto unavailable experimental possibilities allowing to unambiguously correlate changes in atomic configuration and structure to calorimetrically observed signals and can attribute those to changes of the dynamic and vibrational relaxations (α-, β- and γ-transition) in glassy materials. The results suggest that the structural footprint of the β-transition is related to entropic relaxation with characteristics of a first-order transition. Dynamic mechanical analysis data shows that in the range of the β-transition, non-reversible structural rearrangements are preferentially activated. The low-temperature γ-transition is mostly triggering reversible deformations and shows a change of slope in the entropic footprint suggesting second-order characteristics [1].

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C.8. Direct and indirect additive manufacturing

ID 9 - Preparation of Superconcentrated Silver and Copper Oxide Nanoparticle Inks for LIFT-Printing of Electronics

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As the demand for miniaturization of electronics increases, so does the need for high-throughput, additive microfabrication processes and materials. Laser-Induced Forward Transfer (LIFT) responds to this need by enhancing the resolution of micron-sized printed conductive patterns[1]. LIFT prefers highly concentrated, viscous ink formulations with non-volatile solvents[2]. Inks with small particles and uniform size distributions result in dense, highly conductive printed patterns. Although LIFT has been extensively studied, the same cannot be said for the inks used for LIFT[3]. This work focuses on the preparation of highly concentrated silver and copper oxide LIFT nanoinks from commercial nanoparticle suspensions. Particle concentration of the inks prepared varied between 20 and 60 wt.% and their viscosity was greater than 10,000 cP. The nanoparticles were dispersed in glycol-water co-solvent media. Thermogravimetric Analysis confirmed the smooth evaporation of the solvents. The colloidal stability of the nanoparticles was evaluated via UV-Visible Spectroscopy and their size was studied via Transmission Electron Microscopy. The inks prepared were stable in the course of a few months and the particles were smaller than 100 nm, with unimodal size distribution. Thus, the properties of the inks were promising for LIFT.

This research has been co-financed by the European Union and Greek national funds through the Operational Program Competitiveness, Entrepreneurship and Innovation, under the call RESEARCH – CREATE – INNOVATE (project code: T1EDK-00814).

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ID 32 - In-situ detection of defects generated by spatter in laser powder bed fusion

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The reliability of laser powder bed fusion (LPBF) manufactured material is limited, particularly due to the presence of defects. Defects can occur stochastically, despite optimization of process parameters, potentially due to spatter redeposition on the powder bed. As spatter generation is intrinsic to LPBF, increasing the reliability of the process relies on monitoring their presence and determining the defects these particles generate. This study aims to detect spatter redeposits via in-situ monitoring and determine if the detections correspond to internal defects. Identical prints with varying nominal layer thicknesses were performed with optimized parameters. Long-exposure near-infrared imaging was used to monitor the builds. The images output from the monitoring system were processed to layer-wise detect spatter redeposits. It was found that, even though the spatters land primarily close to the gas outlet, there are layer-to-layer variations on their spatial distribution, mainly due to the scan pattern. With increasing nominal layer thicknesses, larger amounts of spatter redeposits are detected on a larger extensions of the build area. Virtually no defects were identified in the specimens where no spatter redeposits were detected, while in the specimens where they were detected, large defects were randomly distributed. These defects were confirmed lack of fusion, and spatters were observed in their adjacencies. Thus, spatters were confirmed as the source of stochastic defects and could be successfully detected in-situ.

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ID 39 - Microstructural Control in Additive Manufacturing of Titanium Alloys

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The control of microstructure in metallic additive manufacturing (AM) is critical to ensuring AM parts are built to as-designed specifications. With the far from equilibrium conditions and unique thermal histories intrinsic to AM build processes, understanding how to achieve microstructural control has proven a challenge across many alloy systems. Titanium alloys in particular are attractive for producing parts via AM, considering the high cost and waste associated with traditional manufacturing of these alloys. However, these alloys may also exhibit solid-state transformations that obscure the as-solidified microstructure. This makes understanding how the as-solidified microstructure and texture influences the post solid-state transformation microstructure difficult, and establishing control over microstructural evolution more challenging. This work highlights recent efforts to contend with these challenges, assessing microstructural and texture response to changes in local AM build environments, and how thermal cycling during a build process can lead to further control of titanium microstructures after solidification and solid-state transformations.

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ID 76 - WC-Co additive manufactured depositions on a HSS 390 steel substrate: a microstructural investigation

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The Direct Energy Deposition (DED-AM) is a novel 3D printing process widely employed for building layer by layer components for several industrial fields, such as biomedical, aerospace and automotive. WC-Co is a well-known cermet coating for tribological applications, mainly characterized by the dispersion of WC particles into a Co matrix acting as a binder. Considering its high wear resistance as well as its high hardness and strength, the thermal spraying technique is commonly used to perform WC-Co coatings on several substrates, but currently DED may be successfully employed as an alternative manufacturing process.

In the present study, WC-12%Co powders were deposited on the HSS 390 tool steel using two DED-laser deposition methods and different process parameters; power, scanning speed and feed rate were appropriately changed in order to consider their effect on the microstructural properties of the hardfacing material. Microstructural analyses were carried out by means of optical microscopy (OM), scanning electron microscopy (SEM) and X-ray diffraction (XRD). Preliminary results demonstrated that the use of different deposition methods as well as the number of layers could significantly affect the distribution of phases across the additive manufactured coating. Moreover, the results obtained by this experimental investigation enabled to suggest possible optimization of the process parameters to prevent the presence of different defects such as cracks and porosity.

Keywords
WC-Co, DED, microstructure, HSS 390, deposition

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ID 89 - Multiscale characterisation of maraging steel microstructure obtained by laser powder bed fusion

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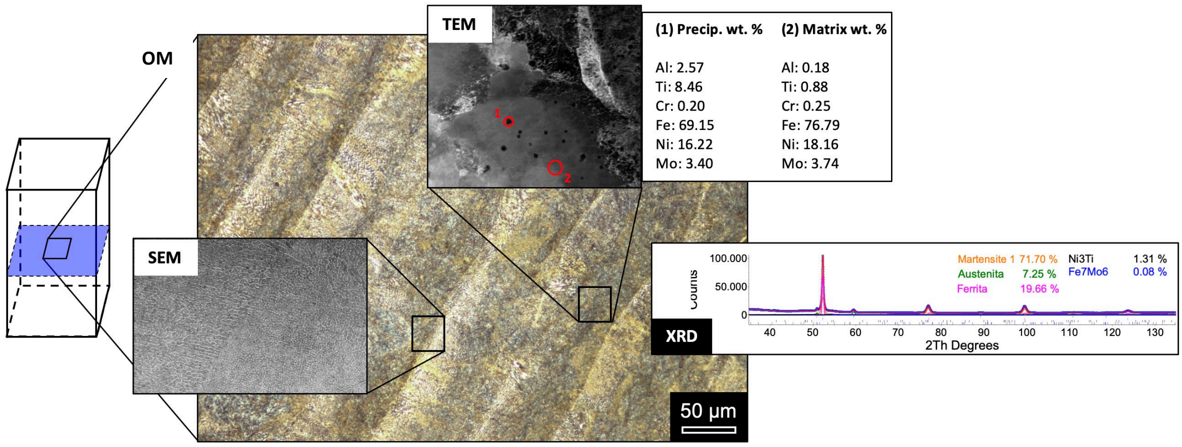
Laser powder bed fusion (LPBF) is an additive manufacturing process that allows to produce complex steel parts using a minimum amount of material. In this work, the as-built microstructure of a high performance 300 maraging steel was characterised during each stage of the process: melting, solidification, phase transformation and precipitation. Different printing parameters such as layer thickness and laser emission mode were modified. For all conditions, samples were subjected to different heat treatments with the aim of studying the microstructure evolution after ageing at different temperatures (T_{ag}), ranging from 480 to 540 ºC. Different characterisation techniques such as X-ray diffraction (XRD) analysis, transmission electron microscopy (TEM), electron probe micro analyser (EPMA) and electron backscatter diffraction (EBSD) were used.

XRD results enabled to identify three main phases in the microstructure, BCC, BCT and FCC. The EPMA analysis showed solute segregation at melt pool boundaries. TEM results revealed the presence of Ti, and Al enriched nanoparticles in the as-built microstructure. With the increase in T_{ag}, austenite growth and precipitation of Ni_3Ti and Fe_7Mo_6 intermetallics was observed. The hardness peak was reached at T_{ag} = 480 ºC. No significant effect was observed for the printing parameters studied in the evolution of the microstructure during aging, neither in the precipitated phases (carbides and intermetallics) nor in their volume fractions. It was observed that the aged microstructures are comparable to that obtained by conventional manufacturing methods.



(1) Precip. wt. %	(2) Matrix wt. %
Al: 2.57	Al: 0.18
Ti: 8.46	Ti: 0.88
Cr: 0.20	Cr: 0.25
Fe: 69.15	Fe: 76.79
Ni: 16.22	Ni: 18.16
Mo: 3.40	Mo: 3.74

Figure 1. Result selection of OM, SEM, TEM and XRD analyses.

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ID 117 - Integration of NiTi crack sensors in aluminum alloys by material extrusion (MEX)

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Novel systems that respond to unusual requests, such as self-healing, can now be efficiently produced through indirect additive manufacturing (AM) technologies such as material extrusion (MEX). In aeronautic applications, the integration of sensors to detect cracks and actuators to repair failures in critical components is essential. In this study, the material extrusion (MEX) AM technology was selected for ensuring the best adhesion between the matrix (component material) and the sensor (wire). Consequently, the layer-by-layer shaping of the part/system could be simultaneous with the integration of superelastic NiTi shape memory alloy (SMA) wires as crack sensors, followed by debinding and sintering thermal cycles without promoting the formation of other phases. Therefore, filaments were produced using feedstocks of 60 vol.% metallic powders (Aluminum Alloy (AA 7050)), organic binder, and additives. Stainless Steel 316L as the matrix was the selected standard material. The main goal was to improve the procedures for integrating wire sensors into metallic matrixes, ensuring high performance during its application while retaining the shape memory alloy properties. Before and after sintering, X-ray microtomography was performed to evaluate the adhesion efficiency between matrixes and crack sensors, and electrical resistivity measurements were carried out to assess the sensor's performance. Although the AA7050 matrix manufactured through MEX with the NiTi wire inserted shows good adhesion as green, an optimized sintering process is required to produce the whole system accurately. Even so, cold isostatic pressing improved the overall processing steps.

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ID 138 - Correlation between process parameters, microstructure and properties of $Al_{92}Mn_6Ce_2$ processed by laser powder bed fusion

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Processing by rapid solidification can enhance the performance of Al alloys due to (i) supersaturation of the Al matrix, (ii) homogeneously distributed phases, (iii) structural refinement and (iv) high-strength, non-equilibrium phase formation. Additive manufacturing, viz. laser powder bed fusion (LPBF), enables high cooling rates between 10^4 to 10^6 K/s [1, 2] irrespective of the sample geometry. This offers a unique potential for the fabrication of novel Al alloys and tailored lightweight designs. In this study, the effect of processing parameters on the microstructure of an $Al_{92}Mn_6Ce_2$ alloy was investigated by SEM, EBSD, TEM and XRD. High cooling rates caused a high amount and a strong refinement of the intermetallic non-equilibrium $Al_{20}Mn_2Ce$ phase. This microstructure combined with low porosity (0.05%) led to a high hardness (180 HV 5) and a pronounced tolerable compressive stress (> 1200 MPa). Furthermore, we have investigated the effect of baseplate heating to reduce residual stresses in scaled-up geometries. This in-situ heat treatment results in a coarsening of the microstructure and an increased amount of other phases like $Al_{11}Ce_3$ and Al_6Mn. Additionally, the decreased density (< 97%) induces a premature failure of the alloy during tensile testing. In summary, this work focuses on a better understanding of the process-microstructure-property-relationship of $Al_{92}Mn_6Ce_2$ and discusses perspectives and limits of its application in lightweight construction.

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ID 157 - Laser printed and sintered Ag nanoparticle metal-grids as bottom electrode for ITO-free organic photovoltaics

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Laser induced forward transfer (LIFT) of metal nanoparticle dispersions, combined with laser sintering in a two-step digital fabrication approach [1], has been proven a key enabling technology for the manufacturing of functional flexible microelectronic devices [2]. In this work we investigate the laser printing and sintering process of Ag nanoparticle inks, an effective technique capable of producing conductive patterns with a printing speed up to 4 m/sec. The laser-printing technique is employed to develop Ag metal nanoparticle parallel line grids as replacement to the bottom Indium Tin Oxide (ITO) electrode in organic photovoltaics (OPVs). We investigate the effect of a range of essential to the laser printing and sintering technique parameters to the overall electrical performance and morphological characteristics of the laser printed conductive grid. The formed jet is characterized with the employment of a highspeed camera, while the laser printed metal-grid optical transmittance was evaluated with a spectrophotometer and the electrical behavior of the conductive lines is assessed by means of electrical measurements in a 4-point probe IV station. The laser-printed Ag grid lines have adequate line morphology and high electrical conductivity while the Ag metal grid transparency was varied by altering the number of Ag grid lines within the OPVs ITO-free bottom electrode. Consequently, ITO-free OPVs incorporating laser-printed Ag grids bottom electrode with up to 11% power conversion efficiency will be presented [3]. The results validate the versatility of the laser printing and sintering technique, which can offer a specific solution for applications in highly efficient ITO-free OPVs.

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ID 170 - Tuning the shape memory polymer properties through 3D printing strategy: An experimental study

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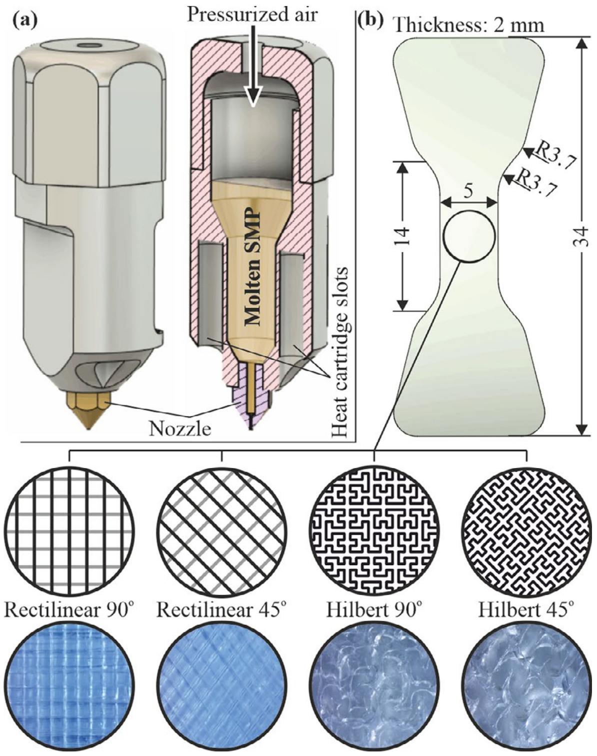
Shape memory polymers (SMPs) are a class of smart materials constituting an exceptional solution for critical applications in biomedical, aerospace and energy sectors, owing to their unique thermal shape recovery and chemical stability. Although additive manufacturing (AM) emerges as a promising method for handling SMPs to fabricate functionally graded freeform structures, while overcoming geometry limitations, the effect of AM process signature and printing strategy on the shape recovery characteristics has yet to be investigated.



To that end, a new pneumatic extrusion system (image 1a) was developed to benchmark the shape programming and recovery of a polyurethane based SMP against a conventional fused filament fabrication (FFF) process eliminating the restriction of using filaments (typical FFF) and erasing the thermomechanical history caused due to the filament production, while evaluating different printing strategies and parameters (image 1b). To demonstrate that shape recovery characteristics, such as parameters of shape fixity, recovery and transition temperature can be significantly enhanced and ‘programmed’ through a variation of AM building strategies and process parameters beyond tuning of material compositions alone, an experimental study was developed. In-situ thermo-micro-mechanical testing was applied to capture the shape memory properties, both during shape programming and post that. The pneumatic extrusion system offered higher consistency and better shape recovery characteristics compared to the conventional FFF. Finally, different shape memory response and recovery were observed through various AM process parameters proving that it is possible to modify the shape memory polymer properties by controlling the 3D printing strategy.

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ID 217 - Parametric Study of Jet/Droplet Formation Process during LIFT Printing of Living Cell-Laden Bio-ink

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Bioprinting enables the formation of living tissues in three dimensions by the precise layer-by-layer printing of biomaterials, including living cells and cell-laden hydrogels. Among the main bioprinting techniques, such as inkjet, laser and extrusion printing, laser-induced forward transfer technique (LIFT) offers the highest degree of spatial resolution (minimum feature of <10 μm) and post-printing cell viability. In this study, LIFT printing of different cell-laden bioinks has been investigated as a function of three key parameters: cell density, laser fluence, and the distance between the donor–receiver substrate using time-resolved imaging technique for visualizing the printing process. These bioinks were compared with cell-free bioinks under the same printing conditions to understand the effect of the particle physical properties on the droplet formation procedure. To evaluate the influence of LIFT on the printed cells, their growth and DNA damage profiles were assessed and evaluated quantitatively. Results from different cell densities indicated that the laser fluence and the distance of the donor–receiver substrates play an important role in the printing impingement type; consequently, a careful adjustment of these parameters can lead to high-quality printing. Overall, we find that LIFT is able to print patterns of different cell densities with high viability, little to no heat or shear damage to the cells as indicated by unperturbed growth and negligible gross DNA damage.

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C.9. Joining

ID 166 - Study of interfacial interactions between a gold layer and indium bumps for photonic packaging applications

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Photonic components such as micro-displays or infrared detectors are developed by 3D integration, where devices are stacked on top of each other or packaged. The electrical, optical and thermal connections are created between stacks and package by interconnects. Interconnection system consists mainly of a solder material (e.g. indium bumps) and Under-Bump Metallization (UBM). The contact is ensured by wetting of the top gold layer of UBM by liquid indium and formation of Au-In intermetallics (IMCs) at interfaces.
Functional detector at 7.5µm pitch was recently produced by LETI and compatibility of this technology at very fine pitch is demonstrated. However, this development highlighted the limits of this configuration because proportion of IMCs in interconnection increases with pitch reduction and may cause problems during manufacturing of components and/or their use. Therefore, thickness reduction of IMCs seems mandatory to reach pitches below 7.5µm.
The first step to control the intermetallic growth is to understand the phenomena that occur at the interface indium bumps/standard UBM. In this work, the interfacial interactions at Au/In are studied between room temperature and 200°C.
Firstly, we characterized the interfaces In bumps/Au pads at different pitches (30µm, 15µm and 7,5µm) before and after hybridization. Thicknesses and compositions of IMCs are determined using scanning electron microscopy coupled with energy-dispersive spectroscopy. Afterwards, growth kinetics of IMCs are determined in the temperature range 20-200°C using solid/solid and solid/liquid diffusion couples. Finally, wetting and spreading kinetics of liquid indium on solid gold are studied by using the dispensed drop method.

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D.1. Advanced techniques with focused beams

ID 119 - Determining the Dislocation Density of Nuclear Materials Using CMWP

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In the UK, Advanced Gas-Cooled Reactors (AGRs) use a variation of 316 stainless steel as the fuel cladding due to its high temperature and corrosion resistance. The operating lives of all of the UK AGRs have been extended over recent years. As the older AGR stations reach the point where further extensions are not viable, preparation for removing the fuel and decommissioning is the next step. As a result of these extensions, and necessary lifetime management of irreplaceable core components, the UO_2 fuel may have experienced increased burn-up and time in reactor, potentially impacting the cladding end of life properties (and therefore performance) during post-discharge and storage activities. Therefore, it is paramount to investigate and establish any physical and mechanical properties changes, in order to mitigate any potential future handling issues.

The aim of this PhD is to gain a fundamental understanding of the effects of radiation damage on the AGR fuel cladding material, stainless steel, by extrapolating data to predict damage for higher burn-up nuclear fuel. The current focus of the project is to use a novel X-ray Diffraction (XRD) technique, Convolutional Multiple Whole Profile (CMWP) analysis, in order to determine the dislocation density and defect type within a material. This is done by analysing diffractograms, specifically parameters such as peak broadening. In order to fully understand the applicability of this technique to AGR material, a metallic zirconium system will first be investigated to establish operating parameters.

In this contribution, the analysis of zirconium will be presented. An initial investigation on as-received zirconium (AR-Zr) compared to an AR-zirconium that has been heat treated, in order to anneal and recrystallise the sample, has been undertaken. The sensitivity of CMWP has been tested; data for an AR-Zr sample from two XRD machines that use a different radiation source have been compared. Future work packages, which include comparison stainless steel materials and microscopy data will also be described.

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ID 129 - "Zooming inside spider silk" - Using synchrotron nanoXRD to compare the ultrastructure of spider silks.

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Spider silk is a very fascinating material, which has been engineered by nature since almost 400 million years [1] and shows exceptional mechanical properties. In addition to its impressive mechanical performance, a variety of possible medical applications have emerged due to its excellent biocompatibility. Specifically, spider silk is a promising material for nerve regeneration and has successfully worked as center piece of nerve guidance conduits. The silk supports the adhesion and movement of Schwann cells which are responsible for the regeneration of peripheral nerves after injury [2]. In cell culture experiments Schwann cells were found to prefer certain types of spider silks to others. In this study we examine whether the ultrastructure of silk affects its acceptance by Schwann cells. For this, differently sterilized silks of *Nephila edulis* were investigated by scanning nanobeam X-ray diffraction (nanoXRD) [3]. Because of the small dimensions of the samples a sub-micron synchrotron X-ray beam was employed to get information about the silk ultrastructure in a spatially resolved manner. Nano-diffraction experiments were carried out at the European Synchrotron Radiation Facility (ESRF) ID13 beamline in Grenoble. We could observe differences regarding the degree of crystallinity as well as in the dimensions and orientation of β-poly(L-alanine) nanocrystals between the different ways of sterilization (UV, autoclavation and ethanol) compared to the native silk. Furthermore, we were able to confirm and correlate these results with findings from Raman spectroscopy.

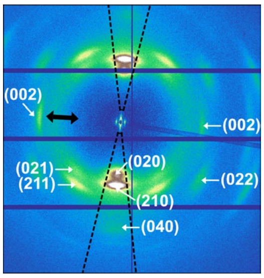

Figure 3. Scattering pattern of spider silk fibre sterilized by an ethanol treatment. Black arrow indicates direction of silk fibre longitudinal axis. (hkl) indices refer to reflections of β-poly(L-alanine) lattice. Black dashed lines enclose integration area for the (020), (210) and (040) reflections.

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Presenting author: Karolina Peter, Department of Material Sciences and Process Engineering, Institute of Physics and Materials Science, University of Natural Resources and Life Sciences, Vienna, Austria.

D.2. Characterization of nanomaterials

ID 37 - Impact of the RbF treatment in high bandgap ACIGS based solar cells

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In recent years, (Ag,Cu)(In,Ga)Se2, (ACIGS), started to gain importance in the scientific community due to its high bandgap value, compatible with tandem solar cells' top layer. Compared with champion Cu(In,Ga)Se2 (CIGS), when both materials are grown at the same temperature, the grain quality of ACIGS is improved Thus, opening the door to grow ACIGS at lower temperatures, compatible with flexible and transparent substrates. In CIGS, the alkali post-deposition treatment (PDT) allows for higher efficiency values, although the mechanisms of the positive impact of PDT are not yet understood. When we address ACIGS, the introduction of another element increases even further the complexity of the impact of the PDT. Hence, the main objective of this work is a preliminary study on the effect of the PDT on high bandgap ACIGS.

In this work, it was grown an ACIGS layer with a composition that allows for high bandgap values, although far from the conventional ratio of the state-of-the-art (A)CIGS. The samples were characterized structurally and morphologically by XRD, GI-XRD, Raman Spectroscopy, XPS, and TEM-EDS, and optoelectronic properties were scrutinized by EQE and admittance spectroscopy measurements. Comparing the XRD and GI-XRD, the plane (112) is more predominant in the sample with the PDT. TEM-EDS revealed the formation of a RbInSe2 compound. The optoelectronic results showed an increase in the open-circuit voltage value by 60 mV, compared with the non-PDT ACIGS, although the PDT led to a decrease of the fill-factor by 8 % in absolute values, possibly indicating, again, the presence of a RbInSe2 layer.

D.3. Micro- and Nano-mechanics and modelling

ID 23 - Classical And Ab-Initio Atomistic Simulations Of $\sum 3\ [\bar{1}10](\bar{1}\bar{1}2)$ Incoherent Twin Boundaries In Gold: Structure And Interactions

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Nanotwinned materials are the subject of many researches because they show mechanical properties that are usually antagonistic, namely high yield strength and good ductility [1]. We performed tensile tests on gold thin films with nanotwins, in-situ under X-ray diffraction and we observed the growth of twins in the crystal [2]. Atomistic simulations are a useful complementary tool to better understand the deformation mechanisms occurring in these materials. We first focused on $\sum 3\ [\bar{1}10](\bar{1}\bar{1}2)$ incoherent twin boundaries (ITBs). These ITBs can be described by a set of three Shockley partial dislocations where two of these dislocations are mixed and one is a pure edge [3]. The structural relaxation of such an ITB is induced by the slip of the pure edge dislocation of each set; this slip leads to the formation of a new crystalline phase, the 9R phase, whose width determines the relative lateral displacements between the two grains bordered by the ITB. We estimated the extension of the 9R phase in gold with molecular statics (with two commonly used potentials) and with density functional theory computations [4]. Finally, we studied the interactions between parallel ITBs. The results showed that, depending on the distance and the associated planes stacking between the ITBs, they can merge, generating an extended 9R phase. This helps in understanding the migration of the ITBs, and hence the stability of nanotwinned materials.

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ID 47 - Investigation of the dynamics of deformation mechanisms in Mg-Gd alloys using *in-situ* experimental methods

Andrea Szabóová[1], Kristián Máthis[1], Daria Drozdenko[1], Michal Knapek[1], Gergely Farkas[2], Gergely Németh[2], Petr Harcuba[1]

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The dynamics of deformation mechanisms activated in binary magnesium-gadolinium alloys with respect to amount of Gd was investigated with *in-situ* experimental methods. Cast alloys were characterized by random texture. Compression test were done at room temperature with simultaneous record of acoustic emission response. The acoustic emission signal was subsequently analysed using advanced clustering method providing information about the dominant deformation mechanisms. High speed camera was used to study the dynamics of twinning, including estimation of the velocity of twin propagation with respect to Gd concentration. The results clearly indicate that the twin propagation rate is of order of 10^1 m/s and it slows down with increasing Gd concentration Deformation tests were repeated in a chamber of scanning electron microscope (*in-situ SEM*) with concurrent following the microstructure development using secondary electrons and electron back-scattered diffraction (EBSD) in different stages of the deformation. Main goal of this measurements was to identify active slip systems and the progress of twin volume fraction during deformation.

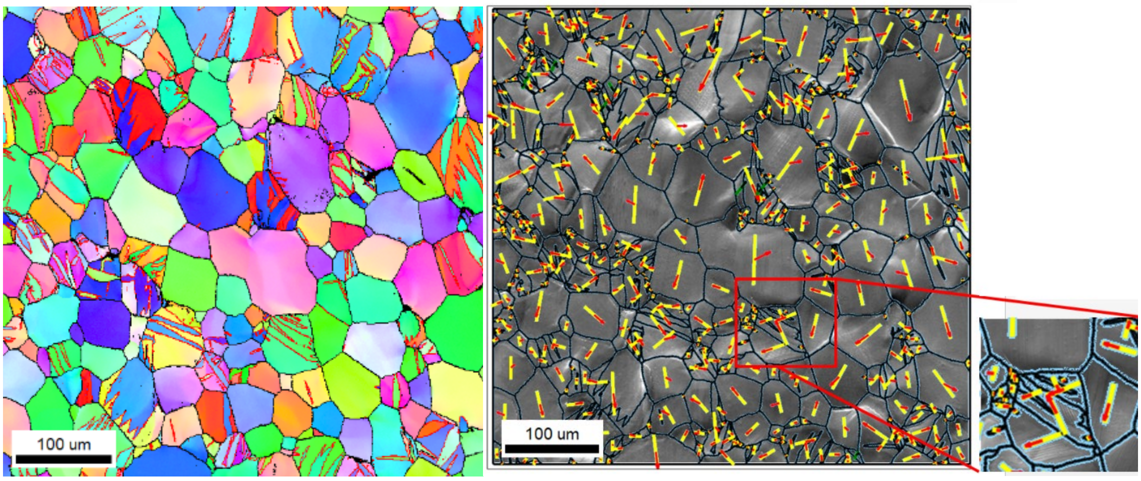

Fig. 1 – IPF map of the deformed sample and slip traces of the SE image

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ID 73 - Characterization of Strain Localizations under Cyclic Loading through Multi-Scale Digital Image Correlation Study

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Fatigue damage of polycrystalline metallic alloys involves highly localized plastic strains at a microstructural scale. The mechanistic understanding of damage accumulation and the development of microstructural scale deformation models require the characterization of localized plastic strains under cyclic loading. Digital Image Correlation (DIC) is a versatile experimental technique, which is used for quantitative analysis of local strain distributions at various length scales. In the context of microscopic strain field quantification, high-resolution DIC measurements using nano-sized speckle patterns are conducted to achieve details on the sub-grain scale strain localizations through the formation of slip bands. Although this method provides valuable information for the mechanism of strain localization, it is applicable only to a small field of view and in-situ analysis is limited with a few examples. In this work, two different DIC approaches are employed to characterize the cyclic plastic behavior of Stainless Steel 316L. First, in-situ DIC analysis with stereomicroscopy technique is performed to obtain statistically representative, grain-scale strain response through a larger field of view. This is achieved by transferring the pattern with micron-scale details to the specimen surface with the lithography method. Second, high-resolution DIC in SEM is conducted ex-situ to resolve sub-grain scale strain localizations around individual slip bands. The information gathered through DIC measurements is linked with the microstructural characteristics of Stainless Steel 316L investigated using Electron Back Scattering Diffraction. The methodology has the potential to reveal the scale-dependent response of material microstructure under fatigue loading and to promote the development of microstructural deformation models.

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ID 88 - Generating Synthetic Image Data for Titanium Alloys via Generative Adversarial Networks and Variational Autoencoders

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Machine learning is gaining traction amongst the materials science community. The potential applications are extensive, but a common drawback is the current lack of available labelled data on the scale required for complicated learning tasks. Such vast datasets are expensive, time-consuming and cumbersome to acquire. As such, it is common to utilise convolutional neural networks (CNNs) that are pretrained on the ImageNet dataset (over 1 million natural images across 1000 classes including vehicles and pets). These pretrained networks work relatively well in some applications, but intuition says that bespoke training on an equivalent dataset for microstructural images would offer more meaningful image representations for classification. Truly representative synthetic image data could be used to construct such datasets, to verify this without the cost and labour required to physically acquire and label the real image data.

Here, we explore generative adversarial networks (GANs) and variational autoencoders (VAEs). These methods share a similar architecture - they are both generative algorithms comprised of a pair of CNNs. However, GANs aim to generate realistic synthetic images from a random vector contained in the so-called latent space and VAEs aim to construct the latent space, usually referred to as the encoded space in this instance, with the aim of reconstructing the input image exactly. This parity is investigated here, with the goal of furthering our knowledge of the GAN latent space, which is currently poorly understood.

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ID 98 - TEM in-situ straining experiments applied to investigate localized deformation in irradiated stainless steels

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The objective of this work is to contribute to the understanding of processes occurring at early stages of deformation leading to localized deformation in austenitic stainless steels.

To achieve this, our approach is to perform in-situ straining (ISS) experiments, at the interior of a Transmission Electron Microscope (TEM). This type of experiments provides direct information of the microstructural processes occurring while the sample is being strained, such as nucleation and movement of dislocations, interaction between dislocations and defects and processes occurring at grain boundaries.

The material under study is the AISI-316L, because due to its high corrosion resistance and good mechanical properties, it is a recurrent material in the construction of internal components of nuclear power plants, and as an FCC metal, the slip systems that allow deformation in this type of structures are limited and are easily recognizable. Two irradiation conditions have been studied: (1) ISS specimens implanted with helium ions and (2) ISS specimens irradiated with iron ions. Both of them along with the non-implanted material, have been tested and the evolution of the microstructure under deformation has been video recorded. After the ISS experiments inside the TEM, the resulting microstructure have also been characterized by means of SEM/EBSD.

The specific qualitative and quantitative information obtained from dislocation-defect interaction or early stages of defect-free channels formation will be presented and discussed.

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ID 124 - Elastic buckling of graphene: a molecular static and DFT study

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Buckling of coatings and thin films is a well-known phenomenon that has been widely studied both from a numerical and an experimental point of view. Due to high compressive stresses, a thin film is likely to delaminate and buckle from its substrate. This phenomenon is well described by the Föppl von-Kàrmàn (FvK) equations within the framework of linear elasticity [1].

In the case of 2D materials such as graphene, the validity of continuum elastic behavior extracted from the FvK equations may be questionable. In particular, the thickness is a parameter that needs to be known to describe, in the framework of the theory of elasticity, the thin film buckling. Unlike conventional materials, the thickness of 2D materials is not accurately defined and different values have already been proposed in the existing literature for graphene [2]. This is the scope of the study to determine the thickness of graphene that best enables an elastic description of this 2D material buckling.

To do so, static molecular (SM) simulations and Density Functional Theory (DFT) calculations have been performed. Characteristic equilibrium profiles at the atomic scale have been calculated for different compressive strains (see one configuration in Figure 1). The evolution of the critical strain for buckling will be presented as a function of the buckle size and the crystallographic orientation, armchair or zigzag [3].

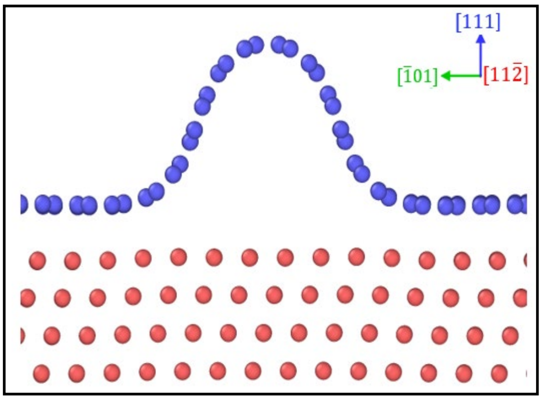

Figure 1: Characterisitic buckled graphene (in blue) on a Cu (111) substrate (in red), submitted to a compressive strain at 4% along the $[\bar{1}01]$ axis corresponding to armchair orientation (static moleculat simulations).

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D.4. Atomic scale modelling of advanced materials

ID 42 - Atomistic simulations of surface-induced amorphization kinetics in γ-Alumina nanoparticles

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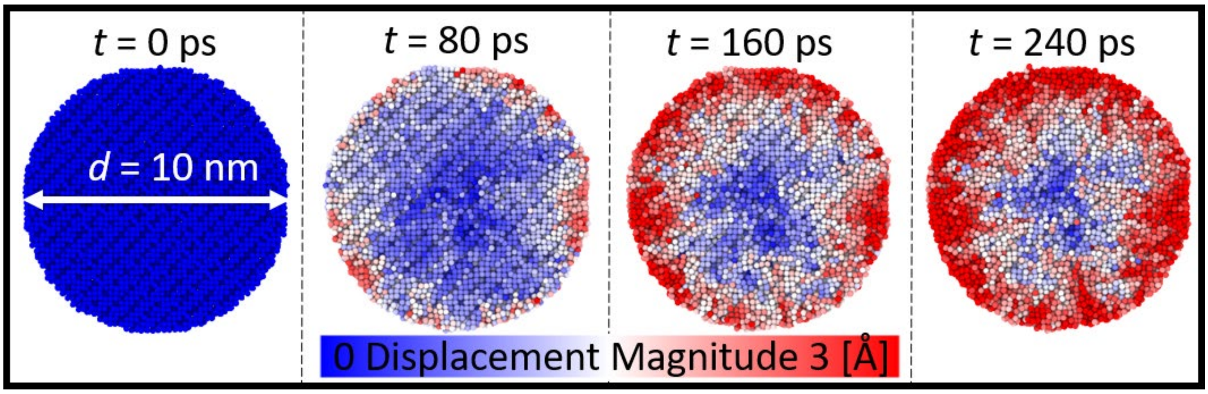
Aluminum oxide nanoparticles (NPs) are of great relevance in the fields of catalytic reactions as well as oxide dispersed strengthening of metal matrix composites, in particular, applied to metal additive manufacturing [1,2]. Specifically, the surface structure in relation to nanoparticle size and temperature is defining their performance as catalysts and nucleation sites. Atomistic simulations provide a comprehensive atomic-scale description of the surface-related phenomena.



In this work, we used the reactive third-generation charge-optimized many-body potential to access the structure and chemistry of gamma-Al2O3 NPs in large-scale molecular dynamics simulations. We provide a unique study of relatively-large nanoparticles, corresponding to commercially available ones in order of 10-nm size. We found that at an intermediate temperature of 900 K gamma-Al2O3 NPs undergo surface-induced structural transformation to the amorphous phase, which is in alignment with available experimental observations [3]. The concept of distorted octahedral coordination polyhedras is introduced as a common structural element of both gamma and amorphous Al2O3. This new study highlights the importance of amorphous alumina as the common structure of nanoparticle surfaces, defining the application of nanosized Al2O3 at intermediate temperatures.

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ID 99 - β-TiNb in presence of antibacterial Ga and Cu by ab initio calculations

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β-TiNb alloys belong to the second generation of biocompatible materials suitable for orthopaedics due to their low stiffness, excellent corrosion resistance and relatively low Young's modulus. The presence of Ga and/or Cu antibacterial elements could enrich these β-TiNb properties making them more effective against biofilm-related infections. This work aims to study the structural and electronic properties as well as the Bulk modulus of β-Ti-45NbX (X=Ga and Cu up to 8%). It is found that both Ga and Cu in Ti-45Nb prefer to have Ti atoms as first neighbours against Nb in agreement with Sn, In and Hf substitution in Ti-40Nb. The β-Ti-45NbX (X=Ga, Cu) lattice constants decrease with X content due to their smaller atomic radius compared to Ti and Nb. Ga substitutions introduce new low energy states (even at -7.60eV) with Ga-4s and/or Ga-4p character revealing bonding and/or anti-bonding features with the Ti first neighbours altering the system's mechanical properties while the Ga-3d electrons are well localized at -14.30eV. On the contrary, Cu 3d band stands together with the main β-Ti-45Nb band from -4eV up to the Fermi level showing a clear peak at -3.50eV due to d-d interactions. These results might be use for a fundamental understanding of the β-TiNb alloys in presence of Ga or Cu substitution aiming to design an implant with antibacterial properties.

ID 122 - Atomistic assessment of copper melting point depression in confinement with AlN

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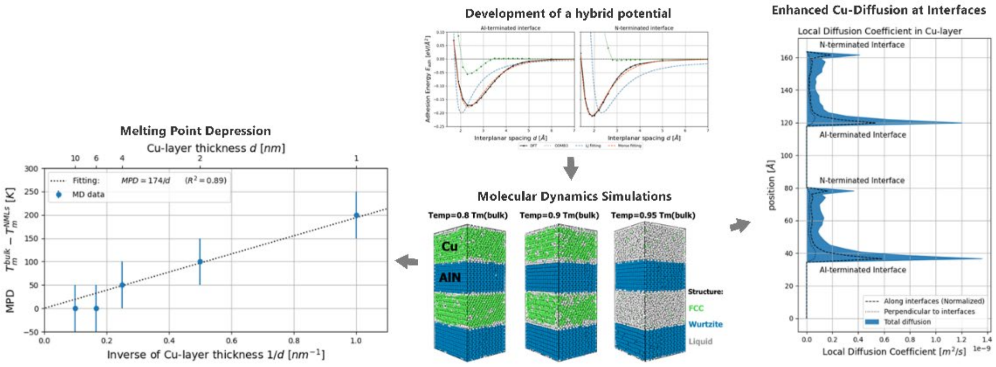
Experiments demonstrated Nano-multilayers (NMLs) of Cu confined with AlN to gain an extraordinary high atomic mobility, well below its bulk melting temperature. Except the known strong dependency on the defect structure of the confining interfaces, a detailed atomistic understanding of this extraordinary property of metals in nanoconfined systems is lacking up to date, bringing to the goal of this work.

We used *ab initio* calculations to construct a hybrid potential, and performed large-scale atomistic simulations to clarify the origin of the anamolous high atomic mobility of Cu in confinement with AlN at the nanoscale. We made several important observations. Cu metal exhibits a melting point depression, which is proportional to the inverse thickness of the confined Cu nanolayer, in excellent agreement with thermodynamics predictions. Interfacial premelting of Cu metal occurs at even lower temperatures and propagates through the entire layer. Formation of defects/premelting at interfaces results in a drastic enhancement of the diffusion rate of Cu atoms along the Cu/AlN interfaces.

The work clearly demonstrates that the extraordinary fast diffusion of Cu in Cu/AlN NMLs is driven by premelting within the Cu layer occurring at the incoherent Cu/AlN interfaces. These fundamental findings provide essential ground for tailoring low-temperature atomic mobilities along heterointerfaces for a wide variety of nanotechnologies, such as in the fields of joining, solid-state batteries, and low-temperature sintering technologies

We thank Prof. Nicola Marzari (EPFL, Switzerland), Dr. Andrej Antušek (STU, Slovakia), and Dr. Olivier Politano (UBFC, France) for fruitful discussions and valuable feedback on our work



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D.5. Multiscale and multiphysics modelling of materials

ID 45 - Glass forming ability exploration of Ti-based bulk metallic glass systems using machine learning tools.

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Bulk Metallic Glasses (BMGs), in particular Ti-based BMGs, are attractive materials presenting an excellent set of properties for use in biomedical applications. Due to their amorphous nature, they generally exhibit enhanced corrosion resistance and much higher yield strength than their crystalline counterparts. However, the conventional design of metallic glass systems and the optimization their Glass Forming Ability (GFA) often require laborious experimental steps. Hence, new investigation strategies such as combinatorial and computational approaches have emerged recently to accelerate their discovery. In this work, a Machine Learning (ML) guided study is proposed to explore new Ti-based BMG systems and predict their relative GFA. The ML regression model used consists of a genetic algorithm combined with a neural network. It evaluates the GFA of multicomponent systems through the prediction of two main experimental glass former parameters: the critical diameter (D_c) and the supercooled liquid range (ΔT_x). Our model is built on a dataset gathering nearly 500 different BMG compositions with their respective D_c and ΔT_x collected from literature. The algorithm is employed to answer two objectives. Firstly, a prediction of the composition with the highest GFA within the well-studied TiZrCuPd system was completed. Secondly, the prediction was extended to find the composition with the highest GFA in a Cu-free Ti-based system. For both objectives, the compositions predicted as the best glass formers were produced and characterized to assess the quality of the model.

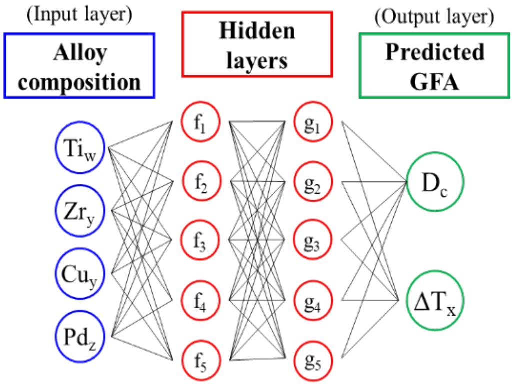

Fig. 1. Schematic of a regression neural network.

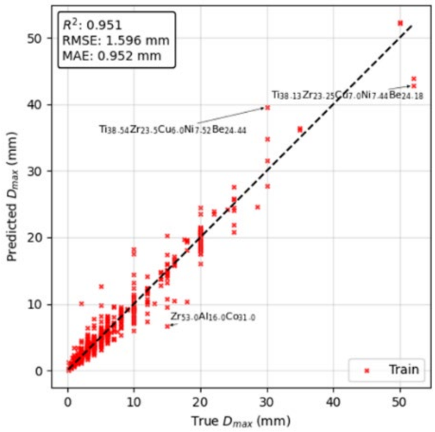

Fig. 2. Fitted Dc vs. experimental Dc obtained by the model.

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ID 63 - Accelerating phase-field microstructure simulations by combining Fourier transforms and parallelization on graphic cards

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Microstructural features like grain morphology, size, and crystal orientations are of great importance due to their close relationship with the mechanical performance of structural materials. These features are greatly dependent upon the conditions in which the solidification and heat treatments are developed.

Phase-field models play a prominent role in modern material science since they allow simulating the evolution of complex microstructures at the scale of representative volume elements. The problem to solve numerically takes the form of relatively simple, yet strongly coupled, partial differential equations. However, a fine spatial discretization is required if one aims at describing a sufficient level of detail in the microstructural features, therefore making simulations quite computationally expensive.

Here, we present an original method to tremendously accelerate phase-field simulations, at the expense of a relatively minor accuracy cost. We combine semi-implicit Fourier spectral-based methods with massive parallelization using graphic processing units (GPUs), using Python programming language with PyCUDA and Scikit-CUDA packages. We use a benchmark simulation for Ostwald ripening as a test case combining Cahn-Hilliard and Allen-Cahn coupled differential equations, and demonstrate a remarkable speedup (by orders of magnitude) for a comparable accuracy, to a great extent thanks to the ease of building a semi-implicit scheme using FFT and the resulting enhanced stability of the resolution with a greater time step.

This publication has emanated from research conducted with the financial support of Science Foundation Ireland under Grant number 16/RC/3872. D.T. also gratefully acknowledges support from the Spanish Ministry of Science through a Ramón y Cajal Fellowship (Ref. RYC2019-028233-I).

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ID 69 - Modelling the Microstructure Evolution During Hot Working of Titanium Alloys

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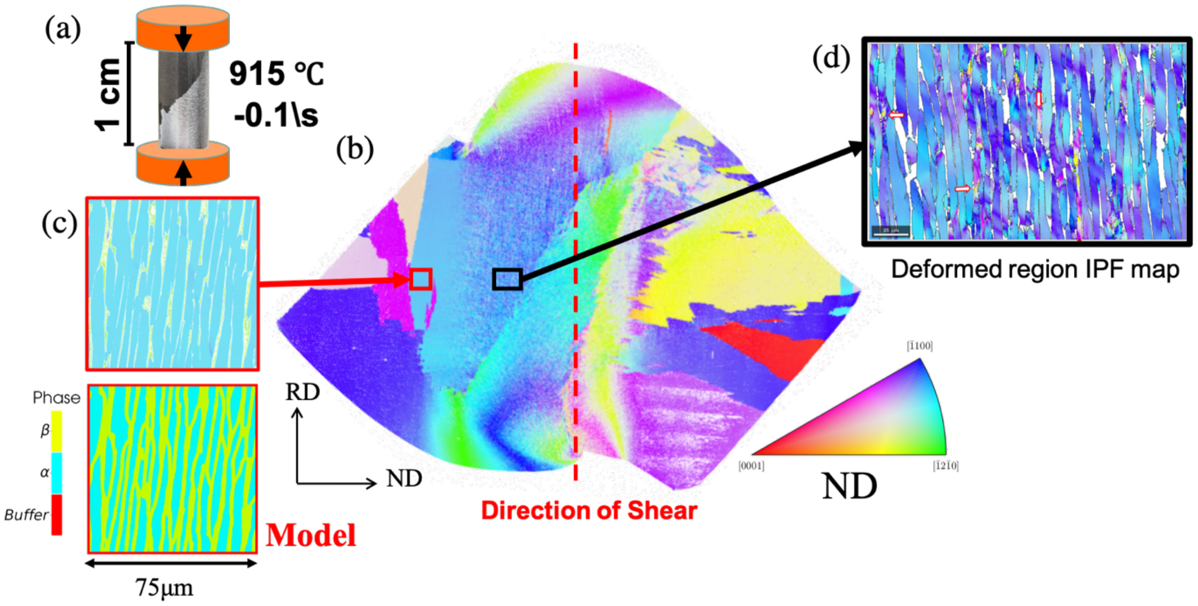
Crystal plasticity modelling has been used to determine the effect of strain rate, critical resolved shear stresses and morphology of the α and β phases on their texture evolution in Ti-6Al-4V during rolling in the β-transus temperature region. The model was used to replicate a uniaxial compression experiment on a sample consisting of large prior β-phase grains with few α-phase colonies within. Neglecting phase transformation mechanisms in the model, it has been shown that for low volume fractions of β-phase, no change in strain rate sensitivity, CRSS or initial morphology could account for the development of $(0002) \parallel TD$ texture components in α-phase observed in literature, suggesting this component forms due to variant-selective phase transformation or recrystallisation mechanism.



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ID 136 - Modelling Environmentally Assisted Cracking in Ni-based Superalloys

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Turbine disc Ni-based superalloys form a surface oxide layer when exposed to high-temperatures and corrosive reagents. Growth of the oxide layer results in a γ' precipitate depleted region at the oxide-matrix interface due to preferential transport of oxide-forming elements to the growing surface oxide. As these superalloys are strengthened by the dispersion of these coherent γ' precipitates, their depletion results in a deterioration of mechanical properties. A mean-field model has been developed to predict the γ' precipitate depletion zone in RR1000, a commonly used Ni-based turbine-disc superalloy. Initial particle size distributions (PSD) are divided into a series of discrete size classes, corresponding to the equilibrium phase fraction of γ'. A multicomponent diffusion driven growth-rate law is formulated to determine the dissolution rate of each discrete particle size class. This is based on CALPHAD obtained energy and mobility expressions of the solutes contained within the oxide. The model is first validated against existing precipitate depletion zone kinetics for RR1000. The effect of temperature on the PSD, specifically as the γ' solvus temperature (~1100°C) is approached, is then investigated, and the effect of oxygen partial pressure (ppO_2) on the PSD, where a larger ppO_2 leads to an increased oxide growth rate, hence a higher γ' dissolution rate. Finally, the effect of altering alloy composition on the PSD is studied. Further work would involve coupling this model to a crystal plasticity model for deformation, to predict the competition between stress build-up due to oxide wedge protrusion and relaxation due to precipitate dissolution.

This work is supported by the Engineering and Physical Sciences Research Council (EP/S022635/1) and Science Foundation Ireland (18/ EPSRC-CDT/3584) through the Advanced Metallics Center for Doctoral Training.

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ID 195 - Effect of casting parameters and geometry on 26-ton ingot defects evaluated by numerical simulation analysis

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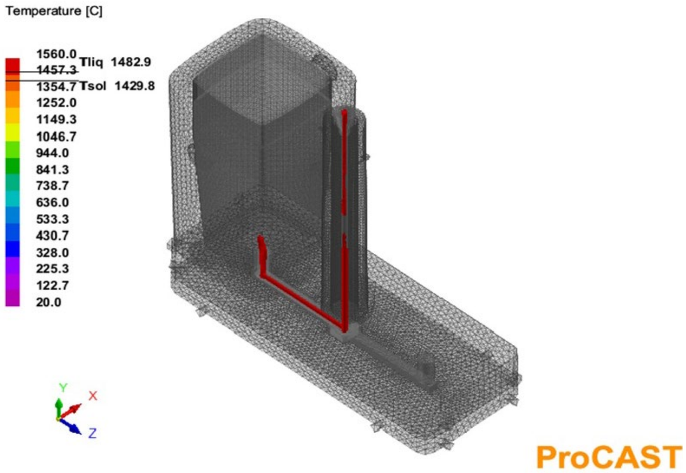
The demand for high-quality ingots leads companies to use advanced tools to predict defects within components, such as numerical simulation software. However, difficulties in defining detailed and reliable input data lead to partial or incomplete simulation results, thus limiting the attention only on approximate outputs. In fact, to perform a more realistic simulation, it is necessary to set the proper material properties, process parameters (flow rate, initial temperatures, etc.), heat transfer coefficient as well as the appropriate geometry and corresponding discretization. Another important aspect to consider in simulation of large castings is the calculation of convective flows during solidification, which increases the computational complexity but provides more realistic information.
This paper shows the results of a complete 3D numerical simulation of a 26-ton steel ingot casting, by using ProCAST® commercial software. Different casting and geometric parameters were modified to evaluate the risk of defects formation within the ingot, like inclusions originating from the entrapment of casting powder. It was also determined the impact of different filling methods and convective motions during solidification on the prediction of defects, such as inclusions, macrosegregations and shrinkage porosities. Furthermore, the reliability of the developed simulation model was assessed by means of casting experimental tests performed at industrial scale. Finally, on the basis of the findings, the runner and gating system was optimized by using simplified simulations.



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E.1. Critical raw materials

ID 59 - Biosurfactants for separation of ultrafine mineral particles

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Froth flotation is a foam-based industrial method for mineral separation. The selective separation is achieved by making the target mineral particles hydrophobic by surfactants (collectors). Separation of fine mineral particles, especially using environmentally less impactful approaches, is one of the main problems in processing of low-grade ores and re-processing of tailings. Fine (- 20 µm) particles present a long-standing problem for flotation due to many effects such as high surface area, high reactivity (oxidation and dissolution), heterocoagulation, and non-selective entrainment. One of the solutions could be found in replacing conventional petroleum-based surfactants used to selectively float target minerals with biosurfactants (eco-friendly surfactants produced by benign microbes from food waste). Since the current knowledge of the interaction of biosurfactants with minerals in general and fine mineral particles in particular is limited, more investigation is required to fill this gap. This work aims at filling this gap to find an environmentally friendly way for separating ultrafine particles (< 10 µm). Toward this aim, we investigate the interaction of acetylated acidic and acetylated lactonic sophorolipids with oxide particles of hematite, quartz, and cerium oxide in terms of the separation of valuable metals. Our results demonstrate that biosurfactants can separate hematite from cerium oxide and quartz. We discuss possible mechanisms of the adsorption behavior of biosurfactants onto the mineral surfaces

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E.2. Materials addressing the 3R Reduce

ID 27 - Preparation of geopolymer material from used AZS refractory and unrecycled glass fibers

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Ordinary portland cement OPC is the most widely used binder material in the construction industry but it is related to environmental problems with its high CO_2 emission during production. Geopolymer-based binders are found to be alternative binder materials that are environmentally friendly compared to OPC since they can be synthesized from inorganic waste materials and reduce the carbon footprint of OPC. Some of these materials are glass waste and refractory wastes. unrecycled waste glasses from various sources have non-degradable chemistry and they are disposed of in landfills having an enormous impact on the environment. A huge amount of used refractories is becoming a concern for landfilling. Since these materials contain a high amount of SiO_2 and Al_2O_3, they can be used as precursors for synthesizing geopolymer cement with excellent chemical and mechanical properties. Geopolymer samples were prepared by mixing different amounts of glass fiber waste with AZS refractory powder using two activators, mainly NaOH and/or KOH, where their concentration ranges from 3-8M. After the samples were cured at ambient temperature, FTIR, XRD, and SEM were used to characterize the produced geopolymers. Porosity, density, and strength tests were made on the samples after 28 days of curing.

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E.3. Carbon-neutral production of materials

ID 83 - Pathways for the Italian Steel Industry of the Future: Possibilities and Criticalities

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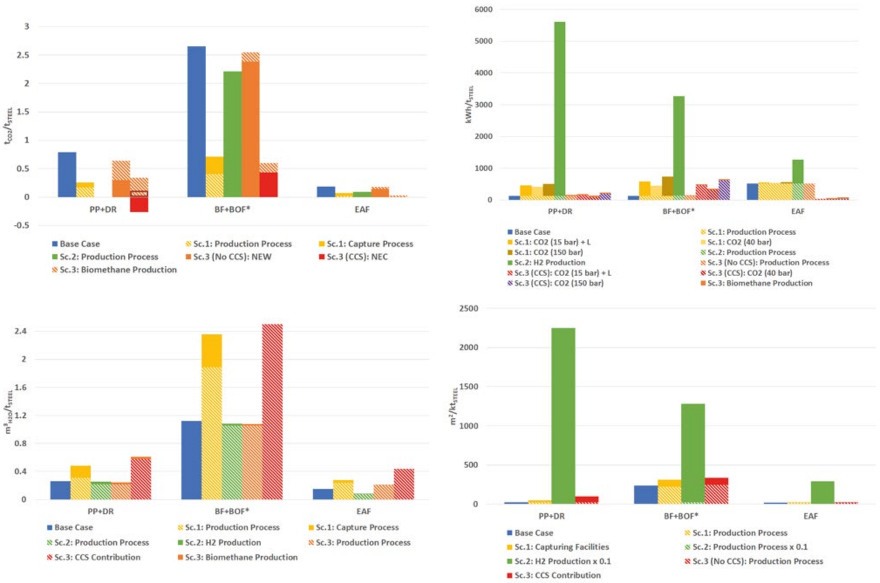
Carbon dioxide emissions, water and electricity consumption and soil exploitation are used to investigate the possible future pathways for the steel production, in Italy. The environmental advantages and criticalities for the application of three decarbonization solutions (carbon capture and storage, use of green hydrogen and use of biomethane) were analyzed for each of the following production routes: integrated cycle, scrap recycling and iron ore direct reduction. The theoretical assumptions were integrated by background production data collected from five Italian steel mills, and a cradle-to-gate approach was followed to characterize the whole steel production cycle (from the iron ore extraction to the final steel product), taking into account also the auxiliary sources of emissions (i.e., limestone calcination and gas compression for CCS). Overall, the results of the assessment should be considered as a starting point for understanding of the sustainability level of current Italian steel industry and allow to highlight the most promising and plausible and its criticalities in each of the three production route analyzed. The natural gas substitution by biomethane proved to be the most promising pathway, with significant environmental emission reduction when applied to the integrated cycle, especially if combined with CCS systems. On the other hand, even if the green hydrogen pathway is the lowest emitting one overall, its huge soil exploitation related to the correspondent energetic demand, create a boundary and practical issues on its feasibility in the Italian context.



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E.4. Bio-based materials

ID 132 - Cellulose-based biopolymer optical fibers and their theoretical attenuation limit

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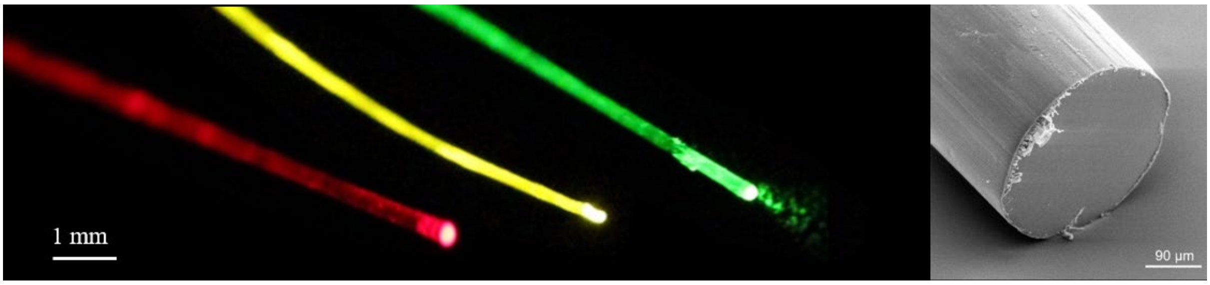
Cellulose is one of the most abundant biopolymers on earth. Due to its sustainable availability and its ability to manage light interactions, cellulose is increasingly used in optoelectronic devices to replace fossil-based polymers. To optimize cellulose-based optical fibers, it is necessary to counteract the well-known yellowing. Accordingly, an improved dissolution process for cellulose was developed. Optical properties such as transmission and refractive index as a function of wavelength were determined from planar films of regenerated cellulose, cellulose diacetate, cellulose acetate propionate and cellulose acetate butyrate. Cellulose fibers were produced via a simple wet-spinning process. They were then coated with cellulose derivatives to form a core-cladding optical fiber structure. The cellulose-based optical fibers exhibit significantly reduced attenuations compared to previously reported ones. The ultimate transmission loss limit was simulated to characterize the attenuation progression. The obtained results show that extrinsic factors still have a significant influence on the attenuation and limit the technical application potential of cellulose-based optical fibers. Improving fiber production thus leads to a reduction of extrinsic attenuation factors and to an approximation of the ultimate transmission loss limit. This opens up the possibility to substitute fossil-based polymer optical fibers in the future.



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ID 139 - Cottonid – a forgotten biogenic material with a promise

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Cottonid, vulcanized fiber and leather stone - many names exist for one of the oldest known bio-based plastics. It is made from unsized cotton and/or pulp paper via a parchmentizing process using zinc dichloride. All materials used throughout the manufacturing process can be recovered. This makes not only the process environmentally benign, but also Cottonid itself, as it only consists of cellulose. Techniques from wood joining and machining can be used to process the material. It has a high toughness and strength but is yet breathable.

The presented talk will provide an overview of the customizable properties of Cottonid and its possible applications. Through variation of parameters such as temperature and time of the parchmentizing process, it is possible to set the crystallinity and density of the material. This enables us to tune the mechanical properties, hygroscopicity, optical properties, electrical properties and even the rate of the biodegradation. Mechanical testing, differential scanning calorimetry and X-ray diffraction characterize the process-structure-property relationship. This data-driven approach, enables the manufacture of materials for a given tailored applications. These include autonomous actuators, packaging solutions and substrates for catalysts.

In the field of bio-based and sustainable materials, Cottonid has a high potential to replace fossil-based plastics or to open up new applications. Additionally, the reaction route via zinc dichloride can be transferred to the wide area of all cellulose composites and might lead to a new, more sustainable way for their fabrication.

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ID 141 - Amine-cyclodextrin-based nanosponges – novel polymeric sorbent materials

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Cyclodextrin-based nanosponges (CDNSs) are defined as 3D-hyperreticulated nanoporous structures with high thermal stability, and possible response to external stimuli. The usage of cyclodextrins represents a dual advantage due to the ability to form host-guest interactions with hydrophobic molecules, whereas the cavity and porous structure provides a high surface area promoting the interaction with hydrophilic species. Albeit nanosponges have been applied in different fields (see Figure 1), several parameters can influence their performance such hydrophilic/hydrophobic ratio, functional groups, pH and cavity diameter, among others.

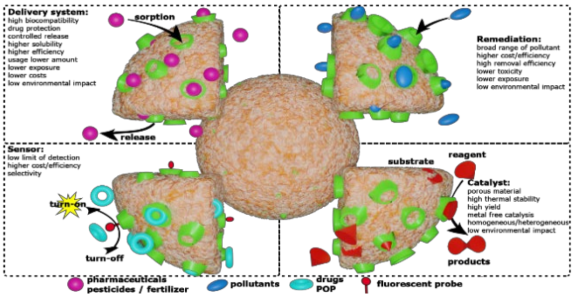

Fig. 1. Schematic representation of CDNSs, main applications and their advantages.

In this study, CDNSs were synthesized using α- and β-CD previously functionalized with two aliphatic diamines: hexane-1,6-diamine and dodecane-1,12-diamine[1]. The characterization of the CDNSs was performed by FTIR, TGA, DLS and ζ-potential, N_2-adsorption/desorption, SEM and elemental analysis.
The α-CDNSs and β-CDNSs were applied as sorbent materials for the removal of the active substance (imidacloprid) of a commercial pesticide formulation: CONFIDOR-OTEQ®.
Our aim is to assess the effect of the α- and β-CD as well as the chain length of the linkers in the CONFIDOR removal. The sorption ability of NSs for CONFIDOR were studied by sorption kinetic and isotherm analysis performed at optimal s/l ratio and pH conditions. The results show an impressive effectiveness in terms of maximum sorbed amount of ca. 200 mg g^{-1} (45% removal efficiency) and 240 mg g^{-1} (96%) for αCD-am$_6$-CD and βCD-am$_6$-CD, respectively.

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Reference: Utzeri G. et al., Colloids Surf A. 635:128044, 2022.

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E.5. Materials circular economy

ID 10 - Isolation, Recovery, Recycle of Critical Metals and Rare Earth Elements from Waste Electric and Electronic Equipment and their Reusability in Additive Manufacturing applications.

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The demand for high-efficiency, low-energy consumption novel products, combined with high durability and thermal stability, has led to an increased demand of critical raw materials (CRM) and Rare Earth Elements (REE)1. Waste electric and electronic equipment (WEEE) contain large quantities of CRM and REE that can be isolated, recovered and recycled, leading to environmental and economic benefits, due to the landfill decongestion of potentially hazardous materials and their reusability in added value materials, respectively.

CRM and REE can be extracted from the plethora of disposed end-of-life WEEE. Their recovery and reusability, constitutes the inception of a circular-economy, zero-waste and sustainable-by-design value-chain paradigm.

Owing to the convenience of preparing complex and customizable shapes with short production time and little material waste, Additive Manufacturing (AM) exhibits unique technical versatility, integrating it gradually into various applications (medicine, electronics, sensors, automation)2.

The introduction of CRM as additives, employed by AM applications, can lead to tremendous potential for the performance and the commercialization of the final products by adding unique characteristics (antibacterial properties, photoluminescence, enhanced mechanical and magnetic properties, thermal and electrical conductivity)3.

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Presenting author: Vasileios Stratiotou-Efstratiadis, vstratio@auth.gr, Physical Metallurgy Laboratory, Mechanical Engineering Department, AUTH, 54124 Thessaloniki, Greece

ID 176 - Potentially Sustainable Way for Neutralization and Iron Recovery from Red Mud

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Bauxite is an important ore that is widely used to produce metallurgical-grade alumina (the precursor for aluminum production) and chemical-grade alumina/aluminum hydroxide for many industrial applications through the Bayer process [1]. During this process, a byproduct is formed, known as red mud (RM). Depending on the quality of the bauxite and its processing, about 15-40% bauxite ore presents the waste in the form of alkaline slurry. In Bosnia and Herzegovina there are two locations for RM disposal: Klisa near city of Zvornik (around 19M tons of solid red mud) and Dobro Selo near city of Čitluk (around 4M tons of solid red mud). The utilization and recycling of this material is currently serious environmental issue not only in Bosnia and Herzegovina, but in the whole Europe and it needs to be addressed as soon as possible. The handling of this byproduct is a major challenge for the alumina industry due to its high volume and high alkalinity which impose severe and alarming environmental problems, such as soil and air pollution [2]. In particular, pH of RM is substantially higher than any other waste - different studies report that pH ranges from 10 to 13.5 regardless of the origin of RM.

However, red mud can be considered as a valuable secondary raw material resource [3], due to the presence of valuable substances such us iron oxide, aluminum oxide, silicon oxide and rare earth elements (REEs) which carry certain economic potential. This material is studied for various industrial applications in the field of building (geopolymers, clay material, cements, ceramics, fired and nonfired building materials, concrete industry), pollution control (in wastewater treatment, absorption and purification of acid waste gases), metal recovery (iron, titanium, aluminum, REEs), coagulant, adsorbent, catalyst and in soil remediation (calcium silicon fertilizer) [4].

Further handling, disposal, and use of RM is the function of several properties, but at the first place pH value. Extraction of valuable constituents is interfered due to its alkalinity. It is necessary to find a sustainable technique for its neutralization in order to make it applicable. This paper demonstrates research on developing an economical way of neutralization of the red mud and methods of iron recovery from it.

Acknowledgements
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ID 184 - Potato washing slurries-derived starch used in the development of aerogel microparticles with ethylene scavenging capacity

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The global wide consumption of potato and its derived food products results in a broad range of not-used by-products even though they contain valuable molecules, such as starch. On the other hand, the non-biodegradable ethylene scavengers are used to decompose or adsorb ethylene, a phytohormone responsible for the induction of mechanisms related with fruits ripening and senescence [1], to prolong their shelf-lives and reduce food waste. In this work, the feasibility of adding value to potato washing slurries-derived starch (PWS) through the development of aerogel microparticles with ethylene scavenging activity was studied, using an emulsion-gelation method followed by a supercritical drying as experimental procedure. Commercial potato (CP) starch was used as control.

The PWS aerogel microparticles showed an average diameter (50 ± 15 μm) similar to the CP ones (53 ± 24 μm). Nitrogen adsorption–desorption IV-type isotherms revealed that both mesoporous microparticles had similar average pore size (13-14 nm). However, PWS aerogel microparticles had a higher surface area (130 m^2/g) and total pore volume (0.600 cm^3/g) than the CP microparticles (102 m^2/g and 0.404 cm^3/g, respectively). These textural properties gave rise to a higher ethylene adsorption capacity (0.557 $mmol. g^{-1}. bar^{-1}$), when compared to CP microparticles (0.424 $mmol. g^{-1}. bar^{-1}$).

Therefore, the PWS revealed to be a suitable raw material for producing aerogel mesoporous particles with ethylene adsorption capacity, opening an opportunity to valorize starch-rich agrifood by-products through the development of renewable ethylene scavengers.

Acknowledgments

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POSTERS

A.2. Flexible and porous materials

ID 29 - Composites based on PDMS and Graphite Flakes for Thermoelectric Touch Detectors Applications

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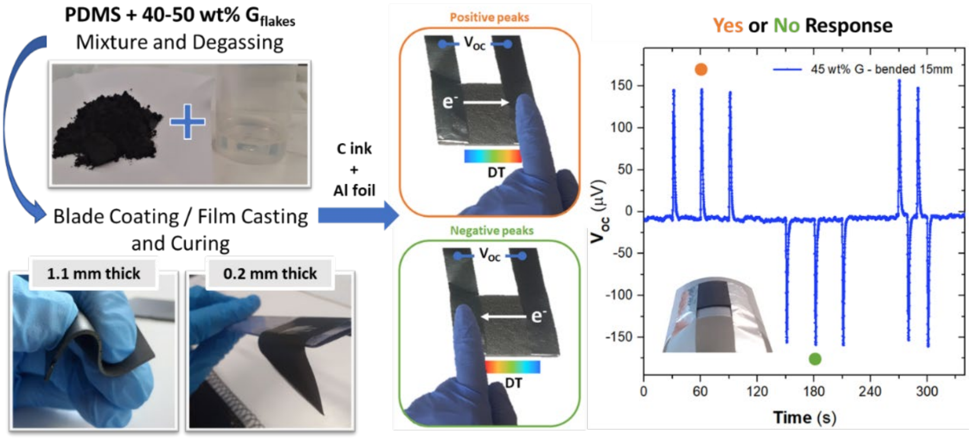
Within the concept of the Internet of Things there is an emerging class of devices that are worn in, on and around the human body and are able to sense, record and exchange data with surrounding networks.

When touched, a thermoelectric detector converts the thermal gradient generated between the skin and the detector into an electrical signal. Among several desirable features, this kind of device should have a high sensing performance (such as, high and stable signal-noise ratio values with fast response), good mechanical properties and, not less meaningful, it should be low-cost and suitable for scalable manufacturing.

This work presents freestanding thermoelectric touch detectors consisting of graphite conductive flakes into a polydimethylsiloxane matrix. An optimal concentration of graphite flakes (45 wt%) lead to robust and homogeneous detectors that exhibited signal-noise ratio values up to 170 with rise and falling times below 1 s and 7 s, respectively. The detectors performance was stable over continuous operation and did not reveal significant degradation while bended under different curvature radii (45, 25 and 15 mm) and consecutive bending cycles. Moreover, the twist of the thermal gradient direction between the electrodes of the detector enables a Yes or No response which opens new usage possibilities. Therefore, this work provides an efficient way to develop robust, low-cost, and scalable thermal detectors with potential use in wearable technologies.



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ID 93 - Improving the properties of flexible hybrid-silica aerogels

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Silica aerogels with their remarkable properties such as low density and thermal conductivity are prime candidates for a wide range of different applications, such as insulation materials in aviation. While pure silica aerogels are very brittle and difficult to handle, adapting the sol-gel process can result in organic-inorganic hybrid-materials with a high mechanical flexibility, a good hydrophobicity while still retaining a sufficient insulating performance. They were first investigated by Hayase et al. [1] and are based on methyltrimethoxysilane (MTMS) and dimethoxydimethylsilane (DMDMS).

For further improvement of the material's properties, the synthesis needs to be tuned. Starting with recipes filed for patenting by the German Aerospace Center (DLR) [2], the synthesis route was first investigated towards varying precursors and their ratios, using different solvents and mixtures, as well as testing a wide range of surfactants and catalysts. Mechanical, thermal, chemical and morphological properties of the samples were investigated.

After further polymeric crosslinking the metal-oxygen bonds can be partially substituted by the more lightweight carbon-backbone, while retaining the flexibility of the material. Therefore, investigations using a consistent precursor system of MTMS and dimethoxymethylvinylsilane (DMMVS) are carried out.

The above-mentioned properties are required in the field of aviation, and the resulting aerogels can possibly outperform the state-of-the-art compressed fiberglass-mats.

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ID 178 - Effect of Heat Treatments on the Mechanical Properties and Microstructure of Open-Cell Al 7075 Foams

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Open-cell metallic foams are a family of porous metals constituted by a solid metallic phase coexisting with an interconnected porous volume. This material combines the advantages of a lightweight and interconnected-porous structure with the properties of metals, such as high electrical and thermal conductivity, as well as high mechanical strength and toughness. Thereby, the development of mechanically enhanced open-cell metallic foams has become a crucial issue to be investigated, aiming to expand the functionality of this material for science and engineering applications, e.g., crash dissipation structures. In this work, the effect of solution and aging heat treatments on the microstructure and mechanical properties of open-cell Al 7075 foams was studied. The investigated foams were produced by the replication casting technique in two different pore sizes, ranging from 2.00 to 4.75 mm. All produced samples were physically characterized through He pycnometry. Solution treatment was carried out at 480 °C for 2 h, whilst aging treatment was carried out at 120 °C for several periods, aiming to assess the precipitation strengthening response of foams as a function of aging time. The microstructural evolution of the porous Al 7075 was evaluated by means of scanning electron microscopy, X-ray diffraction, and scanning differential calorimetry. In addition, the heat-treated foams were mechanically characterized using uniaxial compression and hardness tests. It was found that pore size did affect the response to solution and aging treatments of open-cell Al 7075, due to cell walls thickness variations.

Acknowledgments
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ID 236 - Optimization of flexible and eco-friendly thermoelectric touch detectors based on ethyl cellulose and graphite flakes inks for screen printing

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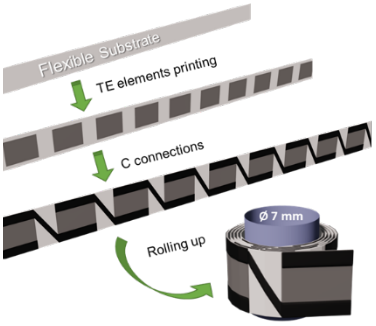
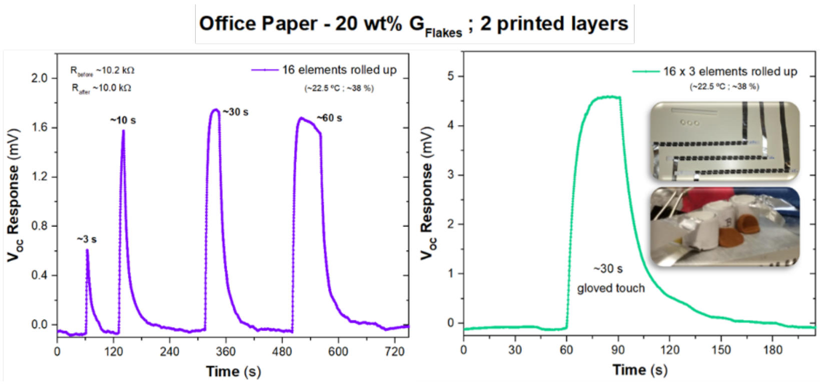
There is a window of opportunity for thermoelectric materials related to the wearable market's growth and the prediction that in a near future everything and everyone will have sensors and will be connected. For this to happen, scientists need to search for biocompatible, flexible, and lightweight materials, and develop technologies that work well with small stimuli, like little temperature gradients and small pressures. When touched, thermoelectric detectors are able to convert thermal gradients into electrical signals and this response can be optimize through the active materials, the substrate's choice and the adopted architecture.

This work presents the fabrication of flexible and eco-friendly thermoelectric touch detectors based on ethyl cellulose and graphite flakes inks that were screen-printed in four different substrates - office paper, sticker label paper, natural cotton fabric and organic poplin fabric - with several geometries and architectures. The best inks formulations, printing conditions and printable substrates were chosen based on sheet resistance measurements, scanning electron microscopy analysis, and open circuit voltage over time tests. We found that office paper and natural cotton were more suitable for the touch detection, in terms of repeatability of results and fast responses. Moreover, the performance of the devices was stable through time, and did not reveal significant degradation after dynamic bending tests as well as static mechanical deformation. The maximum voltage amplitude reached was of 4.69 mV corresponding to a rolled-up detector with 48 elements connected electrically in series wrapped around a cardboard straw with a curvature radii of 3.5 mm.





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A.3. Smart and self-healing materials

ID 77 - Development of amphiphilic polymeric additives for lubricant formulations using SARA ATRP

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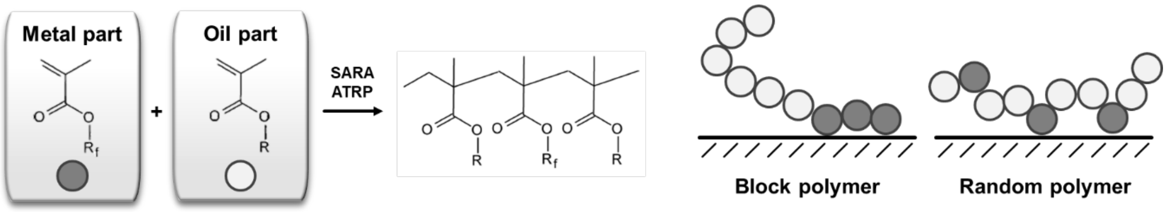
The lubricant viscosity is of uppermost importance in fulfilling its protective and friction-reducing function. Lubricant viscosity can be manipulated by the introduction of a long linear chain polymer [1]. Unfortunately, the different polymers studied so far did not perform well over time due to the impossibility of synthesizing them with tailor made structures, avoiding the stringent control over parameters such as mechanical strength and the specific interaction with the different components of the system.

Some previous studies showed that polymer branching degree is crucial to the additive's performance [2]. Using the Atom Transfer Radical Polymerization (ATRP) method, different monomers were used to prepare a new star-polymer, but the results obtained were not relevant in friction reduction. Recently, Helgeson et al. [1] studied the synthesis of several copolymers based on stearyl methacrylate and methyl methacrylate with different topologies through an ATRP strategy. However, the ramifications make it difficult to form films under high loading conditions, increasing the system's fluidity and lubrication. Moreover, these copolymers synthesis uses expensive reagents, harmful chemicals, and a complex protocol.

In this study, block copolymers with two segments were synthesized: a segment which present excellent compatibility in oil and another segment with an excellent affinity for metal structures [3]. The proposed synthesis method is based on a variation of the ATRP method discovered by our group and is called the Supplemental Activator Reducing Agent (SARA) ATRP [4], allowing the synthesis of the block copolymers under conditions that can be applied in large scale production.



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ID 226 - Healable and recyclable cellulose-based ionic conductors for sustainable electronics on paper

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Novel nature-based engineered functional materials combined with sustainable and economically efficient processes are among the great challenges for the future of mankind. In this context, this work presents a new generation of eco-friendly and sustainable advanced functional materials, obtained from the most abundant biopolymer resource on Earth, which is cellulose.

Highly conformable regenerated cellulose ionic hydrogels with high ionic conductivity, reversible water retention and self-healing ability are successfully produced using a facile and “greener” preparation method based on NaOH/urea aqueous dissolution system of cellulose. Due to their robustness as well as soft and sticky surface, the hydrogels can be supplied in the form of reusable stickers, easily molded as plasticine, and thus applied “on-the-fly”, through a “cut, transfer and stick” process.

Instead of ionic conductive stickers, their electrochemical properties can be merged with the mechanical robustness, thermal resistance, transparency and smoothness of micro/nanofibrillated cellulose nanopaper to yield an ionic conductive “paper-like” substrate.

The engineered cellulose-based ionic conductors are successfully integrated into flexible, recyclable, low-voltage (<3.5 V), printed electrolyte-gated transistors and “universal” logic gates fabricated on office paper or on the ionically modified nanopaper. Ubiquitous calligraphy accessories are used for easy and quick patterning of conductive/resistive tracks “on-the-fly” by merely drawing them out on the substrate hosting the devices. This user-friendly and simplified manufacturing approach holds potential for fast production of low-cost, portable, recyclable and low-power ion-controlled electronics on paper, making it attractive for application in sensors and concepts such as the “Internet-on-Things”, while ensuring a sustainable future without generating waste.

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A.4. Surfaces & Interfaces

ID 6 - Microstructural influence on the growth of anodic aluminum oxide

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Aluminum oxide layers are naturally forming as protective layers on aluminum samples. As these layers are only a few nanometers thick, anodization can promote the formation of higher thicknesses. The thickness and structure of anodic aluminum oxides (AAOs) depends on the applied voltage, the used concentration of the electrolyte and temperature [1,2]. Using properly adjusted acid solutions, hexagonal and highly ordered nano-porous structures are generated with pore diameters of down to a few nanometers by using high-purity aluminum with a mirror-like surface finish [1,2].

Unfortunately, pure aluminum is not suitable for most applications in industry due to its low hardness and strength. Thus, aluminum alloys with predefined alloying elements, i.e. Si or Mn, are chosen to achieve desired mechanical properties. As the formation of the protective alumina layer is suppressed in many alloys, the good chemical resistivity diminishes. Here anodization is mandatory to obtain a necessary protective coating. However, the existence of alloying elements in aluminum alloys hinders the formation of highly ordered AAOs. In addition, mechanical stresses accumulate at the obstacles which promotes the formation of nano- and microcracks inside of the AAOs, causing a reduction of the protective ability of the coating. The growth of AAOs in acid solutions on aluminum alloys is analyzed by means of electron microscopy i.e. EBSD, EDX and (S)TEM to understand the influence of microstructure and segregations on the alloys mechanical and chemical properties of the generated oxide.

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ID 179 - Yttria/ceria stabilized zirconia composites: evaluation of microstructural, mechanical and color properties for biomedical applications

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3-mol% Yttria-Stabilized Tetragonal Zirconia Polycrystal (3Y-TZP), a ceramic biomaterial, has been widely used in the dentistry field due to its biocompatibility, tooth-like color and mechanical properties [1]. Despite these advantages, its physical and mechanical properties tend to degrade as a result of a low-temperature aging process induced by its sensitivity to low-temperature degradation (LTD) when it is in contact with water that is already at human body temperature [2]. Because of this, many studies have been performed aiming at avoiding this phenomenon by replacing 3Y-TZP by 12 mol% ceria stabilized tetragonal zirconia polycrystal (12Ce-TZP). Nevertheless, compared to 3Y-TZP, 12Ce-TZP has lower strength which is partly related to its larger grain size [3].

In this sense, this work intends to improve the aging resistance without substantially affecting the mechanical strength of zirconia by producing 3Y-TZP/12Ce-TZP composites by cold pressing and sintering technique. The test samples such discs were produced with different amounts of ceria additions (wt. %), powders mixture methods (dry mixing and aqueous medium) and sintering temperatures (1400ºC and 1500ºC). The proposed solution presents two different designs (i) bulks of yttria /ceria stabilized zirconia composites and (ii) layered 3Y-TZP/12Ce-TZP composites on the surface of 3Y-TZP substrates. The samples were characterized regarding microstructural, mechanical and color properties. Results demonstrated that the 3Y-TZP/12Ce-TZP composites produced through the aqueous medium and sintering temperature of 1400ºC presented an improved aging resistance, flexural strength complying the requirements of the ISO 13356:2008 standard and suitable color properties, validating both designs for biomedical applications.

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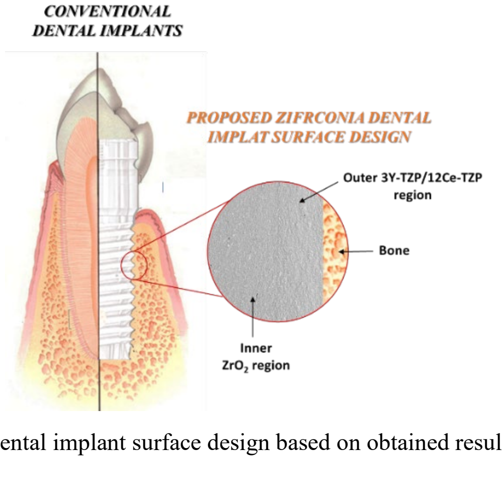

Figure 1. Representative zirconia dental implant surface design based on obtained results.

ID 190 - Cinnamomum camphora leaves as templates for the preparation of hydrophobic films

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Nowadays one of the major boosts in material science is given by inspiration from Nature. Biomimetics is the biologically inspired design, adaptation or derivation from Nature. Green plants can be employed as models for the development of biomimetic surfaces with different surface properties. Leaf wettability is a common phenomenon for plants in a wide variety of habitats. For example, the leaves of the Lotus plant are known for their "self-cleaning" ability and extremely water repellent characteristics [1]. The development of bio-inspired materials and devices with potential commercial interest is of crucial importance for the coating industry.

This work is focused on the physical and chemical characterization of the adaxial (upper) and abaxial (lower) surfaces of the leaves of Cinnamomum camphora collected at the Botanical Garden of UTAD [2]. Wettability results from the interplay between the leaves physics (surface morphology) and chemistry (surface wax composition). The wettability was quantified by means of static contact angle measurements, using the sessile drop method, on different locations of both sides of the leaves. To determine the chemical composition of the waxes, qualitatively and quantitatively, the epicuticular and intracuticular wax layers of each surface were extracted mechanically (cryo-adhesion). The morphology was analysed by Polarized Optical Microscopy (POM) and Atomic Force Microscopy (AFM) in tapping mode. The Replicas of the surface of the plant leaves were prepared and characterized.

Acknowledgments

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ID 198 - Study of the wettability of films obtained by the replication of the surface of plant leaves

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Nature usually shows many marvellous characteristics. Biological surfaces such as the Lotus leaves have micro- and nanostructures and low-surface-energy, and exhibit excellent superhydrophobicity. It has become an important research topic to construct bionic superhydrophobic surfaces and explore their functional applications [1]. In the last few years the wettability of certain plant leaves has been evaluated by the materials science community seeking inspiration for the creation of biomimetic films.The template method is a synthetic method that takes a template as the main configuration to control the morphology and adjust the size of structures to determine the properties of materials. The direct replication of structures of plant leaves is the most straightforward and effective method for fabricating superhydrophobic surfaces. Based on the facts that sol-gel-derived di urea cross-linked short-chain poly(oxyethylene) (POE)/siloxane hybrid di-ureasil films are promising candidates as mold materials for micropatterning [2], and that group has already managed to reproduce a negative replica with a di-ureasil hybrid [3], we proposed to produce replicas of different plants found at the Botanical garden of UTAD [4]. The wettability of the replicas was quantified by means of static contact angle (CA) measurements, using the sessile drop method, performed in different locations of the replicas of the adaxial and abaxial surfaces. The morphology was analysed by Polarized Optical Microscopy (POM) and the fidelity of leaf replication was investigated.

Acknowledgments
This work was funded by the R&D Project PORPLANTSURF - Superhydrophobic films inspired in the surface of plant leaves and petals from Northern Portugal, POCI-01-0145-FEDER-029785, financed by the European Regional Development Fund (ERDF) through COMPETE 2020 - Operational Program for Competitiveness and Internationalization (POCI) and by the Foundation for Science and Technology (FCT). The authors acknowledge CQ-VR for financial support UID/QUI/00616/2013 and UID/QUI/00616/2019. P. Nunes acknowledges CQ-VR/FCT for a PhD grant (UI/BD/151084/2021). M. Fernandes acknowledges FCT-UTAD for the contract in the scope of Decree-Law 57/2016 – Law 57/2017.

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A.5. Energy and environment materials

ID 38 - Hydrogen storage in porous metal hydride - polymer hybrid materials

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Hydrogen is an irreplaceable energy vector of a carbon-neutral future but high-performance storage must be available to allow its proper usage. Although metal hydrides hold the promise to be the superior storage option (energy efficient, highest volumetric energy density), many hurdles, including slow kinetics, insufficient stability, and unfavorable pressure-temperature conditions for loading-unloading operations, need to be surmounted. A promising candidate is FeTi, being one of the few compounds allowing near-ambient absorption and desorption.

The goal of this investigation is to solve the mentioned problems by prototyping a porous metal hydride-polymer composite (Fig. 1). Single-phase FeTi was prepared by arc melting and subsequently crushed and ball milled. The obtained powder was blended with varying amounts of immiscible sacrificial phases (Cu, NaCl or Na_2SO_4) and refined to a nanostructured condition using high-pressure torsion at ambient or elevated temperatures up to 400 °C.[1] The sacrificial phase was subsequently removed selectively by wet-chemical dissolution.[2] The resulting porous structure with high surface area is beneficial for hydrogen absorption.

Further developments include infiltration of the pores with a polymer filler, selectively permeable to hydrogen gas, that provides mechanical stability[3]. The final hybrid material and all intermediate stages are structurally characterized by diffraction methods and electron microscopy and the hydrogen storage properties are evaluated.

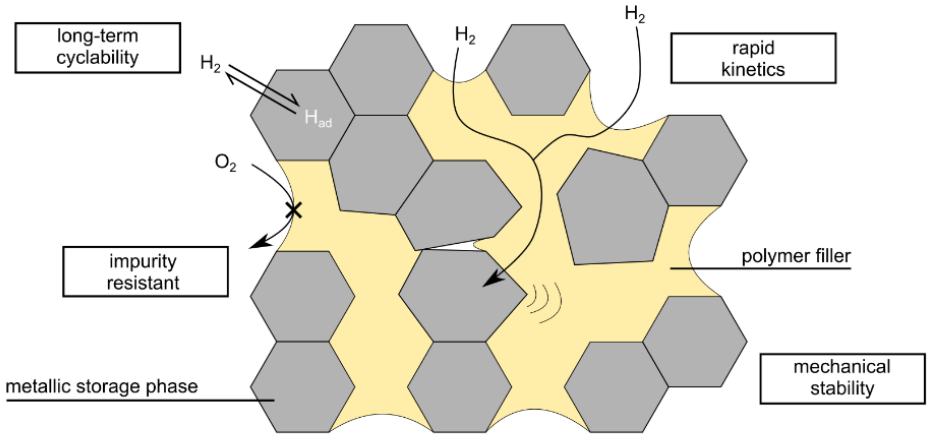

Fig 1: Schematic metal hydride-polymer hybrid material

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ID 43 - Porous PDMS conformable coating for high power output carbon fibers/ ZnO nanorod-based triboelectric energy harvesters

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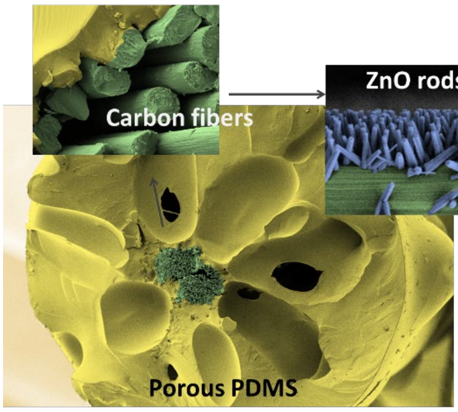
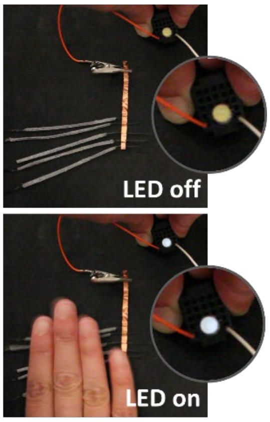
A new method for depositing PDMS directly onto conductive carbon yarns is proposed to produce Triboelectric Nanogenerator Yarns (TENG yarns) that can serve as the basic building blocks for energy scavenging wearables. The in-situ PDMS curing method described in this study allows the fast formation of a uniform thick coating over conductive surfaces regardless of their roughness. Single-electrode configuration TENG yarns are developed and their electrical output is optimized by precisely adjusting the PDMS layer thickness and by changing the chemical and physical nature of the carbon fiber (CF) yarns' surface. Functionalizing the CF yarns' surface with ZnO rods combined with porous PDMS coating can enhance their electrical output. The best results are achieved using this type of TENG yarns with an average diameter of 1.74 mm, which can be obtained after only 3 min of PMDS deposition by "in-situ" curing method. A maximum of 72 V peak-to-peak and 10 µA (74.1 µW cm□ 2 of power density with a load resistance of 20 MΩ) is reached when applying an impact force of 600 N to a set of five TENG yarns connected in parallel. The output is stable even after 10,000 cycles and this set of TENG yarns is also able to light at least 28 LEDs when tapping by hand, proving a contribute towards the development of basic building blocks to power the future generation of wearables.





ID 58 - Cork functional sodium-rich ferroelectric solid state structural batteries

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The advantage of the structural batteries is related to the allocation of their volume and weight to elements that pre-exist with structural functions. The overall weight of the battery is then reduced, and the stability of the structure is highly enhanced. Starting from this concept, several designs for structural power composites have already been proposed.

Here we characterized electrochemically, electrostatically, and structurally the ferroelectric-electrolytes families, Na_3ClO and $Na_{2.99}Ba_{0.005}ClO$ and structural batteries containing these electrolytes [1].

Meanwhile, a drawback of the latter families of electrolytes is their hygroscopicity. The battery cells containing these electrolytes need to be thermally and electrostatically insulated, especially for their use at very low temperatures, as the dielectric constant of the ferroelectric-electrolyte decreases substantially below 0ºC, conditioning the batteries' performance.

Cork is a natural product with extraordinary insulting properties; thermal and moisture insulation can be attained. Besides those attributes, the $(OH)^-$ groups available in suberin (~40%), lignin (22%), polysaccharides (cellulose and hemicellulose) (18%), extractable (15%) and other constituents of the cork [2] can attract the mobile Na^+ ions and facilitate ionic conduction permitting cork usage as separator in a battery cell.

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ID 60 - Direct growth of MoS2 nanostructures on carbon fiber yarns for supercapacitors

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Wearable electronics is nowadays a reality in enabling monitoring, sensing, and storing/harvesting systems. Fiber-based structures owing to the high specific surface area combined with suitable inorganic nanostructures, organic or polymeric materials have unique potentialities to respond either mechanical, thermal, chemical, electrical, or even optical stimulus. [1] When integrating functionality and wearability, these devices can reach high capacitance and enable unique characteristics in electrochemical devices such as batteries and supercapacitors.

Carbon-based materials present high electrical conductivity and chemical stability but limited charge storage capability. This property can be enhanced by functionalizing them with nanomaterials which offer improved ionic transport and electronic conductivity. [2]

Molybdenum disulfide (MoS2) is a 2D transition metal dichalcogenides (TMDs) that has been showing great promise in energy storage applications due to its versatile electronic structure arranged in three different phases, two semiconducting, 2H and 3R, and the metallic identified as 1T. [3]

Herein, we explore the direct growth of MoS2 on stretch-broken carbon fiber yarns (SBCFYs) and evaluate its electrochemical performance that gradually changes from the capacitive to pseudocapacitive by CV, EIS and GCD. The hybridization between SBCFYs and MoS2 displayed a specific capacitance of 133.86 Fg-1 at 5 mVs-1 and 164.3 Fg-1 at 1 Ag-1 and 80% retention after 3000 CV cycles.

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ID 104 - Transparent Niobium-doped titanium Dioxide Thin Films with high Seebeck coefficient for thermoelectric applications

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The design of a transparent thermoelectric material is a promising technology for touch–screen displays and solar cell applications, rendering a more sustainable powering of the device. In order to enhance the thermoelectric performance, the material must have a high Seebeck coefficient, high electrical conductivity but low thermal conductivity. Modifying the atomic structures of TiO_2 by deliberately introducing defects can enhance its properties to a great extent, while a cationic doping of TiO_2 has been documented to improve its electrical conductivity. This work reports the production and characterization of optically transparent Nb-doped TiO_2 thin films with enhanced thermoelectric properties deposited on glass and Si by reactive d.c. magnetron sputtering in high vacuum. Several process parameters, such as reactive and working gas flow rate, deposition temperature, target current density and post-annealing conditions, directly affect the morphology and crystalline structure of the thin films. The optimization of these parameters (gray area in Fig1.a) results in thin films with thickness of 120-300 nm, maximum average optical transmittance in the visible range of 73 %, n-type electrical resistivity of 0.05 W·cm, thermal conductivity below 1.7 $W \cdot m^{-1} \cdot K^{-1}$ and a maximum absolute Seebeck coefficient of 223 $mV \cdot K^{-1}$. The resulting maximum thermoelectric power factor and figure of merit is 60 $mW \cdot K^{-2} \cdot m^{-1}$ and 0.014, respectively. Hence, modifying the optical, electric, thermal and thermoelectric properties of the thin films enables their suitability for applications as transparent electrodes in photovoltaic systems and touch displays, amongst other devices (Fig1.b).

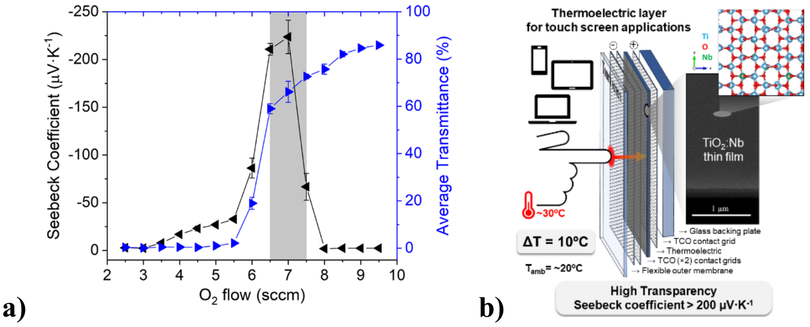

Fig1. (a) Seebeck coefficient and average transmittance (400-700 nm) in relation to the Oxygen flow rate during deposition; (b) thermoelectric layer implemented in a touch screen.

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ID 142 - Mechano-synthesis of an equimolar Li-Sn alloy

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Lithium-Tin (Li-Sn) alloys are attractive candidates for plasma facing components of fusion nuclear reactors. However, production of these alloys presents some challenges: the chemical reactivity of Li must be mitigated, requiring special conditions during processing to avoid sample contamination and the formation of high melting temperature Li-rich intermetallics. Mechano-synthesis (MS), via high energy milling, is an alternative method to produce prototype Li-Sn alloys, allowing access to a wider range of compositions and microstructures. MS has been demonstrated to be an effective technique for production of Li-Sn alloys with Li content up to 50 at.% [1,2]. Nevertheless, the predominant cold-welding behaviour of these alloys results in a low milling mass yield, and, due to the large number of parameters involved in this processing route, their effects on the final microstructure and composition of the alloys remain relatively understudied. In this work, the impact of the milling rate and milling ball diameter on the final microstructure and composition of an equimolar Li-Sn alloy was studied. The resultant microstructure was assessed in detail by a combination of scanning electron microscopy, ion beam analysis, X-ray diffraction and differential scanning calorimetry.

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ID 144 - The effect of smaller and larger ions on the hydrothermal synthesis of doped hydroxyapatite

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Calcium hydroxyapatite (Ca -HAp) is a promising material for the environmental management [1]. Due to its non-toxicity, biocompatibility, and occurrence in nature, it is considered an environmental-friendly material, while low water solubility, high adsorption capacity, thermal stability, and low-cost make it a perfect candidate for applications in environmental remediation [2]. To prepare adsorbents with desirable properties, it is essential to develop synthesis methods that would enable control of the product morphology.

The present work investigates the influence of divalent cations (Mn^{2+}, Mg^{2+}, Ba^{2+}, and Sr^{2+}) on the hydrolysis products of α-TCP. For this purpose, α-TCP, synthesized via wet precipitation method, was treated under varying hydrothermal conditions in the presence of varying concentrations of divalent cations.

It was revealed that both the hydrothermal synthesis conditions (temperature, time, concentration of the foreign ion), and the nature of the foreign ion affected the phase purity and morphology of the final products. Of all the ions investigated, Mn^{2+} had the highest, and Sr^{2+} – the lowest inhibiting effect on the hydrolysis process of α-TCP. Samples synthesized using larger concentrations of foreign cations (Fig. 1) contained not only plate-like, but also rod-like crystals. Under certain synthesis conditions, we were able to obtain samples consisting of rod-like particles only. The results of this study suggest that morphology of HAp could be controlled by applying certain concentrations of the foreign ions during the hydrothermal synthesis.

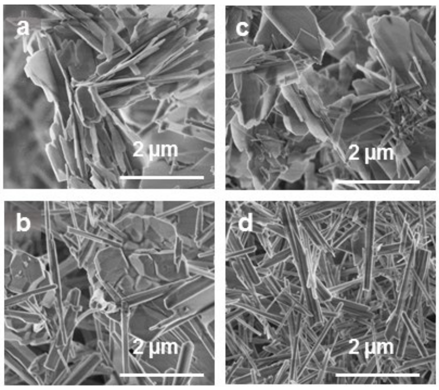

Figure 1. SEM images of the samples: a) 0.1 mol%Mg2+); b) 5 mol% Mg2+; c) 0.1 mol% Ba2+; d) 5 mol% Ba2+.

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ID 145 - Fully DFT large-scale pursue of singlet fission sensitizers

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The current silicon-based solar cells have been constantly improving their performance until efficiencies of around 25% were obtained,[1] close to its theoretical maximum efficiency. In order to overcome this limitation the singlet fission (SF) mechanism was proposed,[3,4] as it is able to generate two low-energy excitons upon the absorption of only one photon. A very specific alignment of the molecular orbitals is required for the obtainment of efficient singlet fission, which can be stated in two conditions:[3,5]

$C_1 = 2T_1 - S_1 \leq 0$ (1)
$C_2 = 2T_1 - T_2 < 0$ (2)

S_1 is the singlet excited state, T_1 is the first triplet state, and T_2 is the second excited triplet state.
Here it is presented a computational study in which the PUBCHEM database[6] is screened to select all species matching the energy requirements for singlet fission, and potentially unveiling new and untested SF candidates. The selected three-stepped protocol[7] uses the conditions (1) and (2) as the main criteria to select the species compatible with singlet fission. The level of theory is gradually increased as the number of remaining candidates diminishes. As a result, out of the initial 100 million species, 72 thousand molecules (0.07%) were found to match singlet fission energy conditions. The resulting set of molecules were structurally classified, considering also practical concerns such as availability or stability in order to further refine the screening.

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ID 171 - Dynamic Host-Guest Hydrate Fluid

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Host-guest hydrates crystalline solids, more commonly known as clathrates, in which a water framework generated at low temperature and high pressure can trap small non-polar molecules, are the great interest in gas separation, storage and transportation. However, most of their current investigations are limited to the study of the crystalline phase so the structure of their molten (liquid) phase has been overlooked. We have shown that a dynamic structure similar to clathrates which involves essentially all H_2O molecules of the solvent can be induced by the hydrophobic solvation of alkylammonium or phosphonium salts, triggering the formation of a unique, highly incompressible supramolecular liquid. It is demonstrated by electrochemical means that strong interaction among H_2O molecules in the tight structure of the supramolecular liquid limits their reactivity. Similarly, reactivity of anion (i. e. Cl-) in the structure is reduced which suggests their incorporation in the multi-polyhedral structure of hydrogen-bonded water. This work introduces the design a new family of supramolecular liquids, stable under atmospheric conditions, which can find important technological applications as solvents and electrochemical media.

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Presenting author: Carlos Herreros-Lucas, CIQUS, Centro de Investigación en Química Biolxóica e Materiais Moleculares, Universidade de Santiago de Compostela, 15782-Santiago de Compostela (Spain), carlosherreros@usc.es

ID 197 - Investigation of Nb doped NiO films for photovoltaic applications

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Nickel oxide (NiO) is a p-type oxide semiconductor and its conductivity is owned to non-stoichiometry due to defects nickel (Ni) vacancies and oxygen interstitials. Because of its p-type behavior, transparency (bandgap energy 3.1 - 3.8 eV) and combined with its high chemical stability, NiO is the most widely used p-type transparent conductive oxide [1, 2].

In this work, undoped NiO, Nitrogen (N) containing NiO and Niobium (Nb) doped NiO were fabricated by RF sputtering technique by employing Ni-Nb targets in plasma containing (Ar-O_2-N_2) gases in 5 mTorr pressure and 300 W power. The purpose of this investigation was to monitor of changes in films' properties due to Nb dopants. The oxide layers were characterized by SEM, EDX, AFM, XRD, XPS, Raman and UV-Vis-NIR spectroscopy.

The optimum films were used to form a transparent all-oxide p/n heterojunction diode and characterize it as UV solar cell. Compact (c-) and mesoporous (m-) TiO_2 films were fabricated by spin coating as the n-type layers, on FTO-covered glass substrates. The behavior of the $NiO/m\text{-}TiO_2/c\text{-}TiO_2/FTO$/glass heterodiode was characterized and analyzed in the dark (I-V) and under UV illumination (Fig.1).

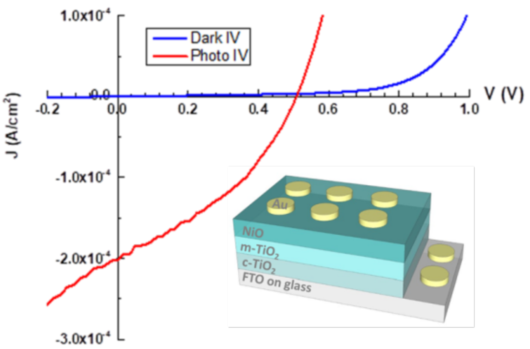

Figure 1: photo UV measurement of $NiO/mTiO_2/cTiO_2/FTO$/glass

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ID 221 - Si_3N_4-TiN substrate for BDD electrodes: from cutting tools and biomedical applications to water treatment

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Boron-doped diamond (BDD) thin films hold great promise for the future of water remediation, being able to completely mineralize persistent pollutants into CO_2 and water, requiring only electrical energy through electrochemical oxidation. However, the main challenges encountered in applying BDDs on a large scale relate to the substrate material. Electrode failure typically occurs due to BDD delamination from the substrate. Silicon nitride (Si_3N_4) ceramics are well-known for their excellent properties, such as high wear resistance, chemical stability, high hardness, high fracture toughness, and high elastic modulus, ideal for cutting tools and biomedical applications. By adding an adequate amount of titanium nitride (TiN) particles to a Si_3N_4 matrix, it is possible to produce an electroconductive Si_3N_4-TiN composite, which combines metal-like conductivity with the Si_3N_4 ceramic properties and high affinity for diamond film growth by Chemical Vapor Deposition (CVD). In this work, BDD films were deposited through Hot Filament CVD (HF-CVD) on a Si_3N_4-30vol% TiN substrate. The resulting BDD/Si_3N_4-TiN electrodes were evaluated, including microstructure analysis, diamond quality, conductivity, adhesion strength, and electrochemical properties. In addition, the performance in the electrochemical oxidation of pollutants was evaluated by choosing phenol as a model pollutant. The BDD/Si_3N_4-TiN electrodes could remove up to 98.1% of phenol after 5 hours of direct anodic oxidation of a spiked aqueous solution with 1 mM of phenol. The results indicate that the BDD/Si_3N_4-TiN electrodes have much potential and could be the solution for producing long service life BDD electrodes for large-scale water treatment by electrochemical oxidation processes, particularly if using electricity from renewable sources.

Acknowledgments
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A.6. Biomaterials and healthcare

ID 5 - Preliminary characterization of reinforced gelatin-chitosan scaffolds for tendon tissue engineering applications

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Gelatin is an attractive biopolymer for the production of scaffolds for tissue engineering due to its biocompatibility, non toxicity, and low cost. Chitosan, a derivative of chitin, is a widely available natural polymer that exhibits antimicrobial and biodegradable behavior, as well as suitable physico-chemical properties after crosslinking. When combined, gelatin and chitosan can produce biocompatible scaffolds with appropriate porosity and morphology for tissue engineering applications. However, these scaffolds often fall short of meeting the required mechanical properties for tendon engineering, such as tensile strength and failure load. Cellulose nanocrystals (CNCs), produced via the acid hydrolysis of cellulose, are an interesting alternative for reinforcing polymeric scaffolds. They possess an elevated length to width ratio, which grants them exceptionally high strength and stiffness, and exhibit no cytotoxicity or immunogenicity.

In this study, CNCs are incorporated into a gelatin-chitosan solution, and subsequently frozen via unidirectional freezing. This technique is well documented in its ability to produce aligned pore structures which resemble tendon's extracellular matrix, which is mainly composed of type I collagen fibrils. The resulting scaffolds are vacuum dried for 48 hours, crosslinked and characterized for porosity, pore size, elemental composition, compressive and tensile strength.

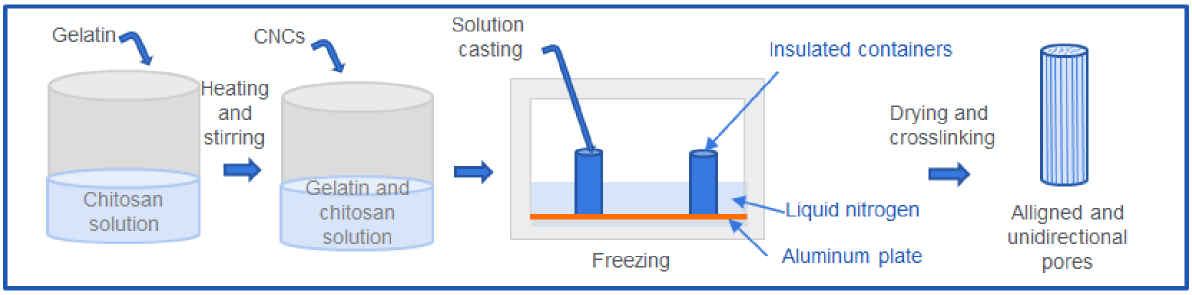

Figure 1 - Graphical abstract

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ID 20 - Tendon biomimetic 3D scaffold enhance amniotic epithelial stem cells biological potential

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Tendon tissue engineering represents an emerging field whose aim focuses on the design of 3D tendon biomimetic scaffolds that should ideally combine adequate physical, mechanical, biological and functional properties of the native tissue [2].

In this research, it was designed a bundle tendon-like PLGA 3D scaffold with highly aligned fibers on which the structure and mechanical properties were evaluated. Moreover, it was assessed scaffold's teno-differentiative and immuno-inductive ability on amniotic epithelial stem cells (AECs).

The fabricated PLGA 3D scaffolds mimic macroscopically and microscopically the structure of native tendon tissue and its biomechanical properties. Biologically, AECs seeded on the fabricated 3D scaffolds acquired a spindle tenocyte-like morphology after just 24h compared to the AECs cultured on petri dishes (CTR) which maintained their cobblestone morphology. The phenotypic change of the engineered AECs was also confirmed by visualizing TNMD protein expression, a mature tendon marker, within their cytoplasm and supported by the analysis of tendon-related genes (SCX, COL1, and TNMD) that were significantly upregulated at 7-day culture, while no TNMD protein expression or significant increase in tendon-related genes was found in CTR cells. Moreover, the 3D construct induced on AECs an upregulation of IL-10, an anti-inflammatory cytokine, maintaining basal levels of IL-12, a pro-inflammatory cytokine, showing a favorable IL10/IL12 ratio.

In conclusion, the fabricated PLGA 3D scaffolds are tendon biomimetic in terms of ultrastructure and biomechanics, making them also suitable for surgical purposes. Moreover, these constructs revealed a high teno- and immuno-inductive potential on AECs and thus represent potential candidates for tendon regeneration.

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ID 40 - Cu-based thin foils for antimicrobial applications: relationship between alloy composition and micromechanical properties

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Nowadays, Health-Care Associated Infections (HAI) are globally responsible for serious morbidity, increased costs and prolonged length of stay in healthcare structures. Frequently touched surfaces play an important role in the transmission chain, hence anticontamination strategies may help fighting HAIs. For some years now, Cu has been attracting widespread interest as anti-contamination material in hygiene-sensitive areas, due to its capacity for rapidly inactivating bacteria, yeasts and viruses, more than other widely used materials such as stainless steel and polymers. Thanks to a specific contact killing mechanism, Cu and Cu-based alloys may help fighting several pathogens such as Sars-Cov-2 and HAI-responsible antibiotic-resistant "superbugs". Within the next few years Cu is set to become an essential means to reduce the infection risk from high-touch surfaces. However, critical information about microstructure and micromechanical properties of Cu-based materials are neglected when solely evaluating antimicrobial capability. Therefore, the aim of this study is to develop Cu-based thin-rolled foils with a suitable tradeoff among workability, micromechanical and antimicrobial properties for high-touch fomites in hygiene applications, by optimizing alloy composition. Hence, Cu15Zn and Cu18Ni20Zn with foil thickness ranging from 15 to 25 µm were investigated, whilst PHC Cu (99.95%) was used as the benchmark. Microstructural characterization was carried out by optical and electronical microscopy (FEG-SEM), energy dispersive spectroscopy (EDS) while micromechanical properties were assessed by micro-hardness (HV), micro-scale abrasion (MSAT) and scratch tests. Subsequently, Cu-based foils were exposed to selected pathogens for different time points, evaluating their antimicrobial capability by quantitative Polymerase Chain Reaction (RT-PCR).

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ID 46 - Preparation of Alginate Films for Cell Seeding Studies: Challenges and Strategies for Improvement

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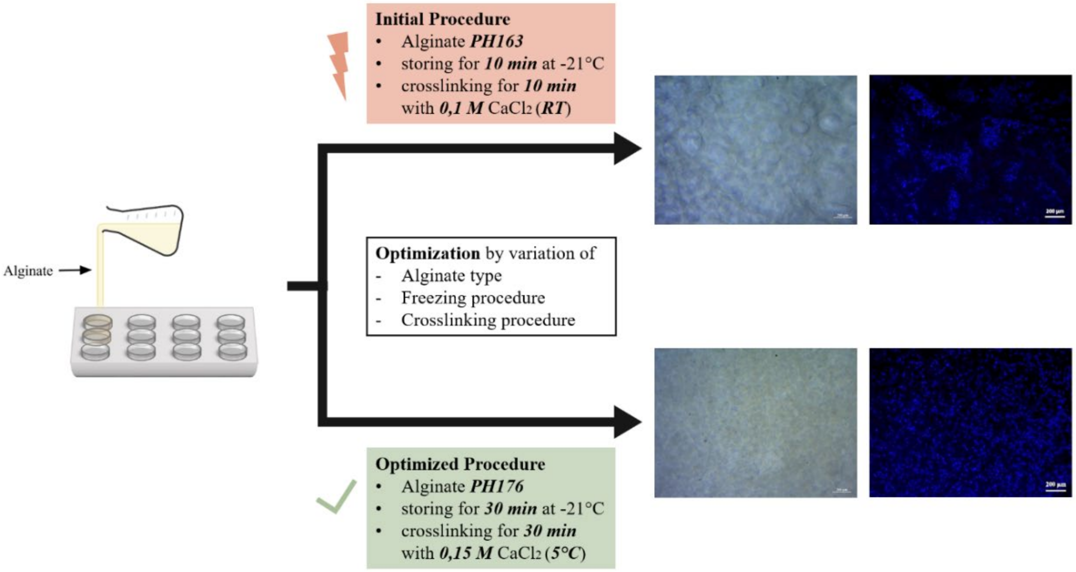
Alginate-based hydrogels show numerous promising properties, i.e. high biocompatibility, and therefore they can be used for soft tissue engineering. It is known that cells do not spread on alginate films but instead show weak attachment, which is why alginate is commonly used as a negative control group for cell seeding experiments. However, it was observed that cells were spreading on alginate films due to the unevenness and irregularity of casted hydrogel films. Therefore, the causes of these irregularities, which result in an inhomogeneous cell distribution and thus in cell spreading due to accumulation of cells in, e.g. cracks, were investigated. Consequently, an optimized protocol for the fabrication of planar 2D hydrogel films enabling a homogenous cell distribution was established by varying alginate types, freezing temperature and duration, concentration of crosslinking agent as well as crosslinking time. Besides the investigation of two-dimensional cell-material-interactions, the films are also characterized in terms of degradation behavior, chemical and mechanical properties as well as morphology. Furthermore, the alginate films are compared to oxidized-alginate gelatin (ADA-GEL) hydrogel films to investigate the influence of the incorporation of gelatin on the previously mentioned properties. The results of this study are of importance for conducting cell seeding studies with alginate-based hydrogels, e.g. ADA-PEG.



Initial Procedure
- Alginate ***PH163***
- storing for ***10 min*** at -21°C
- crosslinking for ***10 min*** with ***0,1 M*** $CaCl_2$ (***RT***)

Optimization by variation of
- Alginate type
- Freezing procedure
- Crosslinking procedure

Optimized Procedure
- Alginate ***PH176***
- storing for ***30 min*** at -21°C
- crosslinking for ***30 min*** with ***0,15 M*** $CaCl_2$ (***5°C***)

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ID 52 - Electrospinning of polycaprolactone-based hybrid nanofibrous scaffolds and their in vitro evaluation for tendon regeneration

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For an effective tissue regeneration, combinations of synthetic and natural polymers are explored to produce scaffolds that provide the requirements such as biocompatibility, biomimetic interface, mechanical properties close to native tissue and promotion of cell attachment and differentiation. The architecture and topography of the scaffolds influence the regeneration process as well as the selected materials. From previous studies, it is obvious that the scaffolds must have a porous structure to provide sufficient nutrient and waste exchange, and to support cell migration. In case of tendon tissue engineering, nanofibrous scaffolds (NS) can mimic the hierarchical collagen fiber structure of native tendon tissue and provide sufficient porosity simultaneously. As a tissue engineering approach, in this study, we aim to synthesize NSs which are made up of polycaprolactone (PCL), polycaprolactone/chitosan (PCL/C) and polycaprolactone/gelatin (PCL/G) in benign solvent systems and to evaluate their potential for tendon tissue regeneration. The electrospinning process parameters are tailored according to fiber diameter and morphology which should be optimized for cell attachment, differentiation and proliferation. The NSs are characterized in terms of fiber morphology and size, chemical composition, hydrophilicity, and biomechanical properties by tensile test measurement. Also, the biocompatibility of the scaffolds is tested in vitro by using stem cells to investigate the potential for cell proliferation and differentiation into tenocytes.

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ID 66 - Hydroxyapatite based bioceramic inserts in restorative dentistry: Fracture resistance of Class I restorations

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This study aimed to process and optimize the properties of bioceramic inserts based on hydroxyapatite, and evaluate the fracture resistance of large Class I restorations in extracted posterior teeth restored with the bioceramic inserts and commercially used restorative materials (CRM).

The hydrothermally obtained hydroxyapatite powder doped with magnesium ions (Mg-HAP) processed into cylindrical inserts ø 4.70 mm was optimized to have adequate mechanical properties and bonding ability with CRM. The extracted posterior teeth were divided in two groups and received Class I cavities restored by a) applying Mg-HAP inserts with CRM and b) applying CRM only, as per the conventional clinical protocol. The teeth were subjected to a compressive axial loading using a 3 mm diameter rod in a universal testing machine. The results showed mechanical properties and bonding ability with CRM similar to dentin, and no statistically significant difference in fracture resistance between the proposed protocol with Mg-HAP inserts and conventionally restored teeth with CRM. Application of the bioceramic inserts could improve the lifespan by the reduction of polymerization shrinkage without jeopardizing the fracture resistance of the restoration. This finding makes Mg-HAP bioceramic insert a promising dentin substitute for large cavity restorations in dentistry.

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ID 87 - Biocompatibility of C-based Sputtered Coatings in Orthodontic

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Biocompatibility is the key property for materials employed in medical applications to assure their safe humane usage. In orthodontics, the malocclusions are treated using fixed metallic appliances, such as Nitinol (47-52% Ni) and Stainless Steels (8-12% Ni and 17 22% Cr), due to the combination of favorable mechanical properties and spontaneous passivation in corrosive environments. Nevertheless, the oral cavity – the "portal entry of the human body" [1], with especially intense corrosive properties – makes all bioalloys unable to resist corrosion which could compromise their biocompatibility. Indeed, biofilm accumulation and microbiologically induced corrosion were clearly found on retrieved brackets and archwires after clinical use [2]. The released corrosion products/metal ions may accumulate in the surrounding tissues and induce allergic reactions. Ni (and its compounds), typically present in orthodontic alloys, is the most noticeable element: a strong immunologic sensitizer, capable of triggering cytotoxic and mutagenic effects, with high carcinogenic potential. Thus, surface modification to obtain protective coatings is one of the key solutions for orthodontic alloys, particularly coatings based on the main element of live: carbon, the well-known *diamond-like carbon* (DLC) coatings. In this class, the hydrogenated amorphous carbon (a-C:H) stands out. Previous studies [3-5] suggested that a-C:H-based coatings deposited on 310 and 316L biomedical AISI steel grades remained chemically inert in simulated oral fluids (artificial saliva). In this research, the cytotoxicity of a-C:H coatings is studied with mono- and co-cultures of fibroblasts (3T3) (ATCC American Type Culture Collection) and naïve macrophages using the MTT [3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide] assay, along with complementary characterization techniques.

Keywords: Cytotoxicity, DLC, Sputtering, Orthodontics.

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ID 109 - Fabrication and preliminary characterization of electrospun poly(glycerol sebacate)-based nanofibrous scaffolds for tendon tissue engineering

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The electrospinning technique is a versatile and widely employed tissue engineering approach to fabricate tailored nanofibrous scaffolds which mimic the three-dimensional architecture and topography of the extracellular matrix of the target tissue. For tendon regeneration in particular, the electrospinning technique allows for the reproduction of the highly hierarchical structure which native tendons are made of, consisting of aligned bundles of collagen fibrils. Poly(glycerol sebacate) (PGS) is a biodegradable polyester with flexible elastomeric properties, whose synthesis can be easily carried out through the polycondensation reaction of glycerol and sebacic acid. Its tunable mechanical properties, bioresorbability and lack of immunogenicity, accounts for its already documented use as a component for scaffolds aimed at soft tissue engineering applications, although its specific incorporation in scaffolds for tendon regeneration remains mostly unexplored compared to the most common synthetic polymers studied in said field. In this study, we aim to fabricate mildly crosslinked PGS-based nanofibrous scaffolds through the electrospinning technique in a benign solvent system, to optimize their production process, and to characterize the morphology and size of the fibers, the chemical composition of the scaffolds, their hydrophilicity, their mechanical properties through tensile testing, and their degradation behavior. Moreover, preliminary biological *in vitro* tests are aimed at evaluating the biocompatibility of the scaffolds for their potential use in tendon tissue engineering.

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ID 116 - A Hybrid System Combining Patterned Ti-based Bulk Metallic Glass and Biopolymer Coating for Implant Applications

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Titanium-based bulk metallic glasses (BMGs) have specific mechanical properties and superior biocompatibility making them promising materials for hard-tissue implant applications such as prosthesis, fixation, and dental implants.1,2 Bacterial adhesion and biofilm formation on implant surfaces are considered the main risks for implant-associated infections which lead to the failure of medical treatments.3 Antibiotics are commonly used to handle implant-related infections, yet the overuse of antibiotics has caused the rapid appearance of antibiotic-resistant bacteria.4 Hence, the purpose of this work is to develop Ti-based implant materials with enhanced antibacterial properties. Biocompatible Ti-based BMG (without toxic elements such as Ni, Al and Be) were cast and further processed to create micro-/nano-patterned surface features by thermoplastic net-shaping (TPN) technique under the supercooled liquid region (SCLR).5,6 The patterned surface features aiming to promote osseointegration were characterized by scanning electron microscopy and X-ray diffraction. Furthermore, polyethylene glycol (PEG) functionalized biopolymers were proven to enhance the biocompatibility and anti-biofilm properties of biomedical materials.7 Therefore, in this research, PEG-based biopolymers were spin-coated on the patterned surface of Ti40Zr10Cu34Pd14Sn2 BMG and then annealed under optimized temperatures and times to induce self-organizing hierarchical structures which were characterized by polarized light and confocal laser microscopy. The antibacterial properties were examined to test for effects on the viability of different bacterial species. This hybrid system aims to inhibit biofilm formation and promote osseointegration for hard-tissue implant applications.

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ID 133 - Zinc Whitlockite ($Ca_{18}Zn_2(HPO_4)_2(PO_4)_{12}$): Dissolution-Precipitation Synthesis, Structural and Morphological Features

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Calcium phosphates (CPs) are widely used in medicine for the regeneration of hard tissues due to their structural and compositional similarity to natural bone. In recent years, there is a highly increased interest in CP additionally containing Mg^{2+} ions – magnesium whitlockite (Mg-WH, $Ca_{18}Mg_2(HPO_4)_2(PO_4)_{12}$), which is believed to be a promising candidate in the context of bone repair biomaterials [1].
Despite the fact that first row transition metal (TM) ions are of comparable size with Mg^{2+} ions, there is just a couple of studies on materials with WH crystal structure, where Mg is replaced by other divalent cation. In our recent study we reported for the first time on the feasibility of the formation of zinc whitlockite (Zn-WH, $Ca_{18}Zn_2(HPO_4)_2(PO_4)_{12}$) – Zn containing analog of Mg-WH [2].
The main goal of the present work is to investigate the peculiarities of phase formation and structural properties of Zn-WH as a potential non-investigated CP for biomedical applications. Zinc was chosen as an element of interest to replace Mg^{2+} ions due to several reasons. Firstly, the ionic radius of Zn^{2+} in six-fold coordination (0.74 pm) is very similar to that of Mg (0.72 pm). Secondly, Zn is an essential element, which is involved in many biological processes, its highest content in human body is observed exactly in hard tissues. Moreover, Zn is relatively harmless element, which means that its content in bone substitutes can be relatively high. Finally, other Zn containing CPs are known for their superior biological performance and antibacterial properties.

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ID 135 - Novel biodegradable Mg-Y and Mg-Y-Li alloys

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This work is focused on the investigation of two Mg-based alloys, Mg-Y and Mg-Li-Y, as potentially new materials for biodegradable applications. Both alloys were processed by equal channel angular pressing, and the effects of yttrium or lithium addition on the microstructure, corrosion resistance, in vitro degradation in biological media, cytotoxicity, and mechanical properties were investigated. The commercially well-known Mg-Y-RE-Zr (WE-type) alloy was processed similarly and used as a benchmark in all tests. The WE-type magnesium alloy provides a satisfactory degradation rate in conjunction with mechanical strength in the field of temporary implantable devices. Microstructure evolution was analysed by scanning and transmission electron microscope and electron backscatter diffraction. The mechanical properties of the investigated alloys were tested by compression deformation tests. The initial corrosion resistance in the salt solution was examined by the electrochemical impedance spectroscopy method and the degradation rate in the biological media was determined employing immersion tests.

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ID 140 - Tuning Cu Ion Release from Pd Bulk Metallic Glass Composites

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The combination of high mechanical strength, corrosion resistance and thermoformability makes precious metal-based bulk metallic glasses (BMGs) particularly attractive for a variety of medical applications (e.g., implants). Antibacterial or antibiofilm properties are a critical requirement for new biomaterials for such applications as device-related infections are a substantial problem that strains the health system [1]. $Pd_{43}Cu_{27}Ni_{10}P_{20}$ (Pd-BMG) is a biocompatible BMG former [2] with one of the lowest critical cooling rates [3], making it easier to use for medical device manufacturing than most other BMGs. Additionally, it also contains Cu which is known for its antibacterial properties. In this work, we create Pd bulk metallic glass composites (Pd-BMGCs) with various crystal fractions and exploit its complex microstructure as a mean to tune its antibacterial properties. We identify three different crystalline structures in the Pd-BMGC and link their amount, structure, and composition with the Cu ion release in vitro. Furthermore, we identify aspects that control the appearance of certain crystalline structures over others.

This work was financed via the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No. 861046.

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ID 154 - Electrophoretic deposition, heat treatment and characterization of HA/ZnS/S-PEEK coatings on the Zr-2.5Nb alloy

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Zirconium alloys are metallic biomaterials widely used for bone implants due to their favorable usage properties. Although excellent biocompatibility Zr-2.5Nb has poor osteointegration and tends to easily form a biofilm on the surface. Therefore bioactive and antibacterial coatings are often needed. The aim of this study was to develop sulfonated polyetheretherketone (S-PEEK)-based coatings incorporating antibacterial ZnS and bioactive nano-hydroxyapatite (HA) particles on the Zr-2.5Nb zirconium alloy substrates by electrophoretic deposition (EPD) and post heat treatment. The heating of the coated alloy at a temperature of 450 ºC and slow cooling resulted in thermal sulfonation of PEEK and coating densification. Suspensions containing 30 g/L of PEEK 704, 0.4 g/L of ZnS, and 1 g/L of HA powders in ethanol and chitosan polyelectrolyte in the contents of 95 and 5 vol%, respectively were used for coatings deposition. Macroscopically homogeneous coatings were deposited at 90 V during 30 s. The zeta potential of suspensions and deposition kinetics were investigated. SEM investigation revealed that heat-treated coatings were continuous and relatively homogenous. XRD and FTIR analysis revealed sulfonation of PEEK as a result of heat treatment. It was found that the HA/ZnS/S-PEEK coatings had greater roughness, hardness, modulus of elasticity and adhesion strength in comparison to the ZnS/S-PEEK coatings. Both coatings improved the electrochemical corrosion resistance of the alloy. Moreover, the HA/ZnS/S-PEEK coating showed apatite forming capability during soaking in simulated body fluid. This work represents an innovative approach to the fabrication of advanced sulfonated PEEK coatings containing bioactive and antibacterial agents for enhancing usage properties of zirconium alloys in medical applications.

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ID 201 - Synthesis and characterization of magnesium based bulk metallic glass composite prepared by high pressure torsion

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Fabrication of BMG composites has been regarded as a useful way to improve the plasticity of the monolithic BMG and therefore expand its potential application as engineering materials.
The present study aims to fabricate bulk Ca-Mg based composites by applying severe plastic deformation (SPD) via high-pressure torsion (HPT) on two metallic glasses 1) $Mg_{72}Ca_{12}Zn_{16}$ and 2) $Mg_{72}Ca_{12}Zn_{14}Sn_2$ (at. %). HPT allowed to consolidate and plastically deform amorphous alloy (1) and partially crystalline alloy (2) into the bulk component for the first time. Methods of X-ray diffraction, differential mechanical and differential scanning analyses, as well as optical and scanning electron microscopy were carried out to characterize the consolidated samples. Furthre more, mechanical properties like micro-hardness and elastic modulus were also studied.

Keywords: Ca-Mg based metallic glasses, high pressure torsion, mechanical properties.

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ID 208 - Advanced electromechanical devices for use in bone tissue engineering

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Bone lesions and diseases are becoming more prevalent, increasing the need for implants. Nowadays most of the implants are made of metallic materials that often suffice the structural and mechanical needs but are often lacking in biological properties. And here is a particular focus on improvement of the biological properties of metallic implants, increasing their bioactivity and response to biological stimuli. This can be achieved using physical, chemical, structural, or surface modifications of metallic materials. These modifications try to mimic natural properties of the bone including the electromechanically assisted potential, which plays an important role in the bone regeneration process. Bioactive polymer material Poly (L-Lactic acid) (PLLA) can be used to coat the metallic implants thanks to its piezoelectric properties. The piezoelectric properties of PLLA in turn are highly dependent on crystallinity degree. This work focused on studying the effect of the different surface treatments applied to the metal and different parameters used during PLLA crystallization that can have an impact on the crystallinity degree of the PLLA. For this purpose, PLLA solution of different concentrations (2.5 and 5.0 wt.%) was deposited on 316L Stainless Steel (previously subjected to a thermal or UV treatment) via spin coating forming a thin PLLA film. The films were then crystallized using a thermal plate or Linkam stage chamber using different cooling rates and different temperatures. The results showed that UV treatment, with a lower cooling rate, seems to drastically increase the crystallinity of PLLA. A significant differences in samples' morphology according to the surface treatment and parameters used were observed.

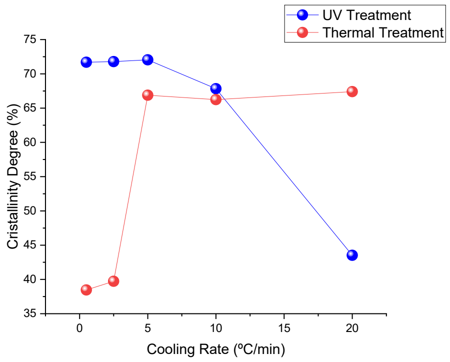

Figure 1. Crystallinity degree as a function of the cooling rate for samples subjected to either UV or thermal treatment.

Acknowledgments: This work was done in the scope of the project "Advanced BioMEMs for tissue engineering: applications in hard tissue (BioMEMs)", POCI-01-0145- FEDER-032095. The authors thank CICECO-Aveiro Institute of Materials, UIDB/50011/2020, UIDP/50011/2020 & LA/P/0006/2020, financed by national funds through the FCT/MEC (PIDDAC).

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ID 227 - Novel L-alanine and glycine based poly(ester amide)s: assessment on their thermal processability and physico-chemical properties

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α-Amino acid based poly(ester amide)s (AAA-PEAs), which combine the biocompatibility and biodegradability of polyesters with the good mechanical properties of polyamides, are polymers whose importance in the biomedical field has increased considerably in recent years. The versatility of the structures obtained, allowing fine-tuning of physicochemical properties (e.g., hydrophilicity, stiffness, degradability) to specific applications, is clearly the main advantage of these materials. In addition, the presence of α-amino acids enables the enhancement of cell-material interactions, thus increasing cell adhesion and cell integrity. Despite the advantages and properties of AAA-PEAs, their thermal processing, which could open new avenues in the field of additive manufacturing, has not been widely explored.

In this work, AAA-PEAs based on L-alanine (PEA-ala) and L-alanine/glycine (PEA-ala-gly (90:10) and PEA-ala-gly (75:25)) were prepared by solution polycondensation. The intended application for these PEAs is the regeneration of articular cartilage (AC), so glycine and *L*-alanine were selected as AAAs. These AAAs are present in greater amounts in the structure of collagen, which is the most abundant protein in AC. After obtaining the PEAs, they were hot pressed and films were prepared. These were characterized in terms of swelling ability, *in vitro* degradability and thermomechanical properties.

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ID 230 - Hyaluronic acid-based chemically crosslinked hydrogels via thiol-ene Michael addition reaction

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Hydrogels are three-dimensional crosslinked networks able to absorb large quantities of water or body fluids. These materials can be obtained by their *in situ* gelation. Hyaluronic acid (HA) is a natural polysaccharide widely used in hydrogels. However, HA presents low mechanical strength and *in vivo* stability, as it is easily degraded by free radicals or enzymatically degraded by hyaluronidase. Free carboxylic acid and hydroxyl groups of HA can be chemically modified, being then able to participate in crosslinking reactions. Michael addition-based click reactions have attracted much attention for chemically crosslinked hydrogel applications since they can occur efficiently at physiological conditions with minimal by-product formation. Thiol-ene Michael addition consists of the reaction between deprotonated thiol groups and electron-deficient double bonds.

In this work, HA-based hydrogels were obtained from the Michael addition between double-bond and thiol modified HA. The HA-based hydrogels were prepared by mixing both thiol and double bond-modified HA dissolved in a phosphate buffered saline (PBS) solutions (pH 7.4), at 37 ºC, mimicking the physiological conditions.

This project is supported by Portugal 2020 through the incentive system for business R&D (Inject4Pain, project nº 45201).

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ID 232 - Development of Polymeric Microparticles as Tailored Drug Delivery Systems for Co-Encapsulated Drugs

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In the past years, there has been an increasing effort to develop drug delivery systems that could help overcome drug limitations, such as low solubility, poor bioavailability and instability to external factors that can compromise an efficient drug delivery. An example of such strategies is the preparation of versatile polymeric microparticles, such as the ones presented in this work.

Through the well-known oil-in-water emulsion method, we prepared biodegradable polymeric microparticles, co-encapsulating two active pharmaceutical ingredients (APIs). These APIs were selected due to synergistic therapeutic effects when combined. Even though they present distinct physicochemical characteristics, we were able to encapsulate both of them within the microparticles, as confirmed by HPLC-PDA analysis. The biodegradable nature of the polymeric microparticles was given by a tailor-made polymer, assuring thus the biocompatibility of the final product, and the concomitant extended release of the encapsulated drugs.

Changes in the microparticle formulation were made according to a Quality by Design (QbD) approach that allowed a better understanding of the process and an adjustment of the particle size distribution (PSD) and drug content. Further *in vitro* release studies showed an extended release profile for both drugs, aiming at a more convenient administration and a better treatment efficiency, able to fit different applications.

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A.7. Sensors & Actuators

ID 155 - Fully-Printed Single Channel P(VDF-TrFE) Transducer for Ultrasound

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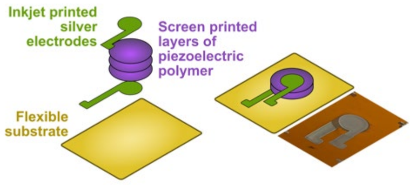
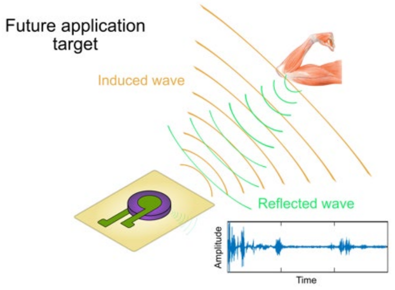
Ultrasound (US) transducers represent an emerging class of wearable sensors. The key component of a US transducer is a piezoelectric crystal which converts an electrical signal into mechanical oscillations to generate US waves that propagate deep into the tissues and converts the echoes back. Our work investigates the effects of different processing techniques on deposited piezoelectric fluorinated polymer films. The organic flexible ultrasound transducer was designed and fabricated. This work is focused on the analysis of the micro- and macrostructure of the printed layers and on the fabrication of the US transducer and study of the processing parameters on the resonance frequency.
The main component of the US transducer is a P(VDF-TrFE) which is a semicrystalline piezoelectric polymer. In this work a layer-by-layer screen-printing deposition of piezoelectric polymer films was performed. The thickness of the film was determined by the number of printed layers and the mesh count of the used screen and was measured with contact and non-contact methods. The obtained data revealed the dependence of resonance frequency of the transducer from thickness and roughness of the functional layer. The effect of postprocessing (thermal, polarization) on the piezoelectric properties of printed films was profoundly studied. Printed films were characterized with XRD, impedance spectroscopy and AFM and matched with values of remnant polarization to understand the contribution of every parameter. A proof-of-concept application of the fabricated transducer on an ultrasound A-mode line in a water phantom was demonstrated.





Presenting author: Kirill Keller, Institute of SolidState Physics, Graz University of Technology, Petersgasse 16, 8010 Graz, Austria, keller@tugraz.at

ID 161 - Modeling and state estimation for the advanced control of a multimaterial shape memory based actuator

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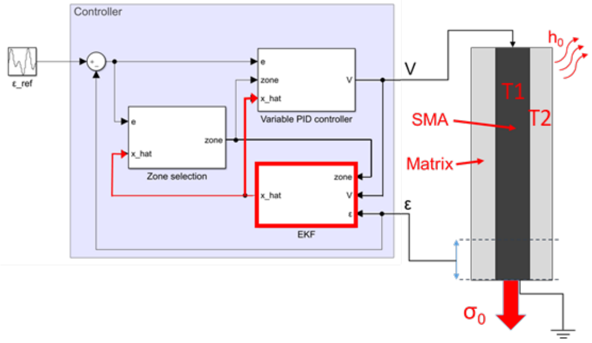
3. Polytechnic of Milan, Dipartimento di Elettronica, Informazione e Bioingegneria (DEIB), Milano, Italy

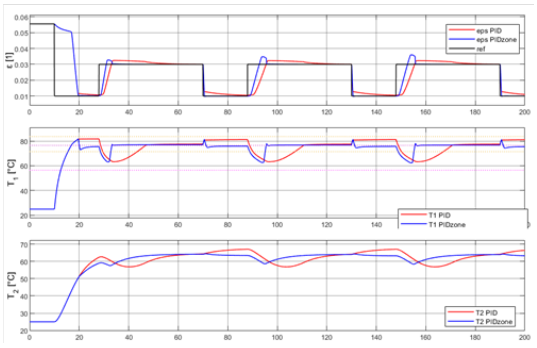
Shape Memory Alloys (SMAs) are a class of smart materials with interesting thermomechanical properties, such as the ability to recover very large deformations when heated. A peculiar application of SMA actuators is soft robotics, where a SMA wire can be embedded inside a soft material, such as a polymeric matrix [1].

In this work, we expand the SMA model proposed in the literature by Brinson [2] so as to develop a new thermomechanical multimaterial model of a SMA-based actuator made by a SMA wire coated by a silicone rubber layer. The system is highly nonlinear, time-varying and hysteretic, adding to the complexity of controlling actuation. We improved the controllability of the multi-material by implementing a new switched controller concept in the form of a Variable PID. Its parameters are tuned based on the changing SMA operating zone, which is determined via a state estimator and a logical block. Key to the switched controller implementation is the development of an Extended Kalman Filter (EKF) for state estimation. The EKF was tested and showed robustness in predicting the state.

Preliminary simulations of the Variable PID show that the innovative strategy allows for faster heating and cooling in the reaching some reference position compared to a conventional PID.

This control method could be used to reduce the requested electrical power and improve the overall material performance.





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B.2. Advanced steels and cast irons

ID 212 – Effect of controlled thermomechanical heat treatment of nanobainitic steel on the microstructure and mechanical properties

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Nanobainitic steel is very popular in recent years because of its excellent mechanical properties and wide application in the automotive or armor industry. Unfortunately, these properties can be obtained only by low-temperature isothermal heat treatment, which lasts from a few hours to a few days. Hence, the controlled thermomechanical heat treatment was applied in the order to shorten the time of this treatment to a few minutes. The high-carbon nanobainitic steel with the following chemical compositions: Fe-0.78C-2.45Mn-1.67Si-1.35Cr-0.21Mo-1.30Al (wt.%) was obtained through controlled thermomechanical heat treatment, which consists of rolling in the range of austenitization, and isothermal transformation at 190°C for 30 min during which in the first stage the stress below the yield strength of parent austenite for 10s was applied. The observations of microstructure using an optical microscope, scanning electron microscope (SEM), and transmission electron microscope (TEM) was carried out. These results confirmed that the microstructure of experimental steel mainly consists of a thin plate of bainitic ferrite and films of retained austenite with a size below 100 nm. The synchrotron X-ray results lead to determining the amount of retained austenite and their carbon concentration. The high mechanical properties of experimental nanobainitic steel, such as Rm=1862 MPa, and YS=1287 MPa were achieved mainly due to nano-metric plates of bainitic ferrite and its high dislocation density. Simultaneously, the high level of elongation, which is 11.8 % was achieved because the TRIP and TWIP effects that take place during tensile deformation were noticed.

Acknowledgments
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B.3. High-performance alloys and intermetallics

ID 35 - Influence of oxygen on the structure of orthorhombic martensitic phase in titanium alloys

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Metastable β titanium alloys with increased oxygen content represent an attractive, but not yet a completely explored area of titanium alloys. Their main advantage over conventional alloy Ti-6Al-4V (wt%), used for the manufacturing of joint implants, is high strength and very low modulus of elasticity. When choosing the right concentration of β stabilizing elements, various phenomena associated with the martensitic transformation β → α'' can occur, such as superelasticity, shape memory effect, or transformation-induced plasticity (TRIP).

In this work, we aimed to systematically study the influence of oxygen and β stabilizing elements (Nb, Zr) on the crystal structure of the martensitic phase α'' arising during quenching or deformation. The main emphasis was given to the investigation of the relative displacement of planes (011)β in the original bcc structure depending on a wide range of oxygen concentrations.

In the studied Ti-Nb-O and Ti-Nb-Zr-O systems, the proportion of Nb and oxygen was optimized to achieve the formation of the α'' phase during quenching from the area above the β transus. The presence of the α'' phase was detected by scanning electron microscopy. The structural parameters (lattice constants, relative displacement of the planes equivalent to (011)β in the bcc structure) were measured by the X-ray powder diffraction method and Rietveld refinement.

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ID 53 - Spatially resolved investigation of irradiation damage and hardening on proton irradiated Zirconium alloys

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[3] *Deutsches Elektronen-Synchrotron (DESY), Hamburg, Germany*

Inside the nuclear reactor, neutrons interact with zirconium atoms during nuclear chain reaction, producing collision cascades which result in irradiation damage. This is observed in the microstructure as dislocation loop formation, chemical segregation of solute elements and dissolution of precipitates. Protons are increasingly used to emulate neutron irradiation for studying irradiation damage due to their lower cost, shorter irradiation times, better accessibility, and similar resulting damage structures to neutron irradiation. In contrast to neutron irradiation, protons produce a damage profile which varies with penetration depth (Fig. 1), providing an opportunity to study damage evolution and its resulting effects. Zirconium samples have been proton irradiated to 0.1 dpa and the induced microstructural changes have been investigated by nanoindentation and micro beam synchrotron X-ray techniques. Both techniques were applied from the cross sections of the samples perpendicular to the irradiation direction. Depth profilings in 30 μm deep irradiated regions with X-rays were carried out for line profile analysis leading to dislocation density determination. Furthermore, hardening and crystal orientation relation was investigated using EBSD (Electron Backscatter Diffraction) mapping technique which reveals the impact of irradiation on mechanical properties. Here we show that the dislocation density and irradiation induced hardening follow the SRIM (Stopping and Range of Ions in Matter) calculated dose profile where they both increase towards peak dpa depth before dropping to non-irradiated region levels. Combining the two spatially resolved techniques has enabled to draw a better picture of the damage evolution at the early stages of irradiation.

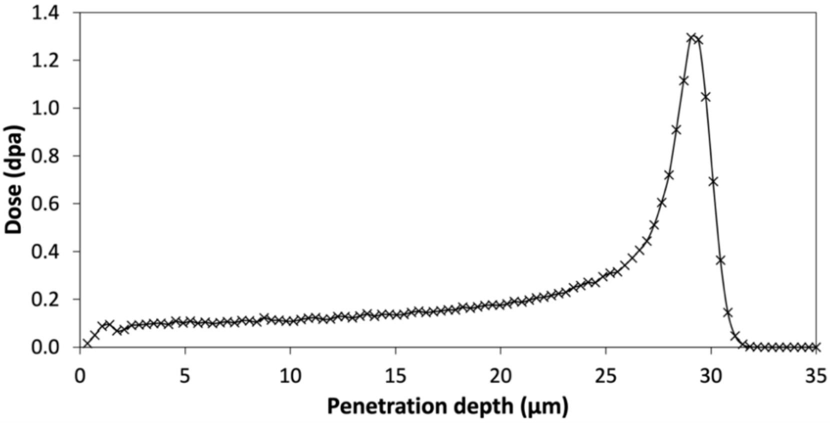

Fig. 1: Proton irradiation damage profile generated by using SRIM software package assuming 40 eV displacement damage for Zr with 2 MeV protons.

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ID 158 - Mechanical and microstructural effects of new thermomechanical treatments to boost damping of Beta III titanium

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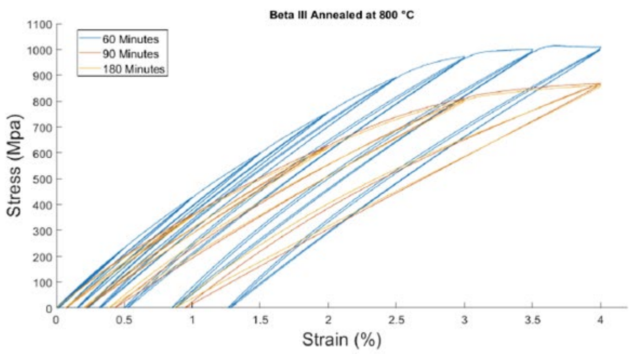
Metastable beta (β) Titanium alloys are a class of metallic materials, which find application in the aerospace field due to their high yield strength and low density [1]. Some compositions in this class of materials also display a pseudoelastic behaviour [2]. The presence of a hysteresis area in the stress-strain response, and the associated energy loss, could, when combined with optimal strength, prove to be of interest in structural applications where intensely loaded elements are subjected to vibrations, impulsive or dynamic forces in general.
The present work aims to provide a better understanding of the effects of microstructural modifications on the damping properties of Beta III titanium alloy (Ti-11.5Mo-6Zr-4.5Sn). We investigated the effects of annealing treatments followed by plastic deformation. Microstructural changes were observed through X-ray diffraction, optical (*Fig.1 Right*) and electron microscopy. The damping behaviour of the material in terms of hysteresis and internal friction response was evaluated by quasi-static tensile tests (*Fig.1 Left*) and dynamic mechanical analysis (DMA).
Through heat treatment at 800°C for 3 hours followed by plastic deformation to 16%, we obtained up to 75.55% increase in hysteresis in quasi-static conditions with good cycle stability, as well as an improved damping capacity in dynamic conditions, especially for 1-2% strain amplitudes. Microstructural analysis results also suggest that, differently from the conclusions of previous works [3,4], the main phenomena underlying the observed increase in damping capacity are likely connected to the reversible motion of semi-constrained structures, namely twin boundaries interacting with dislocations, without the involvement of martensitic transformations.



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ID 202 - Effect of rare earth elements on microstructure and oxidation resistance of γ-γ′ Co-based superalloys

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The γ-γ′ Co-based superalloys are promising heat-resistant alloys. They exhibit high operating temperature and mechanical properties at elevated temperature, which are much higher compared to conventional cobalt superalloys reinforced with carbides. The studies related to the development of this group of materials are focused on increasing the operating temperature, mechanical properties and oxidation resistance, which is much lower compared to that of nickel-based superalloys. One way of influencing the properties is addition of rare earth elements (REE). In this study, the influence of minor REE addition (La, Nd, Ce, Dy) on the microstructure and high temperature oxidation resistance was studied.

The alloys Co-9Al-9W-0.1REE were prepared via vacuum induction melting (VIM) and gravity casting. The heat treatment including solutionizing and aging was performed to obtain the γ–γ′ microstructure. The alloys were subjected to high temperature oxidation at 800 and 900 °C. The microstructure and oxidation products were analyzed via scanning electron microscopy (SEM).

The microstructure of the REE-doped alloys is composed of γ-γ′ phases and Co-REE precipitates enriched in Al. The addition of rare earth elements may improve high temperature oxidation of Co-based alloys.

This work is financed from the budgetary funds for science for the years 2018–2022, as a research project within the Diamond Grant programme (0069/DIA/2018/47).

ID 205 - Characterization and properties of dilute twin-roll cast materials based on the AA2195 aluminum alloy

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AA2195 alloy is a third generation Al-Cu-Li based alloy. These alloys contain less Li and more Cu to facilitate formation of ternary phase strengthening particles. Al-Cu-Li alloys are high strength and low-density materials used in the aerospace industry for structural components. A standard AA2195 alloy is direct-chill cast and subsequently processed and rolled into its final shape. A continuous casting method, such as twin-roll casting, could provide a suitable alternative where the material is meant to be used as a thin sheet, as the as-cast material already comes in a near-net shape. Two dilute AA2195-based materials were studied – one with the addition of Sc and one Sc-free. The effect of Sc addition – a powerful grain refining element in Al alloys – on microstructure and mechanical properties was identified. The proper thermomechanical processing of this class of twin-roll cast materials is currently unknown. However, if well processed, they could reach comparable or better properties than direct-chill cast alloys.

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B.4. High entropy alloys

ID 22 - Atomic-scale modeling of diffusion in concentrated alloys

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Experimental diffusion studies are only feasible at high temperatures and in most systems, it is not possible to measure the full Onsager matrix. It is therefore necessary to model diffusion from the atomic scale. The problem is mostly solved in pure and dilute alloys (e.g. [1]) but remains a challenge in concentrated alloys due to the vastness and complexity of the configuration space. In this work, we aim at extending the kinetic cluster expansion formalism to concentrated alloys in order to get an efficient evaluation of transport coefficients in concentrated alloys based on atomic-scale data. Our formalism is based on the Self-Consistent Mean Field Theory [2] and is implemented in the open-source KineCluE code. We treat the configuration exactly in the vicinity of diffusing species, while further away atoms are replaced by a homogeneous mean-field (Fig. 1). This approach provides an adjustable balance between constructing precise kinetic paths and reducing computational load to keep calculations feasible. The advantage of our model is that it can be applied to any concentrated alloy, e.g. high entropy alloys. We will show how our model performs compared with previous theories, Monte Carlo simulations and experimental studies.

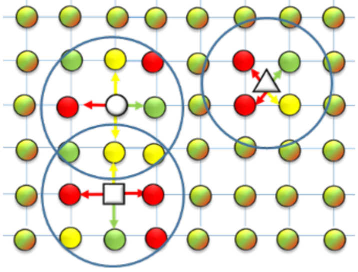

Fig. 1: Schematic representation of our model in the case of a ternary alloy (green, yellow and red) with 3 diffusing species (white). Each diffusing species is surrounded by an "environment" whose limits are drawn in blue and by mean-field atoms (mixed colors).

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[2] M. Nastar, Philosophical Magazine A (2000)

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ID 128 - The designing of concentrated complex alloys new (CCA) for light armor applications

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The candidate materials for armoring military land vehicles require excellent mechanical strength with high deformation capacity. Steels are the materials mainly used for these applications. However, the range of materials used to meet this need is limited, since they are mainly low-carbon, low-alloy steels. This results in a relatively high density of alloys, which opposes the problems of mass reduction and energy saving. In this context, the search for new metallic material solutions with higher specific properties is a necessity.

In our project, we propose the design of concentrated complex alloys (CCA) for light armor applications. Recent studies on CCA compositions, particularly the Co-Cr-Fe-Ni system, have shown a good compromise between mechanical strength, hardness, and ductility at low temperature [1]. To improve this mechanical resistance, different reinforcement strategies are studied. Among the commonly used, the one leading to a reinforcement of the face-centered cubic matrix by a controlled precipitation of an intermetallic phase of L12 and/or B2 type by addition of minority elements (Ti, Al, Mo, or Ta) is envisaged [2][3].

Thus, the first phase of the work consists of performing thermodynamic calculations to screen the Al-Co-Cr-Fe-Ni-Ti system and identify alloy compositions with the desired microstructural characteristics. Secondly, the most promising alloys were produced by a fusion/solidification process and shaped by cold rolling. Their microstructures have been characterized by scanning electron microscopy, by EBSD (Electron BackScatter Diffraction), by EDS (Energy Dispersive Spectroscopy) and transmission electron microscopy, as well as by X-ray diffraction. The mechanical properties (microhardness, tensile strength) are also studied.

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B.6. Carbon based materials

ID 115 - Carbon-based monoliths with improved thermal and mechanical properties for methane storage

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A series of activated carbon materials have been prepared from petroleum residue using KOH as activating agent. The gravimetric adsorption capacity for methane of the synthesized samples increases with the activation degree, albeit at a lower packing density of the carbon material. These results anticipate an optimum pitch/KOH ratio (1:3) to achieve an upper limit in the volumetric storage capacity. Activated carbon powders have been conformed into monoliths using a small amount of a binder (5 wt.%), either carboxymethyl cellulose or polyvinyl alcohol, with proper mechanical properties. Incorporation of graphite or graphene in the initial formulation does not alter and/or modify the textural properties of the original activated carbon. However, once conformed into monoliths, the presence of graphite or graphene allows to improve i) the packing density of the monolith (up to 0.52 g/cm3), ii) the mechanical properties of the monoliths (compressive strength 12.3 MPa) and iii) their thermal conductivity (up to 0.49 W/mK) without compromising the methane storage capacity (ca. 100 V/V).

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ID 203 - Multilayer Ti-DLC/ DLC coatings deposited by DC magnetron sputtering

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DLC coatings are metastable films of amorphous carbon obtained by achieving sp^2 and sp^3 structures in a single coating [1]. These coatings have emerged as valuable coatings for mechanical and tribological applications where lower wear and friction are required as compared to conventional materials [2]. The coatings are often doped with metals and non-metals to obtain desired properties.

Ti-doped DLC coatings are used where low friction and wear rate is required along with optimal mechanical properties [3]. Conventionally, doping of DLC coatings with metals like Ti, results in a better tribological behavior but lower mechanical properties such as hardness and elastic modulus. To overcome this problem, multilayered Ti-DLC/ DLC coatings, with different titanium contents, were deposited in this work on M2 tool steel and silicon substrates by DC magnetron sputtering. The coatings have been characterized by nanoindentation technique, for measurement of hardness and elastic modulus, XRD for crystal structural analysis, scratch test for adhesion, and SEM/EDS for morphological and chemical compositional assessment. The results will be presented and discussed.

Acknowledgments: GreenTRIBOS Project Number 860246.

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ID 231 - Investigating the interplay and interface of water and carbon species

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Water and carbon are important building blocks of universe. Carbon is often referred to as being a hydrophobic (water-hating) material. However, water and carbon species often coexist such as in icy comets and clathrate hydrates. The structural arrangement of water molecules hydrating hydrophobic species undergo changes depending on the shape of the hydrophobic species. This research will place the water molecule in different carbon environments and investigate how the chemical and physical properties of water are affected. It will detail how various carbon species affect the crystallization of amorphous ice as well as well as other phase transitions. We use a high-vacuum chamber to synthesize H_2O-carbon samples at various ratios which are characterized by differential scanning calorimetry and X-ray diffraction. Upon heating, various structural changes are seen such as a delay in crystallization of H_2O when carbon species are incorporated into the matrix of amorphous ice. This research will impact astrochemistry, atmospherics and materials science and provides a better understanding across a multi-disciplinary range of applications ranging from biological to physical processes in ice.

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B.7. Composites and nanocomposites

ID 2 - Compressive strength, water absorption, surface hardness and abrasion resistance of geopolymer concretes

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Conventional concrete structures have demanded considerable costs for the repair of their areas affected by surface abrasion. Furthermore, these conventional materials often couldn't meet the requirements imposed by the environment where they are located. Therefore, the growing number of research on geopolymers in recent years reports that this material has satisfactory durability. The main objective of this paper is to investigate some strength (compressive, hardness and abrasion) and tightness properties of geopolymer concrete. For this purpose, mixtures of conventional concrete with cement and geopolymer mixtures with metakaolin and rice husk ash were produced. Bulk density, consistence index, compressive strength, surface hardness, water absorption and an accelerated method of surface wear by abrasion were made. Preliminary results showed that geopolymer concretes had smaller fresh density and a higher consistency index. Moreover, the partial replacement of metakaolin to rice husk ash decreases the water absorption. Furthermore, despite geopolymer and conventional concrete having the same mechanical strength, the two geopolymers mixtures showed higher surface hardness. The geopolymers showed satisfactory surface abrasion resistance too. Thus, the use of geopolymers can be an alternative to replace conventional cementitious materials.

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B.8. Fatigue wear and corrosion

ID 48 - Effect of liquid metal on fatigue life of structural materials: an environmental factor approach

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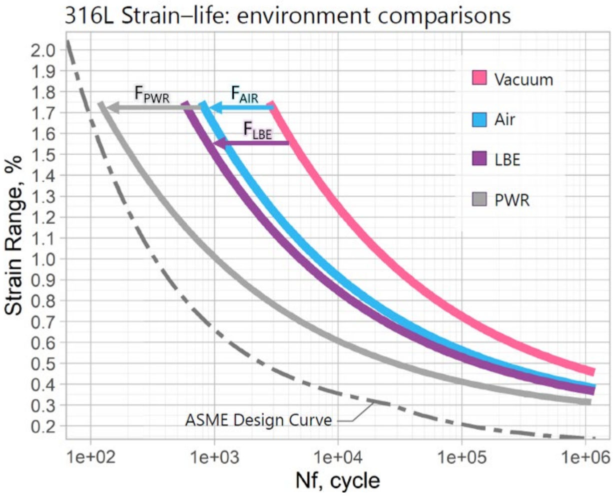
Some types of ferritic and austenitic stainless steels have been long qualified as nuclear-grade structural materials and are widely used in nuclear applications. Their mechanical properties have been well established for a wide range of temperatures, in typical nuclear reactor environments such as air and water. Much less has been investigated about the compatibility and qualification of these materials in contact with heavy liquid metal (HLM) coolants for Generation IV reactors.

One of the key mechanical properties of nuclear materials is fatigue damage, both for reactor design and for life extension programmes. Fairly recently, the effects of light water reactor (LWR) environments on fatigue life have been incorporated in nuclear design codes through the environmental factor (Fen) approach [1].

In this paper, the Fen approach is adapted to assess the environmental effect of a liquid metal, lead–bismuth eutectic (LBE), on the fatigue lives of two stainless steels: ferritic-martensitic T91 and austenitic 316L. The Fen approach is applied to results of low cycle fatigue (LCF) tests performed on cylindrical samples of T91 and 316L in vacuum, air, and LBE environments, setting the vacuum environment as the reference to determine the environmental factor of air and of LBE. The results show that the LBE environment has an effect on fatigue life when compared to the vacuum environment. However, in the conditions tested, the effect is comparable to the effect of air, especially for 316L, where the environmental factors have almost identical values.



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ID 54 - Effect of post treatments on fatigue performance of 18Ni-C300 specimens printed by SLM technic.

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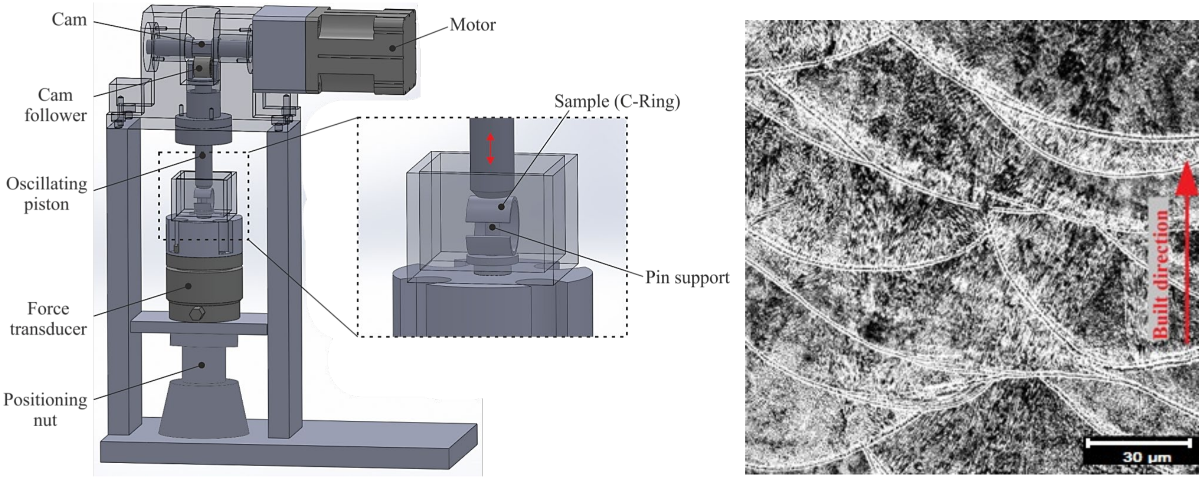
While the potential applications of Selective Laser Melting (SLM) technic are very important, and features such as yield and ultimate tensile strengths with appropriate processes may be very close to those of wrought and cast materials, their final application presupposes the study of fatigue strength that will clarify the range of application possibilities and the modifications of the objects for its mechanical improvement. Printing parameters can affect the structure, roughness and porosity and consequently the mechanical properties of the produced product. The influence of the internal structure as well as post treatments like mechanical grinding, shot peening and heat treatment was investigated on fatigue performance. Hardness measurements as well fractographic and microscopic analysis was made to help analyze the results obtained. The combinations of in situ and post-treatments are highlighted according to the desired final properties.



Innovative fatigue device for c-ring specimens and microstructure of a printed specimen by SLM technic.

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ID 65 - Medium carbon steels: when and why do low loads and holding times increase lifetime?

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With the onset of Very High Cycle Fatigue research, an anomaly in the Miner paradigm started occurring repeatedly: instead of decreasing the residual lifetime of a specimen, low load cycles just below the so-called endurance limit increase lifetime compared to constant amplitude tests even if only the cycles of the highest load block are counted; the low load cycles contribute a negative amount of damage in damage accumulation computations. We give a brief overview over related anomalies and discuss conditions of occurrence as well as possible mechanisms, identifying stress-enhanced static strain aging and oxide-induced crack closure as the most likely ones. We present experiments that show that holding a specimen at tensile stresses enhances static strain aging compared to holding the same specimen at zero stress.

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ID 82 - An investigation into the effect of microtextured regions on the fatigue properties of forged titanium.

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Large industrial forgings of titanium alloy Ti64, are used in modern airframes. These components are used in the β-annealed condition, which produces a microstructure made up of large lamellar grains, with a coarse lath, a consequence of the slow cooling rates in large forgings. When the material is cyclically loaded, strain and stresses will localize within these coarse grains, which could lead to fatigue crack initiation. In this project in-situ high resolution digital image correlations (HRDIC) of monotonic and cyclic loading were carried out. The results were compared to computational modelling, using the crystal plasticity modelling framework DAMASK. Simulations were made using different constitutive laws, with and without back-stresses, to predict how the strain localizes. Both experiments and simulations showed that strain localized in grains that were well-aligned for prismatic and basal slip. Although using the constitutive behavior with back-stresses provide better agreement at the macroscale, it does not significantly change the strain localization at the microstructural scale. The implications of these results for modelling the strain localization in the β-annealed Ti64 microstructures is discussed.

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ID 137 - Effect of simulated body fluid on additively manufactured Ti-6Al-4V under mechanical load

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Titanium alloys are commonly used in orthopaedic implants due to biocompatibility and good osseo-integration, although their corrosion resistance remains to be evaluated. The current work investigates the mechanical performance of additively manufactured (AM) Ti-6Al-4V alloy exposed to simulated body fluid solution (SBF)[1] via in-vitro experiments. Dogbone samples were tested under tensile stress whereas lattice samples were tested under static and cyclic compressive stress. When tested under tensile stress, it was found that prolonged exposure to SBF leads to a decrease in Young's modulus, ultimate tensile strength and ductility. Under compressive stress, it was found that exposure to SBF leads to a decrease in compressive strength of lattices. Electron imaging of fracture surfaces revealed the formation of microscopic cracks in samples exposed to SBF. These results show the effect of corrosion on the degradation of mechanical properties of the alloy, which have not been well studied in AM materials, especially in designs with large surface area: volume ratio. We have evidence that the ingress of oxygen from SBF is crucial in stress-corrosion cracking and further work is currently underway to investigate the oxygen concentration at the crack tips.

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ID 160 - Corrosion behavior of stainless steels in CO2 absorption process using aqueous solution of monoethanolamine (MEA)

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Among the several methods for capturing CO_2 from industrial exhaust gas, the most attractive, in terms of flexibility, is the gas absorption technique employing aqueous solutions of alkanolamines, also known as the amine treatment procedure. However, all amine treatment plants have encountered corrosion issues.

In this study corrosion behavior of two stainless steels (316L and 304L) was evaluated by using a CO_2 -loaded aqueous solution of 30 wt% monoethanolamine (MEA) in a view to simulated corrosion related mechanisms in amine treatment procedures. Corrosion behavior was experimentally evaluated as a function of CO_2 loading in solution and solution temperature, using electrochemical techniques (polarization curves, cyclic polarization and EIS measurement). The results reveal that the aqueous MEA solution containing CO_2 creates a favorable environment for the corrosion of both stainless steels. The rate of corrosion is also accelerated when the temperature of the MEA solution rises. The major contribution to this corrosion enhancement was thought to be dissolved CO2. The substantial reliance of corrosion rate on solution (lean and loaded MEA solution) demonstrates that the corrosion process and reaction are controlled by a diffusion mechanism. This study aims to establish baseline corrosion data for the MEA–CO_2 system for stainless steels, which may be used for subsequent corrosion control research so as to get a better knowledge of corrosion mechanisms and behavior in relation to process parameters that simulate real plant operating conditions.

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C.1. Coatings and surface nanomodification

ID 19 - Influence of Mo on structure-mechanical properties of TiB2±z coatings

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Transition Metal Diboride (TMB2) thin films deposited by physical vapor deposition (PVD) are gaining a lot of attention in industry and academia as next generation protective coatings for diverse applications. So far, TMB2 are known for their refractory character involving high hardness, thermal stability as well as chemical inertness. However, one weak point of these ceramic coating materials is their limited fracture resistance. Here, different theoretical studies [1,2] suggest novel alloying strategies – forming ternary TMBs – to reduce their brittle behavior.

Therefore, in this study we investigated the influence of Mo on the structure-mechanical properties of Ti-Mo-B2±z coatings. Different coatings were grown by DC magnetron sputtering using 6-inch powder metallurgically manufactured TiB2, TiB2/MoB 95/5 mol%, TiB2/MoB 90/10 mol%, as well as TiB2/MoB 80/20 mol% targets. To gain a detailed insight on the structure-morphological properties, a broad set of characterization techniques was used – i.e. electron based microscopy (SEM & TEM), X-ray diffraction (XRD), Nanoindentation, or Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). The fracture toughness was evaluated by micromechanical cantilever bending tests. Furthermore, the oxidation behavior was investigated using a thermogravimetric system. In summary, Mo influences the growth behavior of α-structured TiB2±z films, leading to nanocrystalline but more stoichiometric coating materials. The fracture behavior and super-hardness of Ti-B2±z thin films are not dominated by Mo.

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Keywords: Transition Metal Diborides; Fracture Resistance; Mechanical Properties; Oxidation Resistance; PVD

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ID 21 - Phase formation and oxidation resistance of physical vapor deposited MoSi2 thin films

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Molybdenum disilicide belongs to the group of refractory transition metal silicides, being a highly attractive class of coating material to withstand high-temperature oxidative environments. Due to the formation of a protective silicon-based oxide and having a mixed metallic and covalent bonding character, $MoSi_2$ exhibits an interesting combination of outstanding high-temperature oxidation resistance but also suitable mechanical properties.

Within our study, we deposited $MoSi_2$ thin films by direct current magnetron sputtering (DCMS) and by high power pulsed magnetron sputtering (HPPMS) in pure Ar atmospheres. For the growth of the films an in-house developed (lab-scaled) deposition system was used. The influence of the deposition parameters (e.g. deposition pressure, temperature, or bias potential) on the phase formation and mechanical properties was investigated systematically. All coatings deposited were in-depth analysed concerning their structure-mechanical properties, using a broad set of characterization techniques – i.e. scanning as well as transmission electron microscopy (SEM & TEM), X-ray diffraction (XRD), and nanoindentation. In addition, the oxidation kinetics were studied in detail using a combined differential scanning calorimetry/thermogravimetric system (DSC-TGA). The oxidation kinetics were analysed at different temperature regimes up to 1500 °C.

Keywords: Disilicides; PVD; Protective Coatings; Oxidation; Phase Stability;

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ID 181 - Synthesis and characterization of Si and O doped diamond-like carbon coatings

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Silicon doped diamond-like carbon coatings are well known for their excellent tribological properties and high thermal stability. While, previous studies established that the presence of oxygen dictates the surface properties of these coatings, the effect of oxygen content or O/Si ratio on properties of diamond-like carbon coatings has not been explored in detail. This gap can partially be attributed to the fact that these coatings are conventionally deposited using chemical vapor deposition pathways involving a complex decomposition process of Si-O containing precursors, hence limiting the precise control of dopant concentration in the final coating. This study reports development of silicon and oxygen doped diamond-like carbon coatings by reactive magnetron sputtering. The silicon (0 – 30 at. %) and oxygen (3.2 – 20 at. %) contents in the coatings were varied by controlling the power supplied to the silicon target and oxygen gas flow, respectively. In the case of oxygen-free coatings, silicon content increases linearly with the power supplied to the target. While, for oxygen-containing coatings, a linear relationship between O/Si ratio and oxygen flow/silicon target power, was observed. The hardness of the coatings increases with increasing silicon content (maximum up to 18 GPa) while the introduction of oxygen generally resulted in comparatively lower hardness values (minimum up to 13.5 GPa). A similar trend was observed for the surface energy values of these coatings. The findings of this study will help in the selection of suitable coating stoichiometry for achieving desired coating properties of silicon and oxygen doped diamond-like carbon coatings.

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ID 185 - Hydrogen embrittlement mitigation in austenitic stainless steels by means of surface modification and carbon-based coatings

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Hydrogen is slowly becoming a fundamental building block in the foundations of clean energy and therefore the development of safe and cost-effective storage and transport methods is essential to its success. Hydrogen embrittlement is a widely known phenomenon that particularly affects metals and is responsible for the initiation of fracture and ultimately catastrophic failure of the material or structure. Therefore, one of the main challenges in the development of hydrogen storage and transport methods is related to reducing the interaction of this gas with structural materials. This work focuses on the prevention and mitigation of hydrogen incorporation in austenitic stainless steels for hydrogen storage. Grades 304 and 316L have been chosen within this group because they are two of the most widely used in industry. The objective of the study is to reduce the amount of hydrogen permeating in these materials, either by electrochemical means (cathodic charging) or by gaseous means (at high H_2 pressures). For this purpose, the surface of the materials studied has been modified by laser radiation and carbon-based coatings have been deposited in order to avoid embrittlement.

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ID 196 - Tribological Characterization by Pin-On-Disc test of DLC Coatings Deposited in Ar-Ne Plasma Against Ti6Al4V Counter-Body

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Tungsten carbide in cobalt matrix (WC-Co) is preferably used for various machining applications. However, the increased machining demands, and growing productivity requirement poses added pressure for additional improvement of machining tools performance. Thus, it is being required to coat the cutting edge of the tools with high wear resistance coatings for improve sustainability and longer tool life. Diamond like carbon (DLC) coatings is being used in various applications due to their high stability, good wear resistance and low coefficient of friction properties. Diamond based coatings are used for the machining operation of non-ferrous components like aluminium. However, titanium being a difficult to machine material poses high wear on the tool edge and cause reduction in the tool life. In this study, DLC coatings are deposited on the WC-Co (10% Co binder) having a mirror like surface finish (~0.05 μm) using a High power impulse magnetron sputtering (HiPIMS) technique. The deposition is being accomplished using a plasma of Ar and Mixture of Ar+Ne to produce different variation of coatings. DLC coatings are being tested against the titanium alloy in a pin-on-disc configuration using the equipment build in the CEMMPRE lab [1]. DLC coatings will be tested against the counter-body of Ti6Al4V to analyze the wear mechanism and friction variation against the variation of applied load and sliding distance.

Acknowledgements
This project has received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No. 860246, and from national funds through FCT – Fundação para a Ciência e a Tecnologia, Ref. UIDB/00285/2020. The authors acknowledge Sandvik Coromant Corp., Sweden for supplying WC-Co substrates for deposition and characterization.

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ID 216 - Decorative and functional coatings on polymeric substrates for the automotive industry using an eco-friendly technology

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Many objects found daily in automobile and decorative industries are metal-coated plastics parts that are replacing traditional metallic materials. Most of these parts are manufactured using injection molding and subsequently metallized using eletroplating and eletroless methods. However, these processes include the use of solutions containing extremely toxic and environmentally hazardous compounds such as trivalent ($Cr^{3+)}$ and hexavalent ($Cr^{6+)}$ chromium, which drives the need in industry to develop other methods for deposition of coatings on plastic parts [1]–[4].

Magnetron sputtering (PVD) is a very versatile technique, applied in a non-polluting way, used to produce thin films of a wide range of metals, with excellent properties, being capable to replace the chrome plating. The biggest challenge of this technique involves high temperatures and residual stresses, both intrinsic effects of PVD processes [1], [6].

In the present work, multilayered coatings based on MeN (Me=Ti, Cu) were deposited by magnetron sputtering on polymers substrates. Composition was assessed by Energy Dispersive Spectrometry (EDS), X-ray diffraction (XRD) experiments were performed to evaluate the crystalline structure of the polymer substrates upon thin film deposition and also to assess the crystalline structure of the coatings. The morphology of the films was studied by scanning electron microscopy (SEM) and optical microscopy (OM) and color coordinates were measured by CIELab color space.

The main goal is to achieve a thick coating able to resist external aggressions with good adhesion and without visible cracks.

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ID 233 - TiN/CrN, TiSiN/CrN and NbN/CrN multilayer coatings deposited in an industrial-scale HiPIMS system

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TiN/CrN, TiSiN/CrN and NbN/CrN multilayer coatings of varying bilayer period (Λ) were deposited in an industrial-scale deposition system by means of reactive high power impulse magnetron sputtering (HIPIMS). Three different coatings were deposited for each composition with Λ around 100 nm, 25 nm and 15 nm by directly alternating the sputtering of the different metallic targets in a nitrogen-containing atmosphere. The influence of the Λ was investigated in regard of the chemical composition, microstructure, and mechanical properties of the coatings. All coatings appear to be very smooth and compact in scanning electron microscope images and atomic force microscopy measurements regardless of the bilayer period. X ray diffraction showed separate crystalline phases for the samples with higher bilayer period. However, as the Λ decreases, the position of the peaks overlaps which would indicate the trend to a formation of a single-phase solid solution. More specifically, for the TiN/CrN sample with Λ 25 nm, the presence of satellite peaks around the principal peak confirms that a superlattice structure has formed. The compressive stress, as quantified using the $\sin^2\psi$ method, revealed values between -1 and -3 GPa for all the coating systems.

High hardness values around 25 GPa were obtained by nanoindentation technique for all the coatings.

For the TiN/CrN system, the highest value obtained was 32 GPa while for TiSiN/CrN it was 28 GPa and for NbN/CrN 29 GPa. The maximum hardness value was observed for the samples with Λ 25 nm regardless of the composition.

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C.2. Advanced metallurgical processing

ID 85 - Effects of Precious Metal Doping on Stainless Steels Produced by Spark Plasma Sintering

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The corrosion resistance of stainless steels can prove inadequate in particularly aggressive environments leading to reduced service life and poor performance. Research shows that cathodic modification with precious metals by alloying and coating can improve the corrosion resistance of stainless steels. Research which explores the use of spark plasma sintering (SPS) to produce stainless steels cathodically modified by precious metals remains lacking. This project aims to incorporate precious metals into stainless steels by functional grading using SPS. The metal powders used have been characterised by particle size analysis and SEM. The sintered parts have been analysed using SEM to investigate grain size and elemental segregation. Indentation testing and density measurements of the sintered samples was also carried out.

ID 214 - Kinetic of hardening of 6061 aluminum alloy during electrically assisted artificial aging

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Aluminium alloys are commonly used in the automotive and aeronautical fields due to their excellent mechanical specific properties. The performance of certain alloys such as 6061 can be implemented thanks to artificial ageing heat treatments, increasing mechanical strength and stiffness. Introducing the use of electric current, in particular with high current densities, in several thermal treatments such as recrystallization, solubilization and stress recovery has been shown to significantly increase the transformation and relaxation kinetics by decreasing energy consumption and speeding up the production processes and, in some cases, also improving the performance of the material when compared to traditional heat treatments. This study analyses the effects of artificial ageing on a previously solubilized 6061 aluminium alloy performed by heating the samples to the target temperatures using a resistance furnace and via joule heating generated by continuous and pulsed current applied with a power supply system. The effect of direct current and its application mode on the kinetic of hardening phases precipitation was evaluated and compared with traditionally heat-treated samples by monitoring the hardness and observing the microstructural evolution at different treatment times through optical microscope observations and scanning electron microscopy analysis.

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ID 235 - Process-structure-process relationships of Al-WS_2 self-lubricating composites: Laser Powder-Bed Fusion vs. Spark Plasma Sintering

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Aluminium-based composites have been developed to meet the modern demand for light and energy-efficient materials, thanks to their attractive properties. Laser powder-bed fusion (LPBF) is a metal additive manufacturing (AM) method that selectively melts and fuses metal powder using a high-power density laser beam. Due to the tremendous potential of Al-based composites in various sectors, such as the automotive industry, there has been a global interest in studying their properties when coupled with the unmatched capabilities offered by LPBF. Studies on the tribological behaviour of LPBF Al-based composites are, however, limited, despite their prospects in wear-related automotive parts, e.g. cylinder blocks and pistons. Spark plasma sintering (SPS) is a relatively young solid-state sintering technique that compacts powder materials at lower temperatures and shorter duration compared to traditional methods. Given the enhanced tribological properties of SPS Al-WS_2 composites reported by previous studies, investigations on the microstructure evolution and interfacial reactions between Al and WS_2 during composite production are lacking. The present study used LPBF and SPS to fabricate samples from Al-WS_2 composite powder. The process-structure-property relationship is presented and compared. The samples were characterised for metallurgical and mechanical properties, including hardness mapping, and the effect of SPS and LPBF processing parameters on the properties of the final parts are also studied in depth. This work reports enhanced tribological behaviour for the samples produced using LPBF, which opens up new opportunities for this material-process combination in the field of manufacture for green tribological applications.

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C.3. Thermomechanical processing and severe plastic deformation

ID 55 - Combining thermomechanical processing techniques for rejuvenation of bulk metallic glasses

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Even for a single composition, bulk metallic glasses (BMGs) have access to a relatively wide range of structures and properties [1]. There is particular interest in rejuvenation of BMGs to higher-energy states, as this improves their toughness and plasticity. Among the different rejuvenation techniques used to alter the states of BMGs, techniques exploiting strains within the elastic limit (e.g. elastostatic loading, cryogenic thermal cycling [2]) are of considerable interest: they produce minimal shape change, are relatively easy to apply to a wide range of shapes and compositions, and produce significant changes in properties, including toughness and plasticity. However, recent results highlight the anelastic contributions involved in these processes, which may be relatively short lived [3]. In this work, we combine thermomechanical processing for rejuvenation of BMGs to achieve properties not accessible by single processing techniques and attempt to extend the lifetime of the rejuvenation effects. This work was supported by the European Research Council under the European Union’s Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No. 861046 (BIOREMIA).

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ID 173 - Friction stir welding FSW for AA 6061-t6 with tool without pin

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1 univ djilali libes sidi bel abbes; 2 univ ain el defla

In this article an experimental study of FSW welding with a pinless tool on the 6061-T6 alloy for different parameters is presented. The experimental results are examined by mechanical characterizations in order to judge the quality of the welded joint of each parameter. The parametric study adopted in this part in order to compare the experimental results is a key point to control the welding parameters involved. The studies devoted in this part are: the influence of the welding parameters on the mechanical properties, the observation of the heat flux during welding, study of the microhardness profile after welding for the various parameters.

ID 223 - Incremental Equal Channel Angular Pressing as an Efficient Tool for Grain Refinement: Microstructure and Properties of Large Copper Rods

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Incremental Equal Channel Angular Pressing (I-ECAP) is a continuous method, and its main advantage over conventional ECAP is the ability to process continuous billets like bars, rods, or sheets. It was obtained by separating feeding and deformation processing stages by using movable tools, which substantially reduces friction. The arrangement and processing steps of tools for I-ECAP are shown in Fig. 1. An input channel is established by the clamp pressed towards the die, which, together with the punch, defines the output channel. The plates are fed by the pusher during punch withdrawal. The punch moves in a reciprocating manner at an appropriate angle.

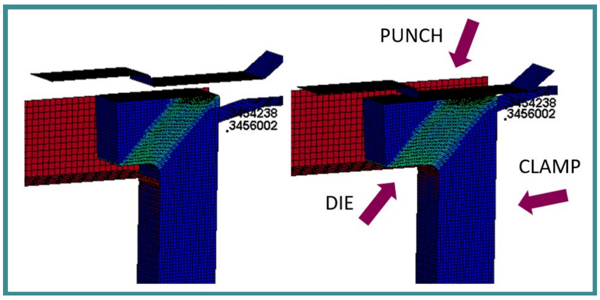

Fig. 1 Arrangement and processing steps of I-ECAP tools

I-ECAP is proposed as a method to produce copper rods with high strength and high electrical conductivity through grain refinement. Pure copper is an excellent conductor of electricity, yet its mechanical strength is limited, which hinders the applicability of this material as a structural component. The mechanical strength of pure metals is usually enhanced by alloying, yet the addition of other elements results in a significant drop in electric conductivity. The proposed alternative method makes it possible to achieve the goal of hardening the material and preserving conductivity.

In this work, large copper rods were processed by I-ECAP up to 8 times. The processing resulted in acquiring ultrafine-grained microstructure. The mechanical strength increased by around 60% in comparison to the initial condition. The electrical conductivity was 81 %IACS in the initial condition and after processing was almost identical as was equal to 82 %IACS. After annealing, it was possible to increase conductivity to 94 %IACS without significant changes in the microstructure and mechanical properties.

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C.7. Powder technologies

ID 31 - Advanced zinc-magnesium alloys prepared by mechanical alloying and spark plasma sintering

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Biodegradable metals are considered for temporary substitutes or fixation devices of damaged body tissue. Zinc alloys are proposed due to excellent biocompatibility and corrosion properties as a promising materials for biodegradable devices (stents, screws etc.). Such materials needs to achieve excellent mechanical properties, which are affected by composition or preparation techniques.

We have focused on the preparation of Zn-1Mg and Zn-6Mg alloys from pure powders by mechanical alloying (MA) and compaction by spark plasma sintering (SPS). MA was selected to form fine-grained, homogeneous powders with the possibility to form metastable phases. Selected powders were compacted by SPS, compaction method enabling the consolidation by current flow with increased temperature and pressure, which prevents grain coarsening.

The microstructure and mechanical properties of prepared materials were studied. Results showed effect of parameters of MA (rotation speed, time of MA, etc.) on process efficiency and powder phase composition. Desired microstructure conditions including fine grains, intermetallic phases and the existence of solid solution of Mg in Zn were observed for relatively short times (60 min) and high rotation speed (1200 RPM). Compaction by SPS have preserved fine, homogeneous microstructure consisted of zinc matrix and intermetallic phases Mg2Zn11, MgZn2. Size of grains and intermetallics achieved hundreds of nanometers which increased hardness and compressive yield strength values up to 436 MPa for Zn-1Mg (574 MPa for Zn-6Mg). However, low plasticity was observed due to the existence of brittle phases.

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ID 194 - Development of NiAl intermetallic coatings from mechanically deposited nickel and aluminum elemental powders

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NiAl intermetallic coatings are of great interest due to their high both strength and wear resistance retained up to high temperatures. Among various methods capable of production of thick coatings, one may distinguish electrodeposition and plasma spraying. Presently developed approach covers co-milling (up to 32 hrs. at 300 rev/ min) of the Ni and Al powders together with steel platelets (serving as substrates) in a planetary mill. The investigation was performed using light, scanning and transmission electron microscopy (LM/SEM/TEM), as well as X-ray diffractometry (XRD). They indicated that already after 4 h of milling the layer consisted of NiAl with a minor contribution of Al_3Ni. The surface of the steel platelets is roughened in a kind of shot-peeing process. Adhering to it are whole particles of elemental powders followed by particles of welded together bands of still non-reacted elemental material. The last layer is formed by NiAl particles formed during milling. Their high brittleness are the main reason behind the suppressing of addition of a new material on the top of the coating. An in-situ TEM heating (20 K/min.) indicated that after reaching ~350°C recovery processes start within the substrate and the defected crystallites of Al and Ni localized at the interface. Only after reaching ~500°C the reaction between the last two phases starts adding more NiAl and Al_3Ni to that already present at the top of the coating. The excess of aluminum in the starting load of elemental powders is crucial as it concerns obtaining a proper adhesion.

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C.8. Direct and indirect additive manufacturing

ID 12 - LIFT of CuO NP inks: Process parameters and High Speed visualization

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Laser-Induced Forward Transfer (LIFT) has emerged as a direct nano- and micro-fabrication technology allowing the digital printing of a large variety of materials for components with excellent form factors exhibiting flexible and stretchable performance. Inks comprising metals like silver have been primarily employed with LIFT due to their high conductivity and environmental stability [1], while copper, exhibiting high conductivity, can be an attractive cost-effective candidate for solution processed metal nanoinks. In this work, we employ a high-speed imaging setup coupled with a LIFT station in order to investigate the liquid jet's propagation and process parameters. A phenomenological analysis of the jet's propagation is carried out using a non-Newtonian CuO NP ink, designed for use with LIFT [2]. Morphological characterization confirmed that controllable printing of droplets is feasible from a donor layer thickness set at 30 μm and a donor-receiver gap distance set at 90μm. In addition, printing of linear patterns has been accomplished by scanning the laser beam over the donor surface with 0.55 m/s scanning speed. Finally, laser sintering was applied to the linear printed patterns on glass substrates in order to investigate the conductivity of these patterns in ambient atmospheric conditions.

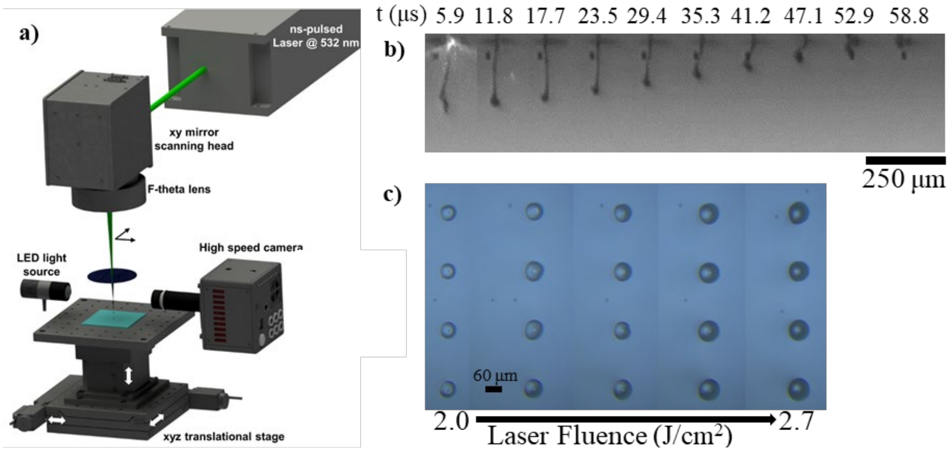

Figure 1: a) Illustration of the LIFT setup, coupled with a high-speed imaging setup; b) CuO NP ink jet's propagation during LIFT printing process; c) Microscope image of the printed droplets at different laser fuences.

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ID 67 - Additive Manufacturing of Bioinspired Architectures with Polysaccharide-Based Photoresists

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The polysaccharide cellulose is next to chitin the most abundant biopolymer on earth and is considered an almost inexhaustible source of raw material for the increasing demand of environmentally friendly and biocompatible products.[1,2] We recently synthesized a bio-based photoresist, where a photo-reactive cellulose-derivative is dissolved in an organic solvent together with a photoinitiator.

This novel photoresist is curable by two-photon absorption at 780 nm in a direct laser writing (DLW) system (Nanoscribe Photonic Professional GT). With this setup, two-dimensional architectures with a linewidth of less than 250 nm and a minimum line distance of 500 nm are achieved. Our bio-based photoresist allows three-dimensional structuring of cellulose on the µm scale via DLW.[3] Curing of our cellulose derivative is generally possible in liquid and solid state via two-photon absorption.

In contrast to common photoresists, which are based on polymers sourced from mineral oil, our approach conserves resources through replacing those polymers by sustainable materials such as polysaccharides. The presented research includes the functionalization of cellulose to enable photo-crosslinking for generating biopolymer-based hierarchical architectures. This chemical modification is a prerequisite for the fabrication of two- and three-dimensional structures by DLW

Moreover, this polysaccharide-based photoresist enables manufacturing of biomimetic architectures, which consist entirely of a natural bulking material. Additionally, this cellulosic photoresist is curable via one-photon absorption with a UV-lamp (365 nm) in liquid as well as in dried state. Our resist opens up a new class of photo-curable polymers based on sustainable and renewable materials.

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ID 72 - 17-4 PH SS single tracks DED depositions over AISI316L: macro and microstructural investigations

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17-4PH is a precipitation hardening stainless steel widely used as an engineering structural material in different industrial sectors, such as aerospace, chemical and biomedical fields due to its ease of fabrication, high strength and elevated corrosion resistance. The peculiar properties of this steel are promoted by both the reinforcement induced by the precipitation of submicron-sized secondary phases occurring during the ageing treatment and its low carbon content. Nowadays, additive manufacturing has gained much attention and, among others, the Direct Energy Deposition technology (DED) is a promising process. The DED technique consists of the deposition of laser melted powders and it is increasingly used to manufacture or repair complex mechanical parts.

In the present investigation 17-4 PH powders were used as a feedstock to perform single-track depositions over an AISI 316L plate employing different process parameters such as laser power, scanning speed and powder feed rate. Some geometrical features of the tracks were preliminary studied. Microstructural investigations were carried by means of optical microscopy (OM), scanning electron microscopy (SEM) and X-ray diffraction (XRD) to deeply investigate the metallurgical characteristics of the joints. Different dilutions were measured according to the process parameters, finding that these conditions affect not only the porosity content but also the evolution of phases along the cross section of the track. Besides that, it was observed an effect of a precipitation hardening treatment performed for a 17-4PH material, from which different hardness and microstructure were detected before and after the heat treatment.

Keywords
17-4PH, DED, microstructure, porosity, geometrical analysis

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ID 114 - Evaluation of softening resistance of DED tool steels at elevated temperatures

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Introduction

As a manufacturing method of materials, Direct Energy Deposition using Laser beam (L-DED has the advantage in the modification of surface property [1]. It can be used for the repair and manufacturing of multilayer coatings [2]. In this work, for the purpose of hard-facing, two different tool steels were deposited on a hot work tool steel and the softening resistance was evaluated.

Method

Uddeholm V4E (a cold work tool steel) and Uddeholm AGDA (a unique boron tool steel) with multilayers were cladded on the base material (Uddeholm Dievar, a hot work tool steel) by means of L-DED. The cladded steels were tempered to obtain the highest hardness. Subsequently, the tempered steels were softened by the treatments at 550 °C and 600 °C for 100 h. The softening resistance of the cladded materials was evaluated by hardness measurement. Microstructure was characteristic by means of SEM and XRD.

Results

The hardness evolution was depicted for the investigated steels with varied softening temperatures. Moreover, the corresponding microstructure was revealed to explain the hardness change. The correlation between microstructure and mechanical propertiy will be discussed for each steel.

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ID 165 - Characterization of pure copper parts manufactured via Fused Filament Fabrication (FFF)

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Fused Filament Fabrication (FFF) is a very versatile technology for manufacturing complex components made of polymers, fiber-reinforced systems, metals and ceramics. Highly-filled filaments composed of a high-volume content of sinterable powders and a binder system are needed to produce metal parts. After the first shaping and printing step, the binder system is removed through catalytic and thermal debinding. Then, the specimens are sintered to obtain dense parts with higher mechanical properties. FFF is free from thermal dissipation and energetic issues caused by high reflectivity and conductivity of copper, which others AM techniques such as selective laser melting (SLM) suffered.

In this work the applicability of Fused Filament Fabrication AM technique to produce pure copper parts is demonstrated. The aim of this research is to provide a preliminary characterisation of the FFF process using Markforged equipment and process parameters with a commercially available highly-filled copper filament to produce copper components with different geometries. Microstructural characterization of the metal-polymer filament, as-printed green parts, washed brown parts and final sintered parts was provided. In addition, the relative density of the final pure copper parts was evaluated, as well as their dimensional accuracy after the shrinkage inherent to the sintering process. The mechanical behaviour of sintered parts was assessed through tensile, hardness and impact toughness characterization. Finally, the thermal and electrical conductivities of final pure copper components were measured and compared with wrought copper and published results of other AM technologies.

Moreover, this work studies for the first time the feasibility of Concentrated Solar Energy (CSE) application in the copper sintering stage. CSE is one of the most promising clean and renewable energy resources, non-polluting and highly efficient for high temperature materials processing. Cylindrical parts were printed using Markforged 3D printer and the sintering stage was carried out by a low-cost solar installation, Fresnel lens. Solar-sintered pure copper parts were evaluated and compared with copper specimens manufactured following the complete Markforged fused filament fabrication technique.

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ID 218 - Laser-Induced Forward Transfer for drug thin film printing

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The use of different 3D printing technologies for pharmaceutical manufacturing provides new opportunities for personalized medicine and custom tailor-made pharmaceutical products with adjusted doses in various shapes and structures. In this work, we demonstrate a novel manufacturing method of printing personalized dosage forms of liquid-phase active substances (i.e. paclitaxel) onto edible wafer papers as orodispersible thin films, for more prolonged drug release and long-term effectiveness, targeting diseases such as types of cancer. Laser-Induced Forward Transfer (LIFT), a direct and non-contact laser printing technique, can be used to laser transfer paclitaxel molecules on various surfaces (e.g. edible wafer paper) that could be used as depot formulations. LIFT has been successfully applied in the nanosecond regime for the controlled transfer of paclitaxel solution onto the receiving substrates. Quantification studies of printed paclitaxel amount were determined by means of liquid chromatography–mass spectrometry (LC–MS/MS) analytical technique. Furthermore, initial studies were performed where the printed paclitaxel wafer papers were used as orodispersible films (ODFs) on mice for oral mucosal drug delivery application, revealing encouraging results.

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ID 224 - Application of the Box-Behnken design in optimization of additive manufacturing process parameters for H13 steel

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Abstract: The Box Behnken design was applied to optimize the laser-based powder bed fusion process parameters for the H13 tool steel. Thirty-nine samples were produced (3^3 with triplicate). Both porosity and Archimedes density were chosen as a response. The porosity values of those samples varied between 7.85% and 0.12%. The optimal parameters determined through the response surface analysis were: laser power = 212 W; hatch distance = 95 µm; scan speed = 580 mm/s. The optimal parameters led to an experimental porosity value of 0.09 ± 0.03%. Similarly, the initial density values were between 7.161 and 7.658 g/cm³. After manufacturing with the optimal parameters, this value increased to 7.688 ± 0.006 g/cm³. The box-Behnken model allowed the optimization to be done faster and at a lower cost than other designs of experiments.

Keywords: AISI H13, laser-based powder bed fusion, Box-Behnken.
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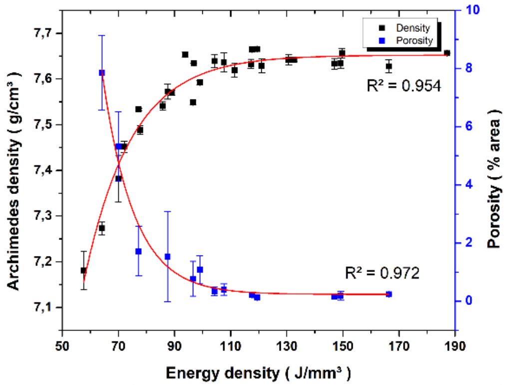

Fig. 1 - Density and porosity curves as a function of energy density.

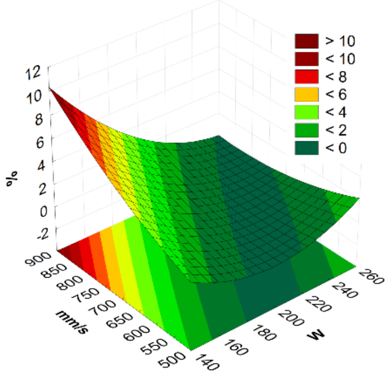

Fig. 2 - Porosity response surface.

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ID 228 - Printability of Reused IN625 Powders without and with Alumina Particles through Direct Energy Deposition

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Additive Manufacturing (AM) processes such as Direct Energy Deposition (DED) or Selective Laser Melting leave powder residues that cannot be reused due to the increase in oxygen and the changes in particle shape and size [1]. The idea of reusing these powder residues can create a sustainable production, adding value to these scrap materials. These powders can be mixed with reinforcements, as composite powders, to produce hard claddings or metal matrix composite structures through AM processes. The application of DED seems feasible since the carrying gas can influence the effect of powder characteristics such as powder shape and size [2].

This study investigates the printability of a reused Inconel 625 powder without and with Alumina particles (average size of 10 µm) through DED process. A mixture of 10 wt.% alumina was prepared through ball mill shaking process for 9 hours. Parametrization of these powders was proceeded by printing different lines on an AISI 4140 substrate, considering laser power, scanning speed and powder feeding rate. This methodology ensures achieving an optimized bead geometry and dilution. Analyses involved visual observations and metallography. Then, 20x20 mm^2 planes of IN625 with and without alumina (one and three layers) were produced by printing lines continuously with an overlapping of 35 %, applying offset-in strategy between subsequent planes. Evaluations on polished cross-sections included porosity, distribution of alumina particles, dendrite and grain structure, phase evolution, the state of substrate-deposition interface and heat affected zone, and microhardness measurements. Microscopic observations involved optical and scanning electron microscopies.

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C.9. Joining

ID 24 - Evaluating Residual Strains in Tungsten-Copper (W/Cu) Dissimilar Joints for Fusion Reactors using Bragg-edge Neutron Imaging

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DEMOnstrative power plant (DEMO) is a proposed fusion reactor design that will act as a successor to the International Thermonuclear Energy Reactor (ITER), which will explore the feasibility of fusion as a sustainable energy source on a more practical and commercial basis. The joining of dissimilar materials is necessary from a design perspective in many DEMO components which comes with the consequences of induced residual stresses from incompatible strain fields arising due to high temperature joining techniques. Such stresses can hugely influence the mechanical properties, service life and performance of components. More importantly, residual stresses that are tensile in nature are contributors to stress corrosion cracking and reduce the fatigue life of materials. Hence, being able to accurately measure and assess the impact of these stresses is essential to the assessment of various design components in DEMO.

The sample considered in this project was an as-cast Tungsten-Copper (W/Cu) dissimilar joint proposed as one of the design configurations for the cooling system of the divertor component within the fusion reactor. The time-of-flight neutron Bragg-edge imaging (ToF-NBEI) technique offers the ability to map bulk residual strains across an entire sample, non-destructively [1]. In this work, the ToF-NBEI experiment was conducted on the IMAT beamline in the UK and 2D macrostrain maps were obtained. Adjacent to the W/Cu interface, a significant tensile residual strain was observed. The results are complimented with further microstructural and texture analysis to provide a complete understanding of the residual stress profile obtained.

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ID 56 - Wetting behavior of liquid pure magnesium on pure tungsten substrates

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Currently, magnesium and its alloys are intensively studied in many industrial fields, especially in the automotive and aerospace industries, due to their low density and high strength-to-weight ratio. Much attention has been paid to interfacial phenomena occurring in the high-temperature liquid-assisted processing and operation of such materials [1-2]. However, due to the high reactivity of magnesium and magnesium-based materials with air components (especially at elevated temperatures), as well as the high vapor pressure of Mg at temperatures near (or above) the melting point of Mg, the thermophysical and physicochemical properties of liquid Mg and its alloys are not fully understood. This information is of practical importance for the proper selection of refractory materials for melting and casting Mg alloys, as well as for reliable measurements of their thermophysical properties by container-assisted methods [3].

In the present work, we focus on studying the high-temperature interaction of liquid Mg in contact with pure tungsten substrate. High-temperature wettability studies were performed in a protective atmosphere (Ar 99.999% or Ar-5%H2) at a temperature of 700 °C using the sessile drop method. To avoid the effect of native oxide film on Mg drop on contact angle measurements, the non-contact heating of examined couple of materials to the test temperature was combined with in situ cleaning of Mg drop directly in a high-temperature chamber using capillary purification procedure [3]. For this, an Mg sample was placed in a graphite capillary above a W substrate and after reaching the test temperature, the Mg drop was mechanically squeezed from the capillary and deposited on the substrate [1,3]. The images of the drop/substrate couple were recorded with two high-resolution high-speed digital cameras from two directions of observation. These images were used for measurements of the contact angle values (θ) formed between the liquid metal and the tungsten substrate. After wettability studies, the samples were subjected to detailed microstructural observations by OM, SEM, and EDS analysis.

Under conditions of this study, liquid Mg showed non-wetting ($\theta > 90°$) behavior and a lack of permanent bonding between liquid Mg on W substrates caused by the non-reactive character of Mg-W system, because W has negligible solubility in liquid Mg and it does not form any compounds with W.

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Acknowledgment
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ID 177 - High-temperature interaction of liquid magnesium with cast iron substrates

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Information on the interaction of liquid magnesium and its alloys with different refractory materials is of practical importance in many liquid-assisted processing of Mg-based components.

In this study, high-temperature sessile-drop wettability tests were conducted with pure Mg on three types of cast iron substrates (Fe-C alloys with different morphology, quantity, and distribution of the graphite phase). The tests were performed at a temperature of 700°C in a protective atmosphere (Ar 99.999%) using two testing procedures, i.e. (1) classical contact heating of Mg/substrate couples to the test temperature and (2) non-contact heating combined with capillary purification of Mg drop from a native oxide film directly at the test temperature in an experimental chamber by squeezing the drop from a graphite capillary placed above a cast iron substrate.

The high-temperature behavior of Mg/substrate couples was recorded with a high-resolution, high-speed CCD camera and collected images were used for measurements of the contact angle values formed between the liquid Mg and selected substrates. The solidified Mg/substrate couples were subjected to detailed microstructural observations by light microscopy (LM), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS).

The results obtained are discussed in terms of the role of primary oxide films on both the Mg and Fe-C specimens on wetting behavior and the spreading of liquid Mg over selected substrates. It involves high-temperature capillarity and the methodological issues favoring the capillary purification method vs the classical sessile drop method combined with the contact heating, thus resulting in significant improvement of wetting and fast-spreading when non-oxidized Mg contacts with non-oxidized Fe-C substrate.

LM, SEM and EDS analysis used for structure and chemistry characterization of interfaces in Mg/substrate couples revealed the effects of morphology, quantity, and distribution of the graphite phase in cast iron alloys used in this study on the wetting behavior of liquid Mg and its bonding with selected substrates.

Acknowledgment

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ID 220 - High-Speed Sinter-Bonding by in situ Reduction of Oxalate Skins on Cu Particles in Reducing Formulation

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As wide-band-gap semiconductor power modules, requiring high-temperature operation, emerge in plug-in electric vehicle industry, sinter-bonding pastes have been widely developed to replace solder material in die attachment. Although application of Cu filler is cost-effective, compared to Ag, Cu easily oxidizes in air and the degree accelerates with increasing bonding temperature. The oxidation on Cu surfaces eventually presents poor sinter-bondability by interrupting the paths of atom diffusion for sintering. In this study, bimodal-sized Cu particles were surface-treated with oxalic acid and a novel sinter-bonding paste were suggested by mixing the particles with a glycol-based reducing solvent. The surface-treated Cu particles exhibited a distinctive surface morphology through forming oxalate ligands on the surfaces of Cu particles, which was confirmed by Fourier-transform infrared spectroscopy. The copper oxalate layer decomposed during the heating to 300 ˚C, which was clearly verified by thermogravimetry-differential thermal analysis and X-ray photoelectron spectroscopy. The bimodal Cu paste was printed a Cu-finished substrate, and a Cu-finished die was attached at 300 ˚C in air by sinter-bonding under compression pressure of 2 MPa. Consequently, only 1 and 3 min sinter-bondings presented the sufficient shear strength of 20.82 and 25.28 MPa, respectively. Furthermore, the 3 min bonded bondline was observed as the near-full-density microstructure containing homogeneously distributed tiny voids. The fast sinter-bonding speed was attributed to oxidation suppression and effective Cu surface reduction by the double effect of the oxalate layers and reducing solvent. The characteristics of suggested Cu paste showed the feasibility of application to a semi-continuous die-attachment process using a conveyor belt.

D.1. Advanced techniques with focused beams

ID 50 - Effect of thermal treatment on the atomic structure and dynamics of PdNiP metallic glass

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In the present work, the impact of thermal treatment on the atomic structure of the well-known bulk metallic glass (BMG) $Pd_{40}Ni_{40}P_{20}$ (at.%) is investigated via transmission electron microscopy (TEM) methods. Compared to other metallic glasses, PdNiP offers high kinetic stability and superior mechanical properties, which depend on composition and thermo-mechanical pre-treatments. However, not much is known about the effect of thermal treatment on the atomic structure and related dynamics.

The amorphous bulk material is produced via induction melting and copper-mold casting (Fig. 1). The correct composition and amorphous structure are confirmed by X-ray diffraction (XRD), differential scanning calorimetry (DSC) and energy-dispersive X-ray spectroscopy (EDX). Subsequent thermal processing routines are performed, which consist of heating routines and sample relaxations well below the glass transition temperature of around 576 K. The impact of thermal treatment on the structure and dynamics of amorphous PdNiP is further examined using TEM analysis methods, including electron correlation microscopy (ECM) at room temperature. ECM is based on analyzing a time series of dark-field TEM images to study local dynamics in a material [1]. This allows investigating the atomic structure of thermally treated PdNiP samples in comparison with a defined reference state, which gives further insights into the properties and the behavior of the amorphous phase.

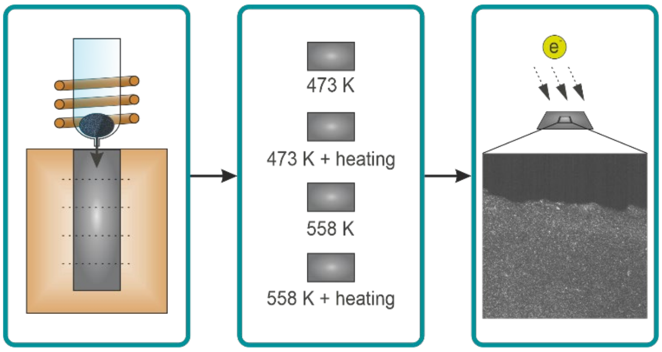

Figure 1: Schematic approach of sample preparation by copper-mold casting, thermal treatment, and investigation of the atomic structure and dynamics of amorphous PdNiP via TEM.

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ID 97 - De Havilland Goblin: seeking materials, processes, and current state of an engineering breakthrough

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The *de Havilland Goblin*, conceived in 1941 by Frank Halford, was the primary engine used in the iconic *de Havilland Vampire*. The model currently under study is the *de Havilland Goblin 35* engine Nº9284. The engine belonged to the Portuguese Air Force, incorporating a jet with a teaching and training purpose. FEUP later acquired the engine, and it is now an icon of the Mechanical Engineering Department. It has been part of the FEUP Museum collection since 2004.

The study of the *de Havilland Goblin* engine aims to contribute to showcasing an important historical aviation component for the scientific community. This study focuses on gathering information about the materials used and their current state of conservation.

The aim of this study is to analyse the different parts of the engine using a set of non-destructive tests (NDTs), considering its preservation. NDTs will provide information about the materials (alloys and coatings) used and their chemical composition. Furthermore, minimal damage sampling is required for more specific testing: mechanical and microstructural analysis by OM, SEM/EDS, and hardness tests. The results made it possible to correlate the historical data of the engine with evidence on materials and processing techniques used at that time. The results also contributed to further knowledge about the engine, such as its working conditions and post discharge life.

Regarding the engine's historical value, all knowledge obtained has a social and technical impact on the community. Guaranteeing its preservation, integration and proper exhibition is also a crucial part of this study.

D.2. Characterization of nanomaterials

ID 187 - Experimental and theoretical study of aluminosilicate gel

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A group of inorganic networks, more descriptively named geopolymers are materials on the verge between ceramics and polymers, are commonly produced using reaction of aluminosilicate rich powders such as metakaolin or fly ash type F and sodium metasilicate. Despite geopolymers can be synthesized in a broad compositional range, they typically follow $Na_2O : Al_2O_3 : SiO_2 = 1 : 1 : 4$ stoichiometry. This was also the case for this study where highly reactive commercial metakaolin (HRM) (Metamax, BASF) was used as precursor for preparing of geopolymeric gel, under different curing conditions. This type of metakaolin is advantageous due to its purity and ability to yield geopolymeric gel at room temperature. The experimentally obtained IR spectra of geopolymer gel was compared with quantum chemical calculations for hydrated aluminosilicate structure known as not fully polymerized geopolymer structure. It is presumed that a fully geopolymerized structure does not have terminal –OH groups, i.e. that all Si and Al atoms are bridged by oxygen. Modelling by Gaussian of such complex structure is difficult. Therefore, aluminosilicate structures containing a certain amount of terminal –OH groups were used for theoretical quantum-chemical considerations. Such structures are called hydrated aluminosilicate gels. Quantum chemical calculations of hydrated aluminosilicate gel were obtained in Gaussian 09 using Density Functional Theory (DFT) with B3LYP functional. The aim of this study is to gain deeper knowledge about geopolymerization process via correlating the experimental and theoretical results of the aforementioned aluminosilicate material networks.

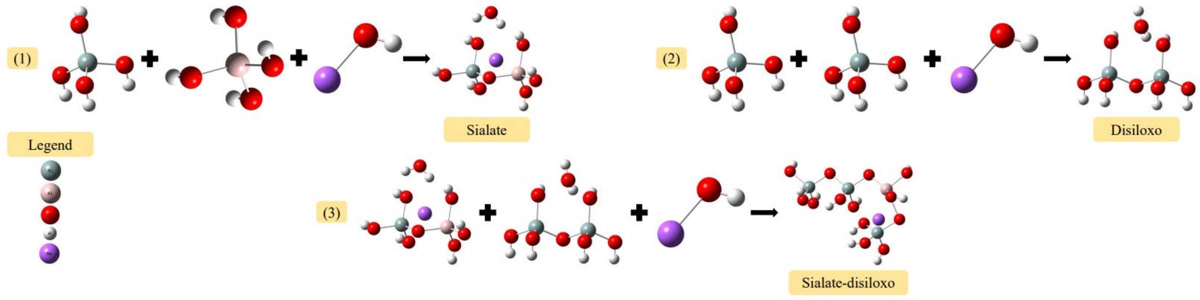

Figure 1. Simplified schematic representation of geopolymerization with terminal hydroxyl groups; (1) sialate formation, (2) disiloxo formation and (3) sialate-disiloxo formation.

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ID 234 - Airborne microplastics: surface chemistry and cloud-crystallization properties

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The amount of micro and sub-micro sized plastic particles polluting the environment is increasing because of human activity. It has been shown that microplastics have an adverse effect on the environment, but little is known about their influence on the formation of ice in clouds. Moreover, our theoretical understanding behind the phenomenon of ice formation is fairly poor. As a result, models cannot be used accurately to predict whether a substrate will promote the formation of an ice nucleus within a body of liquid water. Therefore, experimental investigations are needed to assess whether a plastic species will promote the formation of ice crystals in clouds. In this study, microplastics were synthesized using cryogenic ball milling and separated in size by sieving. Their sizes and surface functionalities were investigated using XPS, optical microscopy, and IR spectroscopy. The ice nucleating properties of the laboratory made microplastics were investigated by performing droplet freezing experiments with suspensions of the plastic samples in ultrapure water. For this the IceBox, a novel open droplet system, was used to cool suspension droplets with a constant rate in an atmosphere with controlled humidity. Our results show that microplastics have a notable effect on the temperature of freezing of the droplets. This research will be of importance to both the field of atmospheric science and ice physics.

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D.3. Micro- and Nano-mechanics and modelling

ID 118 - Effects on Liquid Metal Environment on Slip Band Morphology of 316L Austenitic Stainless Steel

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In the development of Generation IV nuclear reactor, it is essential to understand the interaction between corrosive coolant such as molten salts or lead alloys with structural materials [1]. As one of the candidate materials for Generation IV reactor, 316L so far does not show any evidences on susceptibility to liquid metal embrittlement in contact with liquid lead-bismuth eutectic (LBE), one of the coolant candidates. However, there are evidences that LBE might influence plastic deformation of austenitic stainless steels even not being the embrittling factor. Understanding of the mechanisms of the interaction between liquid lead alloys and steels are necessary for the justification of the assessments on potential materials degradation effects.

In one of the mechanisms proposed to explain liquid metal embrittlement (LME), adsorption of liquid metal atoms eases the nucleation of dislocations [2]. This interaction may manifest as changes in the surface morphology of the sample since surface is where the interaction between environment and material starts to happen.

In this study, we investigated the effect of LBE on the slip morphology and near-surface deformation of 316L. We use uniaxial loading in slow strain rate tests (SSRT) to obtain materials with a specified deformation state. The surface morphology were investigated with scanning electron microscopy (SEM). In addition to that, in this work we tried to assess the use of quantitative SEM image analysis in assessing slip morphology such as slip band spacing. The assessment was carried out by comparing the results with a more advanced technique such as atom force microscopy (AFM).

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ID 130 - Modelling the effects of texture on the stress and strain localization during bending of aluminium alloy sheet

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When aluminium alloy sheets are bended, early failure can occur at grain boundaries on the surface deformed in tension. The cause of this failure is still unknown, but it has been observed that different textures lead to different propensities to failure. In this project, crystal plasticity modelling (CPM) is used to relate texture to bending failure, focusing on the effect of texture on stress and strain localisation, which is suspected to cause crack nucleation. The workflow uses the DAMASK modelling framework to model crystal plasticity, the MATFLOW package "formable" for parameter fitting, and MTEX for texture calculations. The inputs are textures measured with Electron Backscatter Diffraction (EBSD) and experimental tensile test curves. Uniaxial and plane strain tension load-cases, both to 15% strain, were applied to a Representative Volume Element (RVE) to simulate tension in pre-stretching and bending of three different textured samples. The stress and strain tensors at every point of the RVE were extracted and visualised using Paraview. Non-uniform stress and strain distributions were found, and the plane strain tension load-case reached higher stresses and strains. However, small differences were found between three different textured samples at these low strain levels. Nonetheless, these results are the starting point to develop a model capable of predicting the effects of microstructure on failure in bending. Future work will extend the simulations to larger, more representative strains. Grain shape, texture gradients, voids and hard particles, which could also have a strong influence in the initialisation of cracks, will be considered later on.

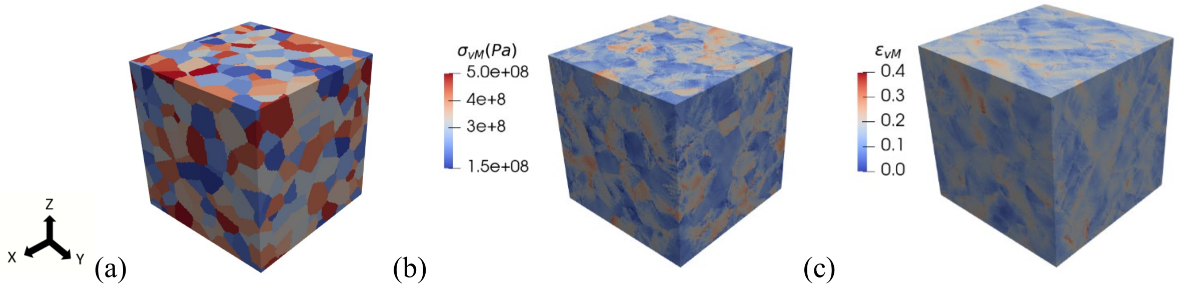

Figure: Representative volume element with 500 grains and 100^3 grid size, showing (a) the microstructure and (b) the stress and (c) strain distributions after simulating uniaxial tension along X to a target strain of 0.15.

Acknowledgments: The authors gratefully acknowledge the EPSRC (Engineering and Physical Sciences Research Council, UK), SFI (Science Foundation Ireland) and Novelis Inc for their financial support. Matflow development is performed in Lightform, a 5-year multidisciplinary project led by The University of Manchester with partners at University of Cambridge and Imperial College, London. We acknowledge the Lightform group members for their technical discussions and support, in particular Adam Plowman and Michael White for their help with the modelling work.

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D.4. Atomic scale modelling of advanced materials

ID 175 - DFT simulations of the behaviour of aggregates of light impurity atoms in a tungsten interface

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Nanostructured materials with multiple grain boundaries showing diverse orientations have shown a great ability to reduce the damage that will be produced by irradiation in a future nuclear fusion reactor [1, 2], being nanostructured tungsten a especially prominent candidate material [3]. Among the diversity of deleterious effects produced by the impact of high energetic particles on first wall or plasma facing materials, the formation and aggregation of vacancies and interstitials and the ubiquitous presence of hydrogen and helium that would eventually end up in bubble formation, in obvious detriment of the performance and operational life of such structural materials, play a major role. To assess the above issues, DFT simulations focused on an energetic, structural and mobility analysis of the effects due to the simultaneous presence of intrinsic (vacancy, self-interstitial atom, SIA) and extrinsic (light impurity atoms, LIA) point defects in a W110/112 grain boundary (GB), appearing as a consequence of the impinging radiation in a fusion environment, have been performed. The aim is to shed light on the experimental controversy about the reduced/suppressed blistering caused by He [4], whereby nanobubble formation near surfaces leads to reduced H retention [5].

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ID 182 - First principle study of Co-catalyzed MgH2 for CO_2 capture and conversion

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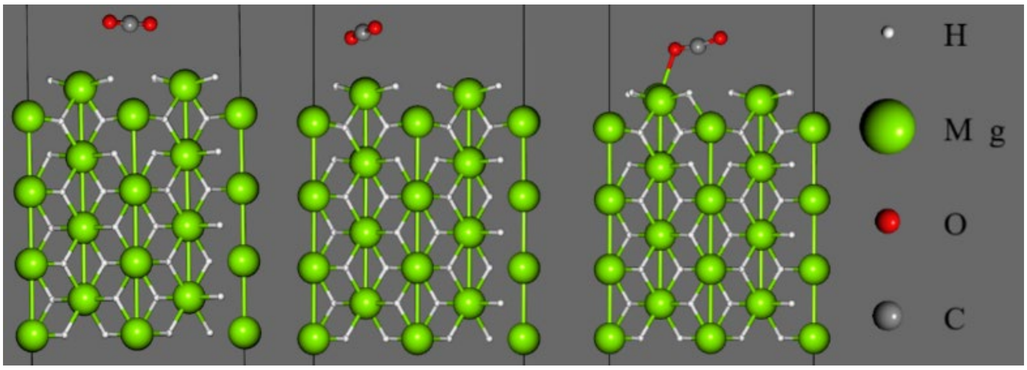
Magnesium hydride and cobalt catalyzed magnesium hydride systems were studied from an atomistic point of view for the purpose of understanding the interaction mechanism with carbon dioxide and hydrogen pure gases using first principle calculations. In a previous work, carbon dioxide reduction and conversion to methane process employing MgH_2 was achieved experimentally showing great cobalt catalytic activity [1]. Hence, plane-wave periodic density functional theory was applied to comprehend the intermolecular short-range interactions between carbon dioxide and the catalyzed and non-catalyzed hydride. Large affinity of CO_2 gas molecules with the hydride material was demonstrated according to the interaction energies, adsorption distances and further material properties, thus, confirming the carbon dioxide capture process. When introducing cobalt atoms to the simulated hydride as catalyzer, suitable carbon dioxide capture performance was demonstrated. Hydrogen molecules were also considered on the capture means for a theoretical approach in the methane formation proving the CO_2 conversion to methane from a theoretical point of view.



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D.5. Multiscale and multiphysics modelling of materials

ID 34 - Multiscale modeling of dendritic solidification with convective transport in the melt

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Metals and alloys exhibit complex solidification microstructures, such as dendrites, which arise from a subtle interplay between phenomena on different scales: from atom attachment kinetics to macroscopic heat and species transport. Hence, solidification modeling techniques require the bridging of a wide range of length and time scales. Resulting computational challenges are even more dire in the presence of inevitable buoyancy-induced fluid flow in the liquid phase.

The Dendritic Needle Network (DNN) model was designed to overcome the scale limitations of phase-field (PF) models and quantitatively predict dendritic crystal growth in undercooled binary alloys. We recently extended the isothermal formulation of the DNN model with convective flow in three dimensions [1]. The morphology of equiaxed dendrites in a forced flow differs substantially from the case of a purely diffusive regime and from similar two-dimensional simulations.

Here, we present an extended study of the effect of the dendritic crystal orientation with respect to the flow direction. The current study provides a broader insight into the effect of fluid flow on the morphological selection of equiaxed dendritic grains, hence paving the way to computationally-efficient simulations of dendritic growth at scales and conditions relevant to experimental solidification processes.

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ID 36 - A GPU-Parallelized Julia Implementation of a Phase Field Model

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An implementation of the Nucleation Benchmark developed by the Center for Hierarchical Materials Design (CHiMaD) [1] is written in the Julia programming language [2]. The nucleation benchmark describes both heterogeneous nucleation and homogeneous nucleation. In the homogeneous nucleation case, Johnson-Mehl-Avrami-Kolmogorov (JMAK) theory is used to quantify the rate of phase transformation. Beyond what is specified in the benchmark, statistical results have been gathered to show the distribution of Avrami slopes described by JMAK theory. The problems in the benchmark were expanded to three dimensions. In certain cases, these 3D results matched the theoretical Avrami slopes. In the cases that the theoretical Avrami slopes are not matching the results, a study of how the local curvature affects the rate of seed growth was performed. Finally, a scaling study shows the influence of using multiple GPUs on the Julia-coded phase field model.

Portions of this research were conducted with the advanced computing resources provided by Texas A&M High Performance Research Computing.

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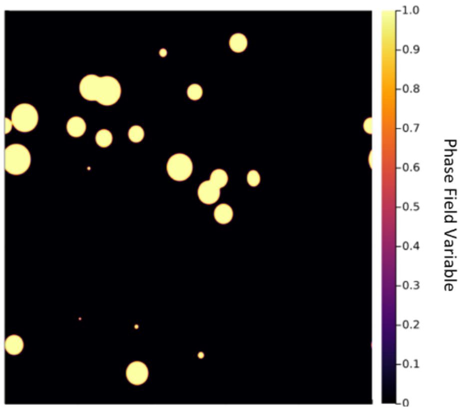

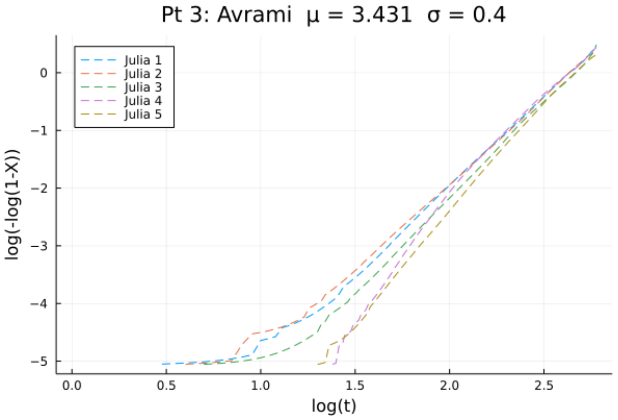

ID 102 - Through Process Modelling of Sustainable Aluminium

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Through process modelling (TPM) can assist in optimizing the thermomechanical processing of rolled aluminium plates and sheets by minimizing trial-and-error experiments. TPM can also accelerate the introduction of new alloys or alloys with high recycled content. A key component of a TPM for aluminium rolling is the prediction of the deformation and recrystallization behaviour. However, deformation and recrystallization are strongly affected by local heterogeneities in aluminium (e.g. second phase particles), especially for recycled alloys with high impurity content. To capture the effects of these heterogeneities, a full field crystal plasticity (CP) model has been used, enabling local strain distribution to be predicted. This model has been applied to particle containing alloys to study the effect of changing distribution of constituent particles, consistent with increased recycled content. The predicted local strain distribution from the CP model is used to initialize a full field recrystallization model. However, the disadvantage of full field modelling is the intense computational requirements, leading to long-run times which is undesirable in industry. To this end, strategies for extracting critical information from the CP model for input into fast running semi-empirical recrystallization models are examined.

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ID 151 - Phase field study of columnar grains growth competition under multi-dimensional temperature fields

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Directional solidification conditions occur in several industrial materials processes, like gas turbine blade casting, fusion-based welding, and metal additive manufacturing. The growth competition of columnar grains greatly determines microstructural features of the final microstructure, e.g. interdendritic/intercellular spacings, solute segregation, and grain texture, which have a significant impact on the final mechanical properties.

The advent of quantitative simulation methods, in particular the phase-field method, resulted in a renewed interest for the study of dendritic grain growth competition – via impingement, elimination, and side-branching mechanisms[1,2,3] – and the resulting selection of grain boundaries and grain textures. However, most studies have focused on simplified one-dimensional thermal fields. Yet, in practice, the isotherms are not planar, but the effect of the multi-dimensional temperature field remain poorly understood.

Here, we use a computationally-efficient (GPU-parallelized) quantitative phase-field model to study the growth competition of columnar grains within non-planar temperature fields. Starting with two-dimensional simulations, we study the effect of the isotherms shape and curvature, using conditions relevant to actual welding processes, in order to identify features of the thermal field that result in remarkable changes in the selected grain texture.

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This investigation is supported by the Spanish Ministry of Science through an International Collaboration Fellowship MiMMoSA (Ref. PCI2021-122023-2B) and a Ramón y Cajal Fellowship (Ref. RYC2019-028233-I).

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D.6. Modelling of Additive Manufacturing

ID 172 - Characterisation of the thermoelastic properties of 3D printed ZTA technical ceramics by Asymptotic Expansion Homogenization

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Nowadays, alumina and zirconia materials are more and more used in the fabrication of the so-called zirconia-toughened alumina (ZTA) technical ceramics, such as supports for catalysts and thermal or electrical insulators, among others. These applications require tight limits in ceramics dimensions and properties, with consequent high-quality control by the manufacturing companies. Additive manufacturing (AM) appears to be a facilitator technology in the manufacture of such components. However, the consequences of AM implementation in the industrial system, if not supported by fundamental research, can lead to consumption and resources increases, contrary to expectations. Therefore, the fine tuning of manufacture of components based on alumina (A), zirconia (Z) or a mixture of both (AZ) by additive manufacturing requires an analysis of the parameters that influence their final properties. In this context, this work aims at the development of (numerical-based) prediction models for the anisotropic thermal conductivity, thermal expansion and elasticity tensors for ZTA ceramics (reticulated sintered alumina-zirconia specimens), considering the influence of both material (alumina/zirconia ratio) and geometrical (fibres diameter/spacing and overlapping ratios) effects at the macrostructural (macroscopically heterogeneous and anisotropic porous lattice) level, considering both Asymptotic Expansion Homogenization (AEH) and Finite Element Method.

Acknowledgements
This work was supported by the project “TAMAZ3D - Development of a Decision Support Tool for Additive Manufacturing of Alumina-Zirconia 3-D structures” (POCI 01-0145-FEDER-030493), and by projects of CICECO (UIDB/50011/2020 & UIDP/50011/2020) and TEMA (UID/EMS/00481/2020), financed by national funds through the FCT/MEC and when appropriate co-financed by the European Regional Development Fund (ERDF) under the PT2020 Partnership Agreement.

ID 225 - Multiscale modeling of powder bed fusion process for Inconel 718

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Integrated Computational Materials Engineering (ICME) is one of the most promising methodologies for speeding up the development of new alloys and processing routes. ICME relies heavily on integrating different modeling tools, which are applicable to different length/time scales and/or distinct physics. Despite the fact that a diverse set of models has been produced, the efficient coupling of these models remains a major challenge.

We developed a multi-scale modeling approach to simulate and formation of microstructure in the melt pool size during powder-bed laser fusion (LPBF) processing of nickel-based superalloys by combining CalPhaD with finite elements (FE) and phase-field (PF) methods. CalPhaD is used for calculating thermophysical data (such as phase diagram features, heat capacity, etc.) used as input to other models. The temperature field under various processing conditions is determined using the FE modeling at the macroscale, which is then used as input for PF simulations of dendritic microstructure formation at the melt pool scale, while retaining the microscopic resolution of individual dendrites.

We apply the framework to simulate LPBF of an Inconel 718 superalloy. We discuss the impact of temperature-dependent properties, the importance of accounting for various properties in the powder-bed, and the microstructure model's key limiting assumptions (e.g. binary alloy approximation, limited solute trapping, two-dimensional slicing of the temperature field). This work provides a deeper insight into microstructure selection via polycrystalline growth competition under realistic LPBF conditions.

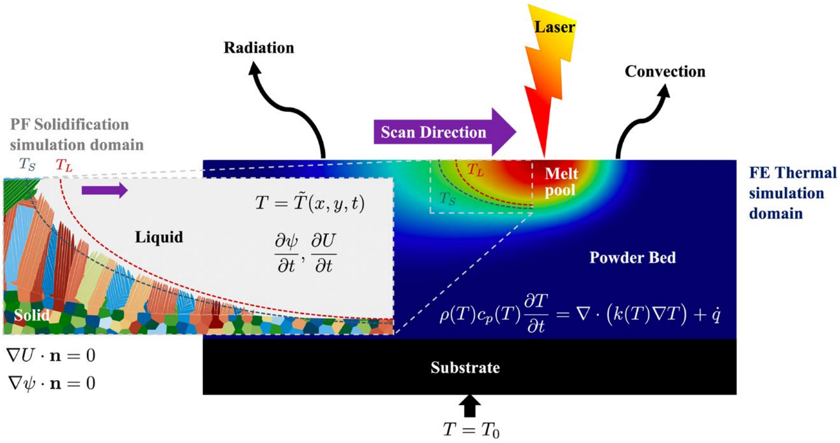

Fig. 1: CalPhaD thermodynamics, Finite Element thermal simulation of SLM processes, and Phase-Field solidification simulation at the melt pool scale as the three main components of the multiscale modeling technique.

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E.1. Critical raw materials

ID 215 - Using Advanced Image Processing to Characterise the Performance of Metallurgical Coke in Blast Furnaces

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Blast furnace operation relies heavily on coke quality and therefore the accurate characterisation of coke can lead to the stable operation of a very expensive asset. The use of petrographic analysis to identify morphologies in metallurgical coke can indicate several key parameters related to its performance in blast furnaces. These include reactivity, which is an important factor for determining the coke's residence time in the furnace and the formation of the hearth layer; and strength, which is indicative of the coke's ability to withstand the weight of the ferrous burden as it reduces in the furnace.

Standard reactivity and strength testing (such as CSR, CRI, Irsid or Micum tests) can take up to 6 hours to perform, while petrographic analysis can be conducted in under an hour. Furthermore, coke petrography can highlight key operational elements of coke-making. Automating the analysis of petrographic analysis can help further speed up characterisation and the communication of these results to lay audiences which, in turn, can help translate these results into operational indicators far quicker. Additionally, a novel technique, Visual Reactivity Analysis (VRA), can be used to model the thermal properties of coke along with more conventional ThermoGravimetric Analysis (TGA) techniques. VRA allows the observation of coke combustion in real time. The change in shape of the sample can be quantified using image analysis techniques and used to infer its reactivity at any given time, thereby granting access to a wealth of information on how coke combusts over the complete range of temperatures and atmospheres during the experiment. A combination of these three techniques can thus be deployed to gain invaluable information on the properties of coke.

Acknowledgement: This research is funded and supported by Engineering and Physical Sciences Research Council (EPSRC), Tata Steel Strip Products UK (TSSPUK) and the University of Nottingham. The project is an Engineering Doctorate at the Centre for Doctoral Training in Carbon Capture and Storage and Cleaner Fossil Energy at the University of Nottingham, grant number EP/L016362/1. The authors would like to thank EPSRC, Tata Steel Strip Products UK (TSSPUK) and the Centre for Doctoral Training in Carbon Capture and Storage and Cleaner Fossil Energy for financial support and guidance.

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E.4. Bio-based materials

ID 134 - Alginate as an additive for switchable and improved biodegradation of biocomposites

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In order to substitute fossil-based polymers, bio-based and biodegradable bioplastics such as PLA or PHB are a common alternative. However, the biodegradation process does not start immediately after release into the environment and requires varying amounts of time. Therefore, an improved and consciously induced (switchable) biological degradation of biocomposites by creating a novel, bio-based and transferrable material system of known bioplastics with alginate is presented here.

Alginate beads and/or fibers are added to compounds including PLA, PHB or PLA/PHB with microfibrillated cellulose. The expansion of the alginate in the compound, caused by the replacement of calcium ions with sodium ions of sea water or soil (environment), is expected to increase the surface available for the microorganisms, the water uptake and therefore the speed of the biodegradation. Without this stimulus, the biocomposite remains stable for applications such as food packaging.

The beads and fibers were prepared in a $CaCl_2$-bath. After drying, they were included in a matrix of PLA, PHB or PLA with microfibrillated cellulose via solution-precipitation-method with dichloromethane. After seven days in artificial seawater a six times weight gain was observed and the mechanical properties decreased, which leads to a better physical and biological degradation.

The biodegradation tests currently running in respirometers, will allow us to compare the biological degradation of PLA, PLA-cellulose-composites and PLA-cellulose-alginate-composites. Additional nutrients such as sugars, amino acids or even bacteria stored in alginate could further improve the biodegradation.

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ID 152 - Molecular weight distributions and critical molecular weights in biodegradation processes of polymers

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The molecular weight is one of the most important factors governing the properties and biodegradation of polymers. Some critical molecular weights for the loss of mechanical properties, water solubility and the beginning of mineralization have been published. Usually, a single number for the molecular weight is reported while the whole molecular weight distribution could be used for a more detailed investigation of the biodegradation process.

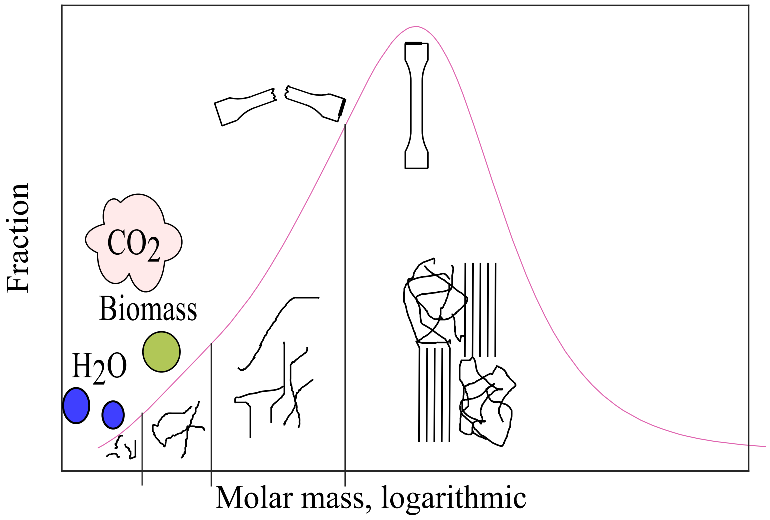
We will correlate molecular weight to changes in mechanical properties, biodegradability, water solubility and crystallinity in consideration of the whole distribution. The binning of the molecular weight distribution is introduced as a valuable tool for the interpretation of biodegradation processes.

Polymer granulates of poly (lactic acid), poly (butylene adipate-co-terephthalate) and poly (butylene succinate) as well as tensile bars were degraded to yield different molecular weights. Tensile tests were performed and size exclusion chromatography was applied to the samples and to hydrolysates to determine water solubility. The crystallinity was analyzed by X-ray diffraction. The biodegradation in soil is investigated by capturing the evolving CO_2 in a KOH-solution with subsequent titration with HCl.

Preliminary results show limits of molecular weight for water solubility and mineralization as well as molecular weight ranges for loss of mechanical properties.

Knowing critical molecular weights will enable tailoring biodegradable products towards their desired functionalities and expected lifetimes for specific applications in a particular environment.

This work was supported by the ‘SuPer (Sustainable Polymer) Hybrid Turf’ project of the “Zentrales Innovationsprogramm Mittelstand’, Federal Ministry for Economic Affairs and Climate Action, Germany.



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E.5. Materials circular economy

ID 81 - Self-Reduction Behavior of Finnish and Italian Jarosite Plus Blast Furnace Sludge Mixtures

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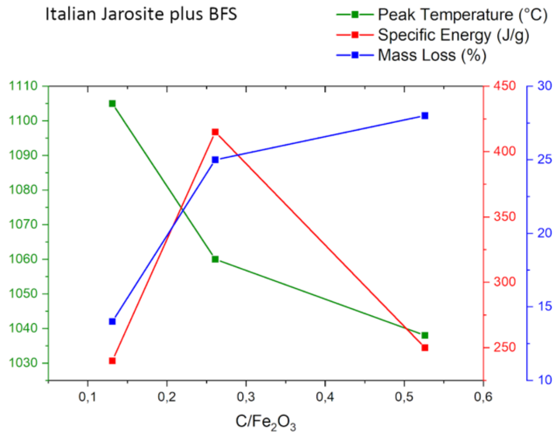
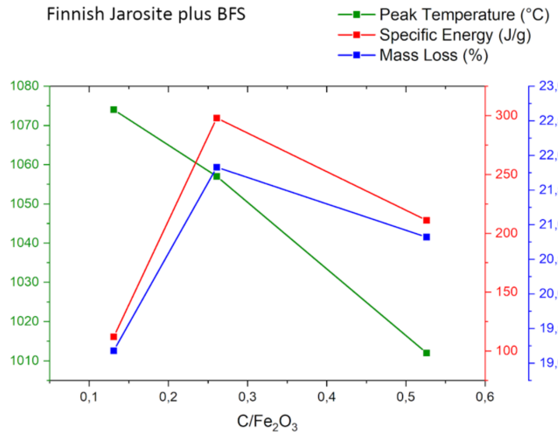
Underlining the importance to develop new technologies and procedures for the valorization and recycling enhancements of industrial byproducts, the present work proposes the utilization of blast furnace sludges (BFS) as reductant for jarosite. Jarosite is considered a dangerous waste derived from the hydrometallurgical route of zinc production due to the presence of hazardous elements, with variable chemical composition dependent from the production plant. The aim of this study is to chemically compare the thermal behavior of an Italian and Finnish jarosite, to define the best BFS/jarosite ratio for the production and subsequent utilization of self-reducing briquettes and to highlight the significance of the jarosite production site on the reduction behavior. Three mixtures per jarosite with constant binary basicity (BI=0.504) and incremental BFS/jarosite ratio were prepared (A=16.51 %wt., B=35.93 %wt. C=81.9 %wt.). The chemical composition of the two jarosites revealed the necessity of different fluxing agents for the Italian and Finnish plus BFS mixtures, quartz for the former and lime for the latter. The best reducibility yield for both the Italian and Finnish jarosite plus BFS mixtures were obtained for the B mixture, corresponding to a C/Fe_2O_3=0.261 ratio. Finally, the data of the Finnish jarosite mixtures were compared with a theoretical model developed for the Italian jarosite reduction behavior, confirming its applicability and coherence also in the case of the Finnish jarosite.





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ID 229 - Direct Energy Deposition of AISI 303 Stainless Steel Powder Particles from Recycled Machining Chips

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Chips resulting from machining processes in metalworking industries can be considered as “secondary” raw materials instead of being consumed as scraps in foundry industries, i.e. transforming chips into powder particles for additive manufacturing (AM) industry as an adding value. Components produced by AM such as direct energy deposition (DED) can substitute those produced by conventional methodologies, taking advantage of producing complex geometries, small series production, and repairing damaged parts. This sustainable approach can be implemented in metal industries supported by AM, creating a closed life cycle [1].

This study investigates the transformation of AISI 303 stainless steel chips to powder particles for DED process. Disc milling was applied for powder production. The resulting powder particles were characterized to identify particle size, particle size distribution, and shape. This characterization was performed using sieving and image analysis by optical and scanning electron microscopies. Moreover, loose density, tap density, and flowability of milled particles were measured. This study was proceeded by parametrization of depositions on an AISI 1045 substrate considering laser power, scanning speed and powder feed rate [2]. Deposited lines were evaluated visually and through metallography to select an optimized bead geometry and dilution. Then, 20x20 mm^2 planes of AISI 303 (one and three layers) were produced by printing lines continuously with an overlapping of 35 %, applying offset-in and zig-zag strategies, with a 90° rotation between subsequent planes for the latter one. Evaluations on polished cross-sections included porosity, microstructure, the state of substrate-deposition interface and heat affected zone, and microhardness measurements.

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