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Book of Abstracts

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List of abstracts

Number	Type	First Name	Family Name	Title	Company/Institute/Laboratory	City	Country
1	Oral	Serhii	Ivanchenko	Development of rheology-related suspension structure parameters	Frantsevich Institute for Problems of Materials Science NASU	Kyiv	Ukraine
2	Poster	Tomé	Silva	Production of lightweight geopolymeric structures for thermal insulation	CICECO-Aveiro Institute of Materials	Aveiro	Portugal
3	Poster	Tomás	Seixas	Mechanosynthesis of calcium phosphates for biomedical applications from chicken eggshell and phosphoric acid	CeFEMA - IST	Lisboa	Portugal
4	Oral	Suzana	Inkret	Comparison of the influence of bovine serum albumine and chitosan on the formation of calcium phosphate and silver nanoparticles composites	Institut Ruđer Bošković	Zagreb	Croatia
5	Oral	Petra	Šimonová	Elastic properties of SnO2- and ZnO-based composite ceramics	University of Chemistry and Technology Prague	Prague	Czech Republic
6	Oral	Samir	Salmanov	Cold sintering of lead-free perovskites	Jožef Stefan Institute; Jožef Stefan International Postgraduate School	Ljubljana	Slovenia
7	Oral	Szymon	Salagierski	Borate bioactive glasses modified with copper ions: structural and bioactivity investigation	AGH University of Science and Technology	Cracow	Poland
8	Oral	Ricardo	Mineiro	Experimental validation of calculated phase diagrams in the system BN-TiCN-Ni	University of Aveiro	Aveiro	Portugal
9	Poster	Olha	Kovalenko	Obtaining of the rod-shaped $BaTiO_3$ nanoparticle and its properties	LLC NanoTechCenter, National Academy of Sciences of Ukraine	Kyiv	Ukraine
10	Oral	Vojtěch	Nečina	Transparent Eu:La2Zr2O7 ceramics – lithium fluoride strikes again	University of Chemistry and Technology, Prague, Department of Glass and Ceramics	Prague	Czech Republic
11	Oral	Narayan	Sahoo	Direct Laser writing based surface texturing for enhanced adhesion between zirconia(3Y-TZP) and resin-matrix cement	CMEMS, UNIVERSITY OF MINHO PORTUGAL	Guimares	Portugal
12	Oral	Muthusundar	Kumar	Influence of liquid nature on the densification of Hydroxyapatite powders through Cold Sintering Process	University of Mons (SMPC)/Polytechnique University f Huts-de-France (CERAMATHS))	Mons	Belgium
13	Poster	Mónica	Faria	Mechanical performance of silicon nitride parts made by additive manufacturing	CICECO	University of Aveiro	Portugal
14	Poster	Milan	Vukšić	Additive Manufacturing of Ceramic Supports for Photocatalytic Degradation of Hydroxychloroquine	Jožef Stefan Institute	Ljubljana	Slovenia
15	Oral	Micaela	Videla-Trevin	Synthesis of AB2S4:Eu2+ scintillator materials by mechanosynthesis and hydrothermal routes for ion detection diagnosis in nuclear fusion reactors	Unversity of Seville	Seville	Spain
16	Oral	Mathilde	Maillard	Optimisation of mechanical properties of 3d printed alumina : linking rheology and printing parameters	LVTS - INSERM	Paris	France
17	Poster	Marinélia	Capela	Can ceramic products use eggshell waste as secondary raw material?	CICECO - Aveiro Institute of Materials	Aveiro	Portugal
18	Oral	Mariana	Almeida	Development of geopolymeric structures by additive manufacturing to remove pollutants from wastewater	University of Aveiro	Aveiro	Portugal
19	Poster	Louis	Masters	Defect-Free Hybrid Additive Manufacturing of Advanced Ceramics	University of Leeds	Leeds	United Kingdom
20	Oral	Lucie	Kotrbová	Analytical modeling of the effective thermal conductivity of alumina-based ceramic matrix composites with interfacial thermal resistance	University of Chemistry and Technology Prague, Department of Glass and Ceramics	Prague	Czech republic

21	Oral	Katelyn	Kirchner	Beyond the Average: The Criticality of Spatial and TempOral Fluctuations in Oxide Glasses	The Pennsylvania State University	State College	United States
22	Poster	Kinga	Kowalska	Bioceramic bone cements modified with bioglass	AGH University of Science and Technology	Krakow	Poland
23	Oral	Jovana	Zvicer	HOW NOVEL BIOMATERIALS BASED ON BIOACTIVE GLASS AND β-TRICALCIUM PHOSPHATE CAN BE EVALUATED UNDER PHYSIOLOGICALLY RELEVANT CONDITIONS?	Faculty of Technology and Metallurgy, University of Belgrade	Belgrade	Serbia
24	Oral	Justyna	Ignaczak	Electrophoretically deposited iron doped manganese- copper spinel coating for prevention of chromium poisoning in Solid Oxide Fuel Cells	Gdańsk University of Technology	Gdańsk	Poland
25	Poster	Juan Ramón	Marín-Rueda	Production of TiO2 ceramic supports via FFF	3DENERMAT (UCLM)	Albacete	España
26	Oral	José Miguel	Ramos-Fajardo	Optimization of Full Ceramic LFP/C cathodes for FFF	3D-Enermat (UCLM)	Albacete	Spain
27	Poster	Joaquim	Rodrigues	Thermodynamic Simulation and Design of Microstructure in Low cBN Composites Produced by Spark Plasma Sintering	University of Aveiro	Aveiro	Portugal
28	Poster	João	L. Miranda	Alternative sintering of K1-xNaxNbO3 (KNN) ceramics: preliminary data on the application of the ultrafast high-temperature method	CICECO	Aveiro	Portugal
29	Oral	João	Carvalheiras	Real wastewater treatment by means of red mud based porous alkali activated materials	University of Aveiro	Aveiro	Portugal
30	Poster	Jesse	Hinricher	Cycling demonstration of Sequential Deposition Synthesized lithium garnet films in full batteries	Massachusetts Institute of Technology	Cambridge, MA	USA
31	Oral	Jaime	González Cuadra	Thin films of transparent oxides with multifunctionality, suitable for both photocatalysis and antimicrobial uses	University Jaume I/ University of Aveiro	Castellón de la Plana, España	España
32	Oral	Isabel María	Peláez Tirado	Manufacture of YSZ-based electrolytes for SOFC by 3D printing	University of Castilla-La Mancha/Renewable Energy Research Institute/3DENERMAT	Albacete	España
33	Oral	Nicola	Gilli	Multi-phase (Zr,Ti,Cr)B2 solid solutions: preparation, microstructure and local properties	CNR-IMM	Bologna	Italy
34	Poster	Marija	Egerić	Assessment of optimum ratio of Metal Organic Framework and active carbon in composites for photocatalytic removal of Congo Red dye	Vinča Institute of nuclear sciences	Belgrade	Serbia
35	Poster	Dmytro	Vedel	Obtaining of ceramics based on (Zr, Mo)B2 solid solution	Frantsevich Institute for Problems in Materials Science	Kyiv	Ukraine
36	Oral	Dariia	Chernomorets	Control of powders morphology as a way of improving of Y2O3 ceramics properties	Institute for Single Crystals of NAS of Ukraine	Kharkiv / Faenza	Ukraine / Italy
37	Poster	Dan	Davie	Hybrid Additive Manufacture and Materials for Advanced Functional Ceramics	University of Leeds/Institute of Design and Robotics Optimisation/Future Manufacturing Processes	Leeds	United Kingdom
38	Poster	Damian	Koszelow	Lifetime extension of high chromium ferritic porous alloys by application of ultra-thin alumina coatings	Gdańsk University of Technology	Gdańsk	Polska
39	Oral	Cristina	Ojalvo Guiberteau	Spark plasma sintering of TiCN at low temperature with Si aids	Materials and Ceramic Engineering (CICECO)	Aveiro	Portugal
40	Oral	Arijeta	Bafti	Optimisation of environmentally friendly tool for geopolymer and zeolite preparation from kaolin	Faculty of Chemical Engineering and Technology, University of Zagreb	Zagreb	Croatia

41	Poster	Ana	Caetano	Rheological behaviour of glazes for stoneware	CICECO-Department of Materials and Ceramic Engineering University of Aveiro	Aveiro	Portugal
42	Poster	Nima	Amousa	Multifunctionalization of SS316L surfaces by immobilization of nano-thin MXenes-films for improving of biological responses	Chair of Ceramics, RWTH Aachen University	Aachen	Germany
43	Oral	Aleksander	Ucakar	Consolidation of ceramic permanent magnets by Radiation assisted sintering (RAS) in WC die	Jožef Stefan Institute	Ljubljana	Country
44	Oral	Abderrahim	Lahlahi Attalhaoui	New fast routes for the synthesis of soft ferrites by microwave decomposition of hydrotalcites for functionalization of porcelain substrates	Universitat Jaume I	Castellón de la Plana	España
45	Oral	Oxel	Urra Sanchez	Material thermal extrusion of conductive 3D electrodes using highly loaded graphene and graphite colloidal feedstock	Institute of Ceramic and Glass	Madrid	Spain
46	Poster	Pablo Jose	Alarcon-Meseguer	Obtaining superparamagnetic MnO nanoparticles as contrast agents	Instituto de Cerámica y Vidrio	Madrid	Spain
47	Oral	Ana	Castellanos-Aliaga	Effective immobilization of TiO2 nanoparticles for water remediation purposes	Instituto de Cerámica y Vidrio-CSIC	Madrid	Spain
48	Poster	Martina	Kocijan	Additive Manufacturing of Ceramic Supports for Photocatalytic Degradation of Hydroxychloroquine	Faculty of Mechanical Engineering and Naval Architecture, University of Zagreb	Zagreb	Croatia
49	Poster	Albina	Murashko	Bioresorbable pyrophosphate ceramics produced by stereolithographic 3D printing	Lomonosov Moscow State University Faculty of Materials Science Laboratory of inorganic materials science	Moscow	Russia
50	Poster	Bernardo	Vidal	Development of waste-based colored glazes for stoneware	University of Aveiro	Aveiro	Portugal
51	Poster	Jacopo Matteo	Tabaglio	Study of WC-based inks for 3D printing	Politecnico in Milan	Milan	Italy
52	Poster	Diana	Suárez	Ion Migration in Glassware and Crystal	University of Aveiro	Aveiro	Portugal
53	Poster	Inês	Vieira	Recovery of metallurgical industry waste in ceramic products	CICECO - Aveiro Institute of Materials	Aveiro	Portugal

Development of rheology-related suspension structure parameters

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Suspension structure analysis by direct methods requires specialized, complex equipment and is rather complicated, especially in the case of the dynamic change of the structure under shear. However, it is possible to calculate parameters that indirectly indicate a difference in the suspension structure using rheological methods.

The flow curve's Power Law model alongside Newton's model for two parallel plates was used for calculating viscosity from a flow curve for a fixed shear rate. This approach allows for comparing suspensions viscosity measured under different conditions.

Power Law's index n can be used to describe suspension's thinning or thickening ability under shear. It helps to predict a deformation or in-flow orientation of the suspension's structural elements.

Thixotropy and rheopexy degree indicates a presence of a regular structure network spread inside the suspension. It was calculated using integration by trapezoids of the area under forward and reverse flow curves. For a proper comparison of the flow curves hystereses measured under different conditions, a specific method of normalization was proposed. This modification allows using a T/R degree more as a structural parameter rather than just a comparative one.

A model based on the Peclet number for establishing the suspension's structural elements size was proposed. The Peclet number was calculated for a model suspension of particles with known size and a model which includes spear stress, viscosity, and the temperature was built.

The set of these parameters can help with the description of the suspension structure and its change under shear.

Production of lightweight geopolymeric structures for thermal insulation

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The continuous growth of the world population and a forecast of reaching 10.1 billion by 2050, implies an increasing challenge on providing sustainable and inclusive future development. Construction and reparation of residential and non-residential infrastructures is an example of a sector where awareness is raised. It is estimated that 127 EJ of energy was consumed in building operations like space heating and cooling, water heating, lighting, and cooking in 2020 and in the following year, space and water heating alone were responsible for 3.5 Gt of direct CO_2 emissions. Thus, the implementation of more sustainable options is crucial in this sector. To assist the decarbonization process, heat pumps (HPs) emerge as crucial equipment. HPs can convert energy from an external heat source, such as air or water, into useful heat for hot water supply and/or residential and commercial building space heating, in one of the most energy-efficient ways. HPs stock is expected to reach 600 million by 2030 to get on track with Net Zero Emissions. However, HPs functional units are potential sources of noise pollution and heat generation. Currently, thermal insulation is guaranteed using expanded polypropylene (EPP). Despite the interesting properties of this foam, it has an embodied energy between 77.4 and 85.3 MJ and a CO_2 footprint of 3.18 - 3.5 kg for each kg produced.

Geopolymers, a subset of alkali-activated materials formed by a chemical reaction between aluminosilicates with alkali metals silicate under strongly alkaline conditions, allow the incorporation of various waste streams as precursors to produce high added-value products. This work aims to produce foamed geopolymers incorporating fly ash from biomass combustion as a precursor and to evaluate its thermal insulation properties to substitute EPP currently used. Foamed geopolymers are expected to present better thermal insulation properties while representing a more sustainable option.

Mechanosynthesis of calcium phosphates for biomedical applications from chicken eggshell and phosphoric acid

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Calcium phosphates, CaP, can be used to promote healing of bone defects. Given their similarity to the inorganic portion of most natural hard tissues, synthetic CaP display biocompatible and bioactive properties which make them ideal for Bone Tissue Engineering applications.
High energy milling has proved to be an adequate route for CaP production, namely hydroxyapatite - HA, given the local increase in reactivity and improved kinetics due to energy transfer upon ball impact. Deriving Ca from a biogenic source provides an advantage regarding bioactivity of produced CaP. Trace ions present in biogenic mineral structures are known to improve osteocell activity compared to pure compounds. One such biogenic Ca source is chicken eggshell.
In the group's work, calcite from chicken eggshell is used as Ca source and H_3PO_4 as P source. Batches of clean, membrane-stripped, eggshell, H_3PO_4 and deionized H_2O are submitted to high energy ball milling. ZrO_2 vial and balls are used and milling speed is kept at 400 rpm. The influence of milling time; wt. % H_2O; Ca/P ratio; and CO_2 pressure build up inside the vial are tested. The effect of pre-milling eggshell particle size and of post-milling treatment are also studied.
Phases present after milling and drying are assessed using micro-Raman confocal spectroscopy and X-ray Diffraction. Particle morphology and size are studied by Scanning Electron Microscopy.
Overall results indicate that, within the parameters tested range, CaP formation reactions follow a direct route: $CaCO_3$ → brushite → HA, and that reactions are initiated within the first milling minutes. Relieving CO_2 pressure seems to accelerate HA production, whereas higher water content delays reaction onset. Lower Ca/P ratios promote reaction completion; post-milling quenching with acetone stops reaction progression and powder agglomeration. These results are instrumental to establish an efficient and reproducible HA production route via high energy milling.

Comparison of the influence of bovine serum albumine and chitosan on the formation of calcium phosphate and silver nanoparticles composites

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Due to their similarity to the mineral component of bone tissue, calcium phosphates (CaPs) are receiving special attention in the development of novel bone biomaterials [1]. To improve their biological and mechanical properties, CaP composites with nanomaterials and/or biomacromolecules are emerging [2]. Since nanomaterials can have antimicrobial or magnetic properties, such composites can be considered as true multifunctional materials [3]. However, for such materials to be successfully applied, the interactions between their components must be understood.
Therefore, this study investigated the formation of CaPs in the presence of two classes of additives, differently stabilized silver nanoparticles (citrate cit-AgNPs; poli(vinylpyrrolidone), PVP-AgNP; sodium bis(2-ethylhexyl) sulfosuccinate, AOT-AgNP) and two biomacromolecules, bovine serum albumin (BSA) and chitosan (Chi). In the absence of additives, CaPs precipitated in two steps, as potentiometric measurements showed. Amorphous calcium phosphate (ACP), formed in the first step, transformed to a mixture of poorly crystalline calcium-deficient hydroxyapatite (CaDHA) and a small quantity of octacalcium phosphate (OCP). BSA and Chi inhibited ACP transformation both in the absence and presence of AgNPs, with Chi being much stronger inhibitor. In the presence of additives, a change in OCP amount was observed, the type and extent of change depending on the specific combination of AgNPs and biomacromolecules. Furthermore, in the presence of both BSA and cit-AgNPs, at two higher BSA concentration, a new phase, calcium hydrogenphosphate dihydrate, was detected DCPD. TEM and SEM micrographs revealed morphological changes of ACP and crystalline precipitate induced by the presence of AgNPs and both biomacromolecules.
The obtained results may contribute to the development of low-temperature procedures for the synthesis of composites.

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Elastic properties of SnO2- and ZnO-based composite ceramics

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Ceramics based on tin oxide (SnO_2) and zinc oxide (ZnO) are important for various types of applications, including varistors, electrodes, gas sensors or catalysts. SnO_2 ceramics is a very specific material because evaporation-condensation (a non-densifying sintering mechanism) is usually dominating during sintering. On the other hand, in ZnO ceramics the dominant sintering mechanism is densifying, which means that it is much easier to obtain a highly dense material by conventional sintering compared to tin oxide ceramics. Combining ZnO and SnO_2 it is possible to create a composite containing the spinel phase $SnZn_2O_4$, which could significantly improve the properties of the material, including mechanical properties.
In this contribution we present experimental results obtained for pure SnO_2 and ZnO ceramics as well as its composites. Samples were prepared by uniaxial pressing and subsequent conventional sintering up to 1400 °C. Porosity and bulk density were measured on sintered samples together with the microstructure characterization by image analysis conducted on SEM micrographs. It was found that the porosity in SnO_2 samples remained constant around 50 %, whereas in ZnO samples the porosity varied from 2 to 30 % and in the composite the porosity varied from 13 to 50 %. X-ray diffraction confirmed the purity of the ZnO and SnO_2 ceramics and the formation of $SnZn_2O_4$ spinel in the composites. Elastic properties were measured by the impulse excitation technique at room temperature (and also at higher temperatures in case of pure ZnO and SnO_2 ceramics). It is shown that both tin oxide and zinc oxide ceramics show a decrease of Young's modulus with temperature and an increase of the latter during sintering. Experimentally measured elastic properties were compared with the model predictions of the porosity dependence of Young's modulus and the unknown Young's modulus of $SnZn_2O_4$ spinel was roughly estimated by back-calculation.

Cold sintering of lead-free perovskites

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Most widely used ferroelectric perovskites contain lead, which is undesirable from an environmental point of view. This is the main reason why researchers in the field of ferroelectrics science are trying to find environmentally friendly ferroelectric perovskites. Potassium sodium niobate $(K_{0.5}Na_{0.5})NbO_3$ (KNN) and bismuth ferrite $BiFeO_3$ (BFO) are among the important lead-free perovskites. Conventional sintering of these materials can be challenging, in particular, conventional sintering of KNN (which requires about 1100 °C) leads to an inhomogeneous microstructure and the formation of secondary phases, while conventional sintering of BFO (about 800 °C) requires a narrow temperature range because secondary phases may form.

One of the solutions to overcome the problems associated with conventional sintering is to use a different technique that allows KNN and BFO to be sintered at drastically low temperatures. The Cold-Sintering Process (CSP) makes it possible to obtain the bulk ceramic sample at uniaxial pressure, which can reach 600 MPa, and at 300 °C, adding compounds that serve as a transient liquid phase.

In our research, we cold sintered KNN and BFO with a mixture of the hydroxides KOH and NaOH. The analysis of the microstructure with the microscopes SEM and TEM shows the dense structure and the size of the grains in the same range as the grains of the starting powder used. The measurement of ferroelectric and dielectric properties showed that the cold-sintered KNN and BFO have lower electrical conductivity, higher dielectric constant and higher dielectric breakdown strength compared to the conventionally sintered ones, making the cold sintering process interesting for tailoring the functional properties of ferroelectrics.

Borate bioactive glasses modified with copper ions: structural and bioactivity investigation

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Bioactive borate glasses (BBGs) create new possibilities in the field of tissue engineering due to their high biocompatibility, gradual degradation, bioactivity and positive effect on bone tissue regeneration. Due to the ease of modifying the composition of bioactive glasses, their properties and release of therapeutic ions can be controlled, which allows them to be used for specific clinical applications. An example of therapeutic ions may be Cu^{2+} copper ions, which promote osteogenesis, angiogenesis and endothelial cell proliferation processes, which are important stages in tissue healing.

In this study BBGs from the B_2O_3–CaO–P_2O_5 system modified with CuO were obtained by sol-gel method and melting technique. For BBGs obtained by the sol-gel synthesis, two different boron oxide precursors were used – boric acid (H_3BO_3) and triethyl borate (TEB). The content of B_2O_3 and P_2O_5 in the BBGs was constant and amounted to 40 and 6 mol% respectively, while CuO was introduced in amounts of 3 and 5 mol% in place of CaO.

The characterization of the obtained BBGs was aimed at assessing the influence of the synthesis technique and the presence of modifying element – copper on their microstructure, morphology and bioactive properties *in vitro*. The BBGs structure and their ability to crystallization was monitored with FTIR and XRD, while morphology and bioactivity were investigated using SEM microscopy.

Based on the conducted study, it was found that the synthesis technique and the presence of copper in the composition of BBGs caused significant structural and morphological changes. Melted BBGs had a completely amorphous character and lower surface area compared to sol-gel bioglasses. Moreover, all tested materials had an excellent bioactive properties.

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Experimental validation of calculated phase diagrams in the system BN-TiCN-Ni

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Cubic Boron Nitride possesses a very high hardness and is used in machining applications as a PcBN material, where it is incorporated in a ceramic and/or metallic matrix. Binders such as Al, Co, Ni, W, elements from groups 4, 5, or 6 of the Periodic Table, or even their ceramic compounds (as carbides, nitrides, borides, silicides) can be used, which results in several different types of microstructures [1,2]. To help dealing with such variety of different systems, thermodynamic tools can be used to allow the prediction of reactions and formation of new phases in each specific system. Throw the manipulation of the initial composition, it is possible to engineer the presence and/or formation of new phases in the system that have more favorable properties. In this work, we used the Thermo-Calc software to calculate the phase diagram of cBN compositions with TiCN and Ni. The predicted reactions and formed phases were experimentally validated for a 50% cBN + 45% TiCN + 5% Ni (vol.) composition, sintered by Spark Plasma Sintering and characterized in terms of its microstructure, chemical composition and physical properties.

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Obtaining of the rod-shaped $BaTiO_3$ nanoparticle and its properties

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Recently, special attention has been paid to the anisotropic, in particular, one-dimensional perovskite-based nanostructure, due to its attractive and unique electrical properties. Understanding the mechanism of formation of such nanoparticles and the influence of its structural and morphological characteristics on the properties are crucial for creating an advanced material with the desired properties. Therefore, this report will consider the features of obtaining a rod-shaped $BaTiO_3$ nanoparticle, its physical and chemical characteristics and electrical properties.

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Transparent Eu:$La_2Zr_2O_7$ ceramics – lithium fluoride strikes again

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Lanthanum zirconate ($La_2Zr_2O_7$) is a member of pyrochlore family and potential candidate for luminescence-related applications upon suitable doping (for example Eu as in the present study). Unfortunately, preparation of transparent $La_2Zr_2O_7$ is difficult due to its reluctance to complete densification. Usually, high temperatures ~1800 °C and dwell times ~6 h in vacuum or somewhat lower temperatures ~1700 °C in oxygen atmosphere but for longer time >20 h are necessary for preparation of transparent material. We present a method for complete densification of Eu:$La_2Zr_2O_7$ (3 at.% of Eu) from lab-synthetized powder up to transparency at considerably lower temperature 1500 °C with total processing time of 5 h (heating and cooling included) under pressure of 80 MPa by spark plasma sintering (SPS) with the help of LiF as sintering additive. The addition of LiF reduced the on-set temperature of densification by ~300 °C and enhanced densification kinetics, however, promoted grain growth as well. Following the same routine, sample without LiF addition was not transparent (despite being completely dense). Post-annealing of samples was necessary to oxidize Eu^{2+} to Eu^{3+} which resulted in color change from dark red to colorless.

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Direct Laser writing based surface texturing for enhanced adhesion between zirconia (3Y-TZP) and resin-matrix cement

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Objective: To evaluate the influence of short-pulse laser (SPL) surface texturing of 3 mol % Yettria-tetragonal zirconia polycrystalline (3Y-TZP) on shear bond strength with resin-matrix cements.

Methods: Zirconia (3Y-TZP) green compacts received two SPL patterns: LD35 - Squared pattern lines 35 µm spacing (0.6 W,128 mm/s); LD10 - Squared pattern lines 10 µm spacing (0.06 W,256 mm/s) and sintered zirconia samples received two treatments: SB - controlled blasting of alumina particles (Al_2O_3); SC - controlled blasting of silica (SiO_2) coated alumina particles. Surface energy, surface roughness, wettability (contact angle: water (H_2O); Diiodomethane (CH_2I_2)) is evaluated for each group (n = 12/group). Treated samples from all groups were ultrasonically cleaned and cemented to resin-matrix cement using primers. The cylindrical shape cements bonded at the center of 3Y-TZP samples were either stored for 24 h at 37º C or thermocycled (5-55 º C, 6000 cycles) and shear bond strength (SBS) was performed subsequently. *Results:* The surface roughness was highest in DL35 (2.1 ± 0.3)……. .The lowest and highest contact angle (water) was produced by SC (27.30 ± 4.66) and SB (61.44 ± 2.97) respectively. Similarly the lowest and highest contact angle (Diiodomethane) was produced by LD10 (26.93 ± 2.85) and SC (36.76 ± 0.27) respectively. Surface free energy was highest for SC (65.30 ± 2.06) and lowest for SB (56.15 ± 1.19). The two lasers groups: LD10 (9.16 ± 1.55) and LD35 (8.89 ± 2.18) produced higher SBS than SC (6.46 ± 1.84) group. The highest SBS was produced by SB (9.69 ± 3.93) however the highest error was also incurred in this group. Thermocycling (TC) reduced the SBS of all groups. The highest SBS after TC was shown by LD35 (8.49 ± 2.02) and the lowest by SB (5.97 ± 2.43). The reduction in SBS was significant in SB and both laser groups retained higher SBS than SB and SC, there was an increase of SBS in SC group after TC.

Influence of liquid nature on the densification of Hydroxyapatite powders through Cold Sintering Process

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Abstract

Cold sintering process (CSP) is an innovative low-energy sintering technique for densifying the ceramics/composites at very low temperatures <300°C using pressure (<500MPa) and transient liquid. Densifying the nano-hydroxyapatite in their original form for bone regeneration applications is viable in CSP and the densification relies upon the factors like powder source, processing parameters (temperature, pressure and hold time) and transient liquid (aqueous, acid and base). Our present investigation is about the densification of commercial nano Hydroxyapatite powders (HA) processed via wet chemical precipitation and spray drying. The spherical morphology, phase purity and particle size are confirmed by scanning electron microscopy, X-ray diffraction and particle size measurement techniques. The cold sintering of HA was carried out using water, acetic acid, and phosphoric acid (10 wt. %) under a moderate pressure of 360MPa at 200°C. Preliminary investigation on the relative density indicated the densification varies with nature of liquids. With respect to the acid concentrations (0.5M, 1.0M, & 2.0M) under the same operating pressure and temperature, the relative densities are varied linearly and a maximum of ~90% densification has been achieved for the experiment carried out with 2.0M phosphoric acid. Overall, the results demonstrate the importance of the nature of transient liquid, and concentration of acids in the cold sintering of HA powders for biomedical applications.

Keywords: Cold sintering, nano Hydroxyapatite, densification, liquid nature, acid concentration, biomaterial

Mechanical performance of silicon nitride parts made by additive manufacturing

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Ceramics based on silicon nitride (Si3N4) are well-recognized materials for numerous structural applications due to their combination of properties, such as mechanical strength, thermal shock resistance, high hardness, and good wear resistance [1]. Although additive manufacturing (AM) appeared as a competitive way to attain complex geometries at lower costs, most of AM technologies still present limitations in producing fully dense ceramics. This is frequently related to the large amounts of organic binders needed to the feedstock formulation [2]. The burn out of the organic components is a critical step, inducing the appearance of several defects in the final ceramic part, consequently reducing the mechanical performance. Robocasting or direct ink writing is one AM technique with high potential for ceramic-based materials, namely Si3N4, since it uses lower amounts of additives in the feedstock when compared to other suspension-based technologies, as for example vat photopolymerization or fused filament fabrication.
The purpose of this work is to produce high dense Si3N4 parts by robocasting, with suitable dimensional resolution, and that present mechanical performance comparable with similar parts obtained by conventional powder processing techniques. The aim of this work is to produce fully dense parts with internal cooling channels suitable for machining applications by pressureless sintering, using Al2O3 and Y2O3 as sintering additives. The final parts were evaluated in terms of mechanical performance, namely flexural strength and hardness.

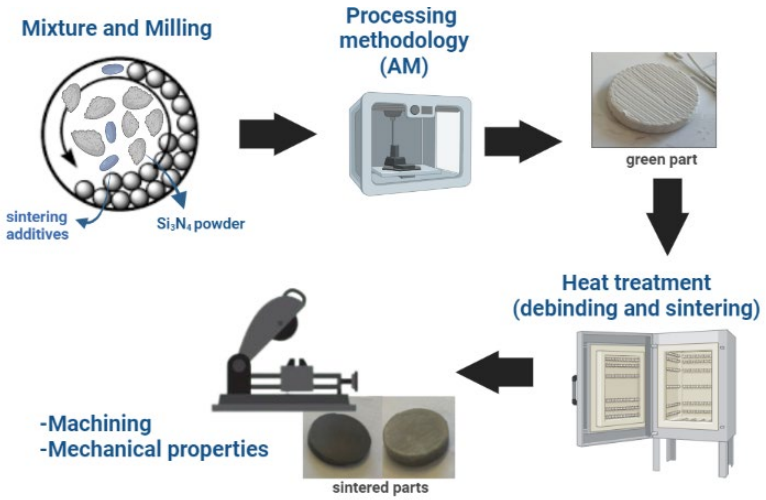

Figure 1. Schematic procedure of silicon nitride parts developed by robocasting

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Additive Manufacturing of Ceramic Supports for Photocatalytic Degradation of Hydroxychloroquine

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Pollution of the aquatic ecosystems from medicines and pharmaceutical products poses a threat to environmental and global health. Consequently, it is necessary urgently to remove them from the environment. The aim of advanced oxidative processes (AOPs) is to produce highly reactive radicals such as hydroxyl radicals ($E°$=2.72 V) that can break down the structure of pollutant molecules to reduce organic contaminants in water. Heterogeneous photocatalysis, which involves light activation of semiconductor materials, stands out among these processes. Due to its great chemical stability, nontoxic, low cost, and strong photocatalytic activity, titanium dioxide (TiO_2) has the potential for use as an efficient photocatalyst for the removal of a wider number of pollutants present in the environment.

Catalysis processes are increasingly being influenced by additive manufacturing. Their connection is based on the need to intensify catalytic processes to make them more efficient and sustainable. Additive manufacturing can satisfy such a need, generating materials with an advanced design, easy production, and great adaptation, in addition to their high catalytic functionality. The immobilization of photocatalysts is considered as an advantage in wastewater treatment due to the durability and stability of immobilized photocatalytic materials compared to suspension form.

In this work, the TiO_2 was immobilized on the surface of the 3D-printed ceramic supports produced by the fuse filament fabrication technique. The post-processing technique of TiO_2 immobilization on the 3D printed supports using solvothermal synthesis in situ growth method was applied. The photocatalytic activity of immobilized TiO_2 was evaluated by the degradation of hydroxychloroquine from an aqueous solution under UV-Vis illumination. A comparison was made between impregnated 3D printed ceramic support and the powdered TiO_2. The prepared photocatalyst was characterized using X-ray diffractometry (XRD), scanning electron microscope (SEM), and energy dispersive spectroscopy (EDS). The obtained results of performed photocatalytic tests show the efficient degradation rate of hydroxychloroquine using the heterogeneous photocatalytic process.

Synthesis of $AB_2S_4:Eu^{2+}$ scintillator materials by mechanosynthesis and hydrothermal routes for ion detection diagnosis in nuclear fusion reactors

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Luminescence scintillation materials are used in nuclear fusion devices as active components for ion detection, for example, in the Fast Ion Loss Detector (FILD) [1].
The most extended scintillator material is the europium doped strontium thiogallate ($SrGa_2S_4$:Eu2+), which shows at the moment the best detection efficiency (emitted photons) when irradiated with light ions (deuterium, protons and alpha particles) in an energy range of 1 to 3 MeV [2].

However, its availability in the market is very scarce, cost is excessively high, and the purity is always compromised by the presence of different subproducts. In addition, its synthesis usually involves the use of hydrogen sulphide (H_2S) toxic gas and /or complex reactions [3].

In this work, we developed two simple, reproducible, and economic routes, which consist on solid-state mechanosynthesis and a single-step hydrothermal route to synthesize different europium doped materials with the strontium thiogallate structure and ($AB_2S_4:Eu^{2+}$ A=Sr, Cd and Cu, B= Bi, Ga and In) with high purity. A complete chemical, physical and microstructural characterization demonstrate the formation of the orthorhombic structure expected, judged mainly by DRX and TEM analysis. Furthermore, ionoluminescent measurements are made in a 3 MV tandem accelerator to characterize the luminescent efficiency of the materials. All synthesized materials showed luminescence in the visible range and with enough intensity to be used as scintillator materials.

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Optimisation of mechanical properties of 3d printed alumina : linking rheology and printing parameters.

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Additive manufacturing of ceramic materials has been rapidly expanding for several years. Robocasting (sometimes referred to as Direct Ink Writing) is one of the few additive manufacturing techniques that allows processing multiple materials simultaneously as multiple cartridges can be loaded in the machine and used to print a single part. Thanks to that, we are able to create dense or porous materials with complex geometries and gradients of composition.

Robocasting uses mechanical forces to extrude ceramic pastes (called « inks ») from one or several syringes through thin nozzles, following a computer-aided design model to form green 3D structures. To allow extrusion through fine nozzles, homogenous ceramic paste must be obtained. The rheological properties are also considered to be one of the most important factors for a successful printing. That's why, we have to work with shear-thinning materials. Shear-thinning behavior is not the only important parameter. Indeed, we have to obtain pastes with a sufficient yield stress and an adequate thixotropic behavior. Both homogeneity and rheology were thus optimized by the formulation of ceramics paste.

In addition, the influence of printing parameters was also studied: path of the nozzle to make the design, use of two syringes to create either support structures or bi-materials architectures, etc. Printed parts were characterized by microscopies assay. Moreover, some mechanical studies will be characterized using diametric compression tests. These studies will allow to better understand links between printing parameters, rheological properties, and mechanical properties in order to define printability for dense parts [1].

This work is a part of the CERAPID ANR project, supported by the French « Agence Nationale de la Recherche » under grant number ANR-17-CE08-0021, and that brings together SIMAP (Grenoble), LGF (St Etienne), CRISMAT (Caen) and MATEIS (Lyon) laboratories [2].

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Can ceramic products use eggshell waste as secondary raw material?

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Interest in waste valorization is increasing due to environmental and economic benefits. In 2018, approximately 8.5 million tons of eggshell waste were generated worldwide and, usually, landfilling is the only option. Consequently, development of added-value applications for eggshell waste is of utmost importance. The present work aims to study, at a pre-industrial scale, the production of ceramic wall tiles with bio-$CaCO_3$ from eggshell waste. Firstly, the eggshell residue was pre-treated in a new and innovative industrial prototype that separates the membrane from the shell through a simple and low-cost process. Then, atomized powders were produced substituting the limestone with different levels (0, 50, 75 and 100 wt.%.) of the eggshell residue. Pre-industrial ceramic tiles (10 x 10 cm^2) were produced for each formulation and tested. The ceramic wall tiles were glazed with opaque and white glazes and fired at an industrial furnace and their properties were evaluated. Through specimens' characterization, no significant differences were observed, and all values are within industrial limits. Therefore, this work proved that, for ceramic wall tiles, the limestone can be totally substituted by bio calcium carbonate from eggshell waste. The developed products will contribute to reducing the amount of waste deposited in landfills and the consumption of virgin raw materials.

Development of geopolymeric structures by additive manufacturing to remove pollutants from wastewater

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Geopolymers are a class of materials synthesized by mixing an aluminosilicate source with an alkaline solution, obtained at room temperature. The use of these alkali-activated materials for heavy metal's adsorption from highly polluted wastewater is promising due to their excellent ability to retain and immobilize contaminants within the structure. Adsorbent materials can be produced through Additive Manufacturing (AM), a disruptive process to produce three-dimensional objects. This innovative technology allows very precise control of the macroscopic porosity and free design of structures, fast production and limited material waste.

The purpose of this work was the development of geopolymeric structures incorporating high amounts of waste (red mud (RM) extracted from alumina production and fly ash (FA) from biomass combustion) prepared through AM, to remove heavy metals from polluted wastewater. It was intended to develop materials with a high adsorption capacity for heavy metals, such as arsenic (As), copper (Cu), cobalt (Co), iron (Fe), zinc (Zn) and cadmium (Cd).

For the incorporation of FA in the mixtures (10, 20, 30 and 40 wt.% of FA), the amount of RM (40 wt.%) was kept constant and the amount of the virgin raw material metakaolin was reduced (from 60 to 20 wt.%). Four different compositions were tested to optimize the FA amount (10 to 40 wt.%), to obtain both high compressive strength and heavy metal adsorption capacity. Very high compressive strength was obtained for the 3D-printed structures, achieving more than 26 MPa. These highly resistant structures also present high adsorption capacity for heavy metals (e.g. removals of 25 % of Zn, 40 % of Cu and 60 % Cd).

This sustainable strategy is in line with the United Nation's sustainable development goals and to move from a linear to a circular economy, reducing the consumption of virgin raw materials while contributing to reducing water pollution.

Acknowledgements

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Defect-Free Hybrid Additive Manufacturing of Advanced Ceramics

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Hybrid manufacturing combines two or more distinct manufacturing processes, leveraging the advantages of each process whilst mitigating their limitations. We use this approach to enable the production of functional parts from advanced ceramic materials. Combining material extrusion with subtractive processes allows for the geometric complexity of freeform fabrication with the surface finish and precision characteristic of conventional manufacturing. However, the extrusion process is susceptible to defects that are hard to predict and often detrimental to the mechanical properties of a part. We propose to address this through automated in-situ monitoring, in line with a general shift towards data-driven manufacturing coined 'Industry 4.0'. Mounting an optical camera and laser on the machine, we capture images and scans of each layer in a part. By comparing this data to the digital model, we aim to evaluate the geometrical accuracy of the part. Defects can be identified, located and measured through image processing. Using closed-loop control, and the flexibility of our hybrid platform, this information can be passed back into the control software, and material can be deposited or removed to correct defects, theoretically enabling defect-free manufacturing. This work demonstrates the reworking of defective layers during hybrid manufacturing, proving the viability of this technology for the creation of defect-free advanced ceramic components for high performance applications.

Content – Maximum 300 words

Analytical modeling of the effective thermal conductivity of alumina-based ceramic matrix composites with interfacial thermal resistance

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The effective thermal conductivity is a key property in many applications of ceramics, ranging from thermally conducting electronic substrates to insulating thermal barriers. In two-phase materials such as ceramic matrix composites it can be modified by tailoring the composition and microstructure. In matrix-inclusion microstructures, for example, the volume fraction of second-phase inclusions has a major influence on the effective thermal conductivity. In general, however, also the shape (e.g., the aspect ratio) and the curvature of the interfaces can play a significant role. Moreover, due to the interfacial thermal resistance also the size of the inclusions can become important. In this contribution we investigate the effect of the interfacial thermal resistance on the effective thermal conductivity of alumina-based ceramic matrix composites. In particular, considering composites of alumina ceramics (thermal conductivity 33 W/mK) with zirconia (3 W/mK) and diamond (1500 W/mK) as examples, and assuming reasonable values of interfacial thermal resistance (of order 10^{-7} m^2K/W) as well as spherical inclusion shape, two types of effective thermal conductivity predictions are compared: the classical Hasselman-Johnson model and a new model that combines Nan's interface correction with the classical Bruggeman-Landauer self-consistent model. It is shown that according to both models the interfacial thermal resistance can reduce the effective thermal conductivity considerably, in extreme cases – below a critical size of inclusions – to such a degree that even for high-conductivity inclusions the effective thermal conductivity can decrease below that of the low-conductivity matrix (so-called "shielding effect"). Last but not least, the porosity dependence of the effective thermal conductivity is taken into account and compared with the inclusion size dependence on the basis of an "equivalent porosity" concept that has been previously introduced for single-phase polycrystalline materials.

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Beyond the Average: The Criticality of Spatial and Temporal Fluctuations in Oxide Glasses

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Atomic structure dictates the performance of all materials systems; characteristic of disordered materials is the significance of spatial and temporal fluctuations on composition-structure-property-performance relationships. Glass has a disordered atomic arrangement, which induces localized distributions in physical properties that are conventionally defined by average values. Quantifying these statistical distributions (including variances, fluctuations, heterogeneities) is necessary to describe the complexity of glass-forming systems. Only recently have rigorous theories been developed to predict heterogeneities to manipulate and optimize glass properties. This oral presentation provides a critical analysis of experimental, computational, and theoretical studies to demonstrate the impact of statistical fluctuations on physical properties (e.g., thermodynamic, kinetic, mechanical, optical) and processes (e.g., relaxation, crystallization, phase separation) in oxide glasses. Further development of rigorous investigations of fluctuations are necessary for the future of the glass field, as these studies contribute to improved fundamental understanding of the chemistry and physics governing glass-forming systems and optimized structure-property-performance relationships for next-generation technological applications of glass (including damage-resistant electronic displays, safer pharmaceutical vials to store and transport vaccines, and lower-attenuation fiber optics). We invite the audience to join us in exploring what can be discovered in glass-forming systems by going beyond the average.

Bioceramic bone cements modified with bioglass

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Recently, there has been a significant increase in the interest of use of biomaterials containing bioglasses in medical applications. However, their impact on the properties of bioceramic bone cements has not been well investigated yet. Our studies focus on the influence of the addition of a special kind of bioglass to chemically bonded materials on the basis of α tricalcium phosphate (α- TCP). A novel gold-doped bioglass from the P_2O_5 – CaO – $Ca(OH)_2$ – KF – TiO_2 system was added to the calcium phosphate matrix in the amount of 10wt% or 20wt.%. The introduction of gold is supposed to have a contrasting effect in magnetic resonance imaging (MRI) in the final biomaterial. Selected physiochemical properties such as: time of setting, compressive strength, phase composition (XRD), and *in vitro* bioactivity have been determined. The addition of bioglass to biocement decreased its compressive strength from 6,5 MPa to 2,2 MPa. The bioactivity studies were performed by 28 days incubation of materials in Simulated Body Fluid (SBF). Bioglass as well as biocements surfaces were observed using a SEM microscope. The Kokubo procedure has been shown to be inappropriate for evaluating the bioactive potential of obtained biomaterials. Further *in vitro* and *in vivo* studies are necessary to determine the biological properties of bioceramic bone cements.

*Keywords: bio*ceramics, bioglass, bone cements, doped bioglass

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HOW NOVEL BIOMATERIALS BASED ON BIOACTIVE GLASS AND β-TRICALCIUM PHOSPHATE CAN BE EVALUATED UNDER PHYSIOLOGICALLY RELEVANT CONDITIONS?

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Comprehensive preclinical studies are essential for the development of novel biomaterials that can be used in biomedical applications. However, traditional methods used for the evaluation of biomaterials have certain limitations. *In vitro* testing in cell monolayers is fast and easily accessible, but the 2D environment can affect cell metabolism and morphology, leading to unreliable results. On the other hand, *in vivo* animal studies are complex, time-consuming, expensive, and raise ethical concerns.
Biomimetic bioreactors, primarily developed for tissue engineering to provide a physiologically relevant, strictly controlled environment that mimics the conditions in specific tissues or organs, could be indispensable tools in physiologically relevant biomaterial characterization and step between *in vitro* and *in vivo* studies. They offer the majority or all the necessary biochemical (e.g. pH, nutrients, gases, growth factors) and biophysical signals (e.g., shear stress, hydrostatic pressure, mechanical strains) highly relevant for biomaterial assessment and prediction of material behavior after implantation.
Our group has developed two types of potential biomaterials aimed for bone and osteochondral tissue engineering based on bioactive glass (BAG), β-tricalcium phosphate (β-TCP), and different natural polymers (gellan gum and alginate). Scaffolds' integrity and mechanical properties were monitored continuously under the physiological level of mechanical compression using a dynamic compression bioreactor coupled with medium perfusion during 14 days. Formation of hydroxyapatite (HAp) within the scaffolds was investigated in a perfusion bioreactor, in the presence of simulated body fluid (SBF) during 14 and 28 days for scaffolds based on BAG and β-TCP, respectively. SEM, EDS, and XRD results have shown a significant increase in the formation of HAp under bioreactor conditions compared to static control conditions. Beyond that, formed HAp crystals were more uniformly distributed throughout scaffolds and presented more cauliflower-like morphology. The obtained results demonstrated the utilization potential of biomimetic bioreactors in physiologically relevant biomaterial characterization.

Electrophoretically deposited iron doped manganese- copper spinel coating for prevention of chromium poisoning in Solid Oxide Fuel Cells

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In intermediate temperature Solid Oxide Fuel Cell (SOFC) stacks, Cr_2O_3- forming ferritic stainless steel is considered as the most suitable material for interconnects due to their thermal expansion coefficient match with other components, high electrical conductivity, and low cost. Moreover, Cr_2O_3 also has significantly higher electrical conductivity compared to other oxides e.g. Al_2O_3 which are form on the alumina forming alloys. However, Cr-poisoning caused by Cr_2O_3 may degrade the cathode of SOFC. Protective coatings are used to prevent this negative phenomenon. The current state of the art protective coating material is the $(Mn,Co)_3O_4$ spinel (MCO), which shows high electronic conductivity and low Cr diffusion. The main issue with the MCO is the cobalt, which is regarded as a carcinogenic element and is also quite expensive due to use in Li-ion batteries.

One of the environmentally benign candidates for the protective layer is $(Mn,Cu)_3O_4$ spinel (MCuO) which can substitute the harmful MCO. It is reported that Mn-Cu oxide spinels have higher electrical conductivity at working temperatures (600- 900°C) than MCO and can also prevent Cr-poisoning. The issue with using MCuO is its structural instability at high temperatures: limited single-phase region in the phase diagram. It can result in precipitation of CuO and change of the physicochemical properties. We have decided to study partially substituted MCuO. We have partially substituted Mn with Fe, which results in structure stabilization and has a compatible TEC and possibly retain low Cr evaporation.

We will present results concerning materials synthesis, electrophoretic deposition on Crofer 22 APU steel coupons, subsequent oxidation exposures, and electrical resistivity tests. Extensive post-mortem characterization of alloys by SEM/TEM microscopy will be also presented.

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Production of TiO_2 ceramic supports via FFF

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Additive Manufacturing (AM) of ceramics has experienced an exponential growth over the last years, mainly due to the advantages related to the production of non-conventional geometries. Among AM technologies, Filament Fused Fabrication (FFF) is one of the most widespread techniques and an easy way to produce freeform objects with reasonable resolution (layer height ≤ 100µm) at low cost.

To date, FFF technology has been limited to the use of polymeric and polymer-based materials for filament fabrication. There is some previous research to produce filaments with high inorganic loading (≥ 50 v%) that could be used in modified printheads [1], and recently some other groups have been explored novel routes [2] [3]. On the other hand, we have developed an alternative filament formulation that allows reaching ceramic contents up to 75 wt%, while the printability characteristics of the filament is maintained using a wide variety of ceramics. [4] [5].

In this work, TiO_2-based filaments (60-75 wt%) have been developed and optimised to print and produce 100% ceramic monoliths with controlled porosity via the addition of carbon microspheres as pore former agent. The pseudoplastic behaviour of the filament reveals an elasticity increase with the ceramic loading. After thermal treatment for debinding and sintering at 1200°C, the 3D printed objects exhibit a low contraction and high quality, with no defects or cracks in the structure. Additionally, sintered specimens have been fabricated including pore formers in the formulation to produce monoliths with controlled porosity from 70% to full density. XRD performed in the 3D printed monoliths after sintering reveals phase stability, with retention of the initial rutile structure (i.e., tetragonal symmetry) without significant changes beyond the expected increase in crystallinity.

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Optimization of Full Ceramic LFP/C cathodes for FFF

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Nowadays, rechargeable lithium batteries (LiBs) are the most widely used energy storage devices due to their high capacity and long-term cycle life, which find application in portable electronics, power tools and electric vehicles. However, LiBs must deal with several challenges yet, as the ever-increasing demand of higher energy density, extended life cycle and safety issues.

Additionally, great effort focuses on maximising the energy density in the final devices via manufacturing routes. Although conventional processes provide adequate performances, they use up to 50% vol of non-electrochemically active materials, which has a negative impact. Additive manufacturing (AM) techniques may play a crucial role to improve the final energy devices performance as they offer total design freedom during fabrication.

Alternative architectures have been successfully explored via direct ink witing (DIW) and material jetting (MJT), showing reversible capacities close to theoretical. However, handling of inks cannot be considered as simple and fully straightforward because ceramic materials tend to agglomerate. Conversely, fused filament fabrication (FFF) employs thermoplastic-based filaments that do no demand special preservation requirements. This combined with the ease of use and low cost, justify the on-going research efforts. To date LiBs applications via FFF are rather limited, showing modest performances mostly due to the large amount of inactive material in the filaments.

We have recently produced full ceramic $LiFePO_4$/C-based electrodes using a novel process via FFF and further sintering[1]. Filaments have been fabricated by optimising the LFP/C ratio and maximising the ceramic content in the formulation, reaching up to 70% wt. The conductivity of the 3D-printed electrodes was the same than in the case of LFP electrodes prepared via conventional processing materials (2.06E-4 S/cm at 50 ºC), while the electrochemical performance yields up to 152 mAh/g and 97% for reversible capacity and coulombic efficiency at C/2, respectively, without significant performance losses after 100 cycles.

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Thermodynamic Simulation and Design of Microstructure in Low cBN Composites Produced by Spark Plasma Sintering

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Abstract

Polycrystalline cubic boron nitride (PcBN) materials with a ceramic and/or metallic matrix are widely used as tips, full tops or solid cBN inserts in machining of iron-based alloys due to their exceptional hardness, together with thermal and chemical stability. Achieving high hardness (>20 GPa) in the cubic BN phase requires a carefully designed microstructure of the matrix, which can be attained by selecting an appropriate composition and processing conditions. Thermodynamic software tools have emerged as an important tool for designing new materials compositions by predicting and designing phase diagrams, as well as calculating thermodynamic properties to facilitate process development. In this study, the matrix microstructures in the cBN-TiCN-Ni system were designed and predicted using the Thermo-calc software, which will be validated experimentally. Compositions of cBN-TiCN (50:50 vol %) with the addition of Ni in different volume percentages will be consolidated by Spark Plasma Sintering (SPS), and their structures and microstructures analyzed using XRD and SEM/EDS to validate the thermodynamic simulations.

Alternative sintering of $K_{1-x}Na_xNbO_3$ (KNN) ceramics: preliminary data on the application of the ultrafast high-temperature method

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The sintering of ceramics is a high-energy-consuming step in the fabrication process. Ultrafast high-temperature sintering (UHS) is a newly developed technique that allows for ceramic sintering in less than one minute. This is achieved by placing a green ceramic between two strips of carbon felt heaters, which generate heat through the Joule effect and rapidly dissipate it throughout the ceramic material.
The extremely high heating rates of this technology facilitate fine microstructure development and high retention of volatile elements. As a result, it is a useful technique for ceramics with low sinterability, such as potassium-sodium niobate ($K_{1-x}Na_xNbO_3$ or KNN).
KNN is a lead-free ferroelectric ceramic that has high potential to replace the current lead-based piezoelectric materials in the market. However, KNN faces several challenges during the sintering stage, such as the rapid volatilization of the alkali elements, the formation of second phases, and the need for precise microstructural control. In this study, KNN was chosen as a model system to evaluate the potential of UHS due to the alignment between the weaknesses of the material processing and the strengths of this sintering technique. A custom UHS apparatus was constructed, tested, and calibrated to perform these experiments. Preliminary tests were conducted with KNN to identify the optimal parameters for achieving high-quality KNN production using UHS.

Real wastewater treatment by means of red mud based porous alkali activated materials

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Red mud (RM) is a highly alkaline and hazardous waste generated in the Bayer with a production of about 150 million tonnes per year [1]. Over the years, around 4 billion tonnes of RM have been stored in dams/lagoons due to lack of appropriate recycling strategies [2, 3]. Several studies have been carried out recently to develop a more sustainable management strategies, yet industrial reuse of these wastes is barely achieved accounting for only 3% of their annual production [1].

Alkali-activated materials (AAMs) can be an alternative to RM reuse, as these new binders can be produced with precursors that have a similar composition to RM [4]. However, RM is known to have low reactivity in an alkaline environment, which hinders its use in structural applications. However, AAMs can be used in other added value applications [5]. One very interesting application could be the use of AAMs as heavy metal adsorbents or pH regulators [4, 6].

In this work several porous AAM's were produced using RM as the main precursor and used for wastewater treatment. The samples were tested to assess their benignity and selected in accordance. Removal efficiency of several heavy metals present was evaluated reaching a removal efficiency as high as 98.4 % depending on the heavy metal and contact time.

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Cycling demonstration of Sequential Deposition Synthesized lithium garnet films in full batteries

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Sequential deposition synthesis (SDS) is a recently-discovered technique that allows a solid-state electrolyte layer to be fabricated directly from a liquid precursor which is then atomized onto a heated substrate where the solvent evaporates and the precursor salts decompose to form a conformal layer. $Li_7La_3Zr_2O_{12}$ (LLZO) is a promising material that has seen much interest in the battery field but has challenges to synthesize this layer between 5-15 µm thick which SDS is capable of fabricating. LLZO films were fabricated directly onto a porous substrate that was infiltrated with a polymer gel electrolyte and the cycling performance was demonstrated in a full cell using Li metal anode and lithium cobalt oxide (LCO) cathode. The cycling performance is discussed in reference to state-of-the-art performance in the battery field and post-mortem analysis on the cells is performed to elucidate failure mechanisms.

Thin films of transparent oxides with multifunctionality, suitable for both photocatalysis and antimicrobial uses

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In recent years, there has been a significant increase in the interest surrounding thin film technology due to its broad range of applications, such as in photovoltaic solar cells [1], energy storage [2], heterogeneous photocatalysis, and antimicrobial action [3]. Additionally, the COVID-19 pandemic has made the development of antivirus and antimicrobial materials a critical priority worldwide. Consequently, the ability to functionalize various surfaces without altering their optical properties has become a primary concern.

To address this need, transparent TiO_2 thin films coated with Ag NPs were synthesized using two industrially applicable techniques, pulsed laser ablation (PLAL) and spray pyrolysis, without the use of high vacuum. These thin films were deposited on glass to create materials with photocatalytic and antimicrobial properties while maintaining high transparency. To analyze the structural, morphological, and optical properties of the thin films, Grazing incidence X-ray diffraction (GIXRD), Raman spectroscopy, Scanning electron microscopy (SEM), and ultraviolet-visible spectroscopy were used. The presence of NPs on the TiO_2 surface was identified using Transmission electron microscopy (TEM). The thin films exhibited a transmittance value of over 80%.

Next, the photocatalytic capacity of the synthesized thin films was evaluated by measuring the degradation of Rhodamine B (RhB) under UV light irradiation. The presence of Ag NPs on the TiO_2 surface resulted in an improvement in photocatalytic properties, with a 99% degradation of RhB achieved in just 210 minutes under UV light. Furthermore, these transparent thin films demonstrated high antimicrobial activity on Gram-negative bacteria when irradiated with UV light for 4 hours, effectively killing 93% of these bacteria.

Keywords: Transparent thin films; Spray pyrolysis; Laser ablation; Antimicrobial activity; Photocatalysis.

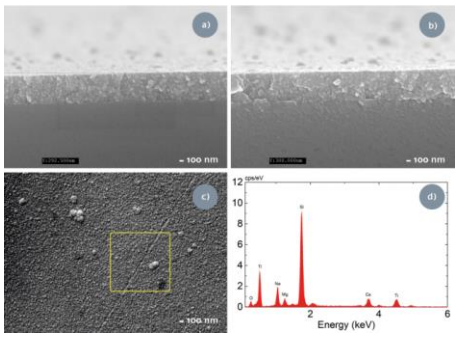

Figure 1: SEM images for the cross section of(a) TiO_2 transparent thin films, (b) TiO_2-Ag NPs, (c) top surface of TiO_2, and (d) EDS spectra for the surface of TiO_2.

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Manufacture of YSZ-based electrolytes for SOFC by 3D printing

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Solid Oxide Fuel Cells (SOFCs) are considered a good alternative to overcome some of the problems related to climate change by reducing the greenhouse gases emissions when using hydrogen as fuel instead of fossil fuels. Furthermore, the high operating temperatures of SOFCs enable the possibility to feed them with alternative fuels, which would facilitate the transition towards a more environmentally friendly energy model [1].
Currently, as alternatives to conventional manufacturing processes for SOFCs electrodes and electrolytes, 3D printing stands out for the possibility to produce, for instance, alternative shapes and geometries that are cannot be obtained with traditional manufacturing technologies. Therefore, this may lead to an enhancement of the volumetric and gravimetric energy density, the extension of the Triple Phase Boundary region, tailoring of the electrodes microstructure, the optimization of heat dissipation, … [2].
Among the electrolyte materials, $(ZrO_2)_{1-x}(Y_2O_3)_x$ (YSZ) is considered the state of the art [1]. In this context, this work shows our recent progress in the characterisation of 3- and 8YSZ electrolytes produced by Fused Filament Fabrication (FFF) and compares the results with those obtained for electrolytes produced via conventional fabrication techniques. Ceramic filaments are obtained by mixture of different organic additives with ceramic powders (65-75%w) to produce a green body that can be extruded [3]. After 3D printing, the organics are removed by a debinding thermal treatment specifically designed to avoid collapse of the manufactured geometries and to promote a high degree of compactness of the YSZ electrolyte after sintering. The ionic conductivity of the 3D-printed electrolytes of 3 and 8YSZ are 0,027 S/cm and 0.064 S/cm, respectively, at 900 °C, which is fairly similar to the values obtained by conventional press and sintering (i.e. 0,031 S/cm and 0,07 S/cm for 3YSZ 8YSZ at 900°C, respectively). From the rheological characterisation, all filament compositions exhibit a solid-like behaviour, with elasticity increasing with the ceramic content in the filaments.

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Multi-phase (Zr,Ti,Cr)B_2 solid solutions: preparation, microstructure and local properties

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Hypersonic systems require materials able to survive important heat fluxes as well as aerodynamic and mechanical loads. Current available materials suffer of a series of technological limitations, like not favorable performance and production costs, to weight ratios or inability to perform under hot ablative fluxes. Ultra-high temperature ceramics (UHTCs) are possible candidates that, upon suitable composition and microstructure tailoring, can be a suitable solution. ZrB_2 is widely recognized as the most prominent ultra-high temperature ceramic for aerospace applications, in view of its melting point above 3000°C, and despite it exhibits lower oxidation and ablation resistance as compared to HfB2, it has a much lower density. The addition of TiB_2 further lowers the overall weight, which is a relevant factor for materials intended to flight, but it also worsen the oxidation resistance. In this work Cr is added to the ZrB_2-TiB_2 system to study its effect on the densification, microstructure and thermo-mechanical properties. By adjusting the processing and sintering cycles, fully dense multi-phase ceramics with density in the 5.3-5.7 g/cm3 range and hardness close to 24 GPa have been obtained. A common feature to all materials, is the formation of solid solutions and microstructural details obtained by x-ray diffraction, scanning and transmission electron microscopy are highlighted. Particularly, we explored the nanotexturing of the shell within micron-sized boride grains of the matrix, which resulted from the preferential precipitation of Me-compounds with poor solubility within ZrB_2 or TiB_2 lattice. Preliminary bending strength and oxidation behavior of these intricate bulk multiphase ceramics are also provided.

Assessment of optimum ratio of Metal Organic Framework and activated carbon in composites for photocatalytic removal of Congo Red dye

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Rapid growth of human population has led to increased water demand by various industries and production of large amount of wastewater. Textile, leather, paper, and plastics industries wastewater, which contains organic pollutants such as dyes, needs a proper treatment in order to be safely discharged into the environment, therefore, new methods are being developed. This research is based on investigation of photocatalytic degradation of an azo-type dye Congo Red (CR) by using powdered UiO-66 type MOF (Metal Organic Framework), activated carbon (AC) and their composite powder mixtures made with different MOF/AC ratios. Additionally, this research includes investigation of renewability of these materials in several experimental cycles. Experiments were performed in batch conditions, samples were exposed to solar light irradiation, then centrifuged to remove solid particles and in order to determine the concentration of the remaining dye, solutions were analyzed by UV-ViS spectrophotometer. Renewability of the photocatalysts used in experiments was investigated by repeating this process three times, with catalysts dried at 50 °C between cycles. Results have shown that pure MOF has excellent and almost identical photocatalytical efficiency in every cycle, compared to all MOF/AC composites and pure AC, probably due to the saturation of available adsorption sites in AC. CR removal efficiency and renewability of various composites depends on MOF/AC ratio, therefore, the most efficient composites are the ones where MOF content varies from 25 wt. % up to 100 wt. %. Samples with MOF content 50 - 100 wt. % have shown excellent renewability, while composites where AC is more dominant (95 - 100 wt. %) have shown almost none. Among the composites examined in this paper, the one with MOF/AC ratio of 50/50 has shown the best cost/performance ratio in removal of Congo red dye from aqueous solution.

Obtaining of ceramics based on (Zr, Mo)B_2 solid solution

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New UHTC are materials with a core-shell microstructure or fully solid-solution composites. In this work, our goal was to obtain ceramics with the (Zr, Mo)B_2 matrix by the *in-situ* reaction of ZrB_2-$MoSi_2$ and ZrB_2-$MoSi_2$-B_4C powders. We chose the (Zr, Mo)B_2 solid solution because it shows the highest oxidation resistance among all ceramics based on ZrB_2. We observed that the degree of transition is affected both by the temperature of the densification and by the amount of B_4C in the material. Oxidation resistance and high-temperature strength depend on the amount of solid solution. The highest properties were obtained during the formation of a 100% solid solution, which was possible at a temperature of densification of 2000°C with a boron carbide content of 5 vol.% or obtaining a material at a temperature of 1850 ° C with followed annealing in vacuum at a temperature of 2000°C.

Control of powders morphology as a way of improving of Y_2O_3 ceramics properties

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Optical Y_2O_3 ceramics are actively researched as a multifunctional material and found wide practical application due to its mechanical and optical properties, chemical and thermal stability. This material should be possibly defect-free since the presence of pores deteriorates its optical and mechanical properties. Therefore, the choice of raw materials and the study of the effect of processing methods are fundamental. This work aims to study the influence of the initial powders and their milling conditions on the characteristics of Y_2O_3 transparent ceramics.

The morphology and sintering behavior of four different commercial Y_2O_3 powders after milling under different conditions was investigated. 3 mol.% ZrO_2 was used as a sintering aid. The samples were compacted by uniaxial pressing and CIP, and sintered in air at 1600°C for 4 h or in vacuum at 1735°C for 32 h.

Firstly, the powders after milling at 80 rpm for 22 h were analysed. All studied powders were characterized by different values of specific surface area, particle size and agglomeration, which influenced densification. Among all samples obtained by sintering in air, only one provided a high density and uniform microstructure, and was thus used for further studies.

Influence of milling conditions on powders parameters and properties of vacuum-sintered Y_2O_3 ceramics was determined. We found that 300 rpm for 65 min is optimal for obtaining powders with high sinterability. The specific surface area of this powder is 21.3 m^2/g, and the average particle size is 480 nm. Decreasing of milling speed leads to an increase in the particles size. An increase in the milling time to 10 h is accompanied by an agglomeration of particles. Y_2O_3 vacuum-sintered ceramics were characterized by a relative density of 100% and transmittance of 78.1% (1100 nm).

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Hybrid Additive Manufacture and Materials for Advanced Functional Ceramics

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Advanced ceramics (ACs) are high performance materials with exceptional properties and widespread applications. Material Extrusion (MEx) has been proposed as a method to increase the geometrical, material, and functional complexity for next-generation components, for example, using hierarchically structured, biomimetic materials, or low temperature co-fired ceramics with embedded electronics and sensing for harsh environments. However, MEx tends to offer lower densities and poorer resolution when compared to conventional ceramics processing routes, which limits the viability of commercial adoption. This research takes a dualistic approach to overcome these limitations; aiming to complete a thorough *material study* and to implement the results within a *hybrid manufacturin*g process. The material study will capitalise on the similarity between MEx feedstocks and conventional ceramic materials – using water to create plasticity in a powdered ceramic body – to elucidate the science that links material formulation, rheology, and printability. Meanwhile, the hybrid process combines MEx with green machining and assistive technologies within a single platform to remove surface defects and improve the manufacturing tolerance. Additionally, a custom software package has been developed to fulfil the control requirements of the bespoke, digitally driven process. So far, parts made of Alumina (doped with Manganese Dioxide and Titanium Dioxide as sintering aids) have been produced in arbitrary geometries with reproducible shrinkages of 17%. Finally, the work streams will reconnect to demonstrate the fabrication of functional ceramic components for high value applications, aiming to achieve comparable production densities and accuracies to conventional methods.

Lifetime extension of high chromium ferritic porous alloys by application of ultra-thin alumina coatings

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Ferritic alloys can be employed in the form of porous supports, replacing ceramic components that allow for decreasing the price of solid oxide cells (SOCs). The disadvantage of using porous alloys is their high-temperature corrosion (> 500 °C). The formation of the oxide scale leads to a decrease in porosity and an increase in electrical resistance. To limit the undesirable oxidation of the alloys, ceramic protective layers are widely used. One of the most effective coating materials is aluminum oxide. Although the Al2O3 coating improves the corrosion resistance of the alloys, its poor electrical conductivity excludes it from some specific applications like solid oxide cell substrates and interconnectors. The SOC supports have to conduct the electrons from the cell, so high electrical conductivity is necessary. In addition to that the lowest possible cost of the device is desirable. In the last few years, porous, high-chromium alloys, which are cheaper than dense alloys, have been considered as support material for SOC interconnectors by several groups.

In this study, we evaluated the corrosion resistance of a porous high-chromium alloy after the deposition of an ultra-thin (< 1000 monolayers) ceramic protective coating using the atomic layer deposition (ALD) technique. We will describe the morphology changes of coated porous alloys oxidized at typical SOC operating conditions (800 °C). We will also discuss the obtained results in the context of the lifetime prediction of porous steel components in SOC applications.

Spark plasma sintering of TiCN at low temperature with Si aids

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The feasibility of densifying TiCN by spark plasma sintering (SPS) with Si aids at only 1400°C was investigated. Importantly, it is demonstrated that TiCN is fully densifiable at such a smooth SPS temperature even with only 5 vol.% Si aids, and that the resulting ceramics are superhard and have fine-grained microstructures. It is shown that this is possible because Si acts as a reactive sintering additive that promotes densification by transient liquid phase sintering, leading to the *in situ* formation of SiC, $TiSi_2$ and N-rich $TiC_{1-x}N_x$ second phases dispersed in the TiCN matrix. The formation of these second phases is consistent with the theoretically calculated pseudo-binary TiCN–Si phase diagram. Interestingly, it is also found that hardness of these TiCN-based multiphase ceramics is little affected, while toughness increases, with the increase of the proportion of Si aids up to 25 vol.%. Finally, implications of interest for the ceramic and hard-material communities are discussed.

Optimisation of environmentally friendly tool for geopolymer and zeolite preparation from kaolin

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Metakaolin is one of the most commonly used precursors for the geopolymer and zeolite preparation due to its high aluminosilicate content, and chemical purity. Generally, metakaolin is obtained by dehydroxylation of kaolinite structure by thermal activation. In this work, we prepared metakaolin through different activation routes, thermal and mechanochemical, respectively. Mechanochemically activated kaolinite (metakaolin) was chosen because it tends to follow the rules of green chemistry, by avoiding energy dissipation when high-temperatures are used. Geopolymers were additionally prepared using the conventional method of alkaline activation of metakaolin with sodium silicate solution. Zeolites were also prepared according to green chemistry rules and by mechanochemical activation instead of the commonly used, hydrothermal one. During the synthesis, both the water amount, and curing temperature were varied. First, the activation reactions were optimized, by monitoring the process and obtaining perfect values for most efficient parameters in terms of time, and other parameters such as temperature and milling speed. Furthermore, the correlation and difference between metakaolin prepared by two different methods were monitored.
All prepared samples were thoroughly characterised. For the precursor, we monitored the degree of dihydroxylation, i.e., the degree of activation of the kaolin, or metakaolin preparation. For this purpose, we used the X-ray diffraction method (XRD) as a function of reaction time (milling or thermal activation). Following characterisation was performed using Fourier Transformed Infrared Spectroscopy (FTIR), which again allowed us to gain insight into the dihydroxylation rate and, to confirm characteristic aluminosillicate bonds present in our samples. Finally, we used Scanning Electron Microscopy to monitor microstructure of our samples, precursors and prepared geopolymes/zeolites.

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Rheological behaviour of glazes for stoneware

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Glazes are always applied in utilitarian and decorative pieces, regarding the material type (porcelain, stoneware or earthenware) and during the finishing process of the pieces. In the ceramic industry, the glaze is always used as an aqueous suspension. Therefore, the thickness and characteristics of the applied layer depends on the application method. To avoid sedimentation, additives are used to improve the stability and fluidity of the suspension, i.e. to control its rheological properties. Deflocculants, binders and suspension agents are additives used to overcome the destabilizing effects created by the particles in suspension, making them away from each other. Polyelectrolytes are the most used ones, namely polyacrylates, sodium pyrophosphate and sodium salts (Dolapix PC67) among others derived from renewable resources as carboxymethyl cellulose. The use of these additives is well-studied in the ceramic industry, being in the last years also explored in suspension-based additive manufacturing technologies, a current area in constant progress. To keep up the industry evolution towards new and more sustainable production methods, the rheological properties of the glazes should be adapted. Therefore, the optimization of glazes composition and its rheological properties is of paramount importance, since the rheological behaviour should be tuned for a certain glazing process. Concerning this, in the present work different additives were tested as rheology modifiers of glazing suspensions. A opaque white glaze used in the ceramic industry was selected and additives, in proper amounts, were added. The rheological behaviour of the suspensions was assessed in a rotational rheometer and the viscosity *versus* shear rate curves, at 25 ºC, acquired. The experimental results showed that, depending on the additives, different viscosities were attained in rest and as the shear rate increases, which is mandatory for additives selection for each glazing process. The additive amount also has a greater influence on the suspension viscosity.

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Multifunctionalization of SS316L surfaces by immobilization of nano-thin MXenes-films for improving of biological responses

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Abstract

MXenes, as two-dimensional (2D) layered transition metal carbides or carbonitrides, have drawn considerable attention due to their unique properties. They have a wide range of applications, including drug-delivery systems, tumor therapies, protective or multifunctional coatings, and biochemical sensing. In this work, we focused on developing a new method for the deposition of $Ti_3C_2T_x$-MXene films on SS316L for improving biological responses. $Ti_3C_2T_x$ MXene was synthesized by selective etching of the Ti_3AlC_2 phase by using LiF/HCl solution. The obtained MXenes after characterization by scanning electron microscope (SEM), X-ray diffraction (XRD), and Energy-dispersive X-ray spectroscopy, were deposited on SS316L substrate to modify the surface. For the coating, the SS316L substrates were firstly functionalized with aminopropydiisopropylethoxysilane (APDS) to covalently attach 2D-nanofilms to the surfaces, and then coated with $Ti_3C_2T_x$ via dip coating method. The obtained MXenes coatings were examined in regard to their morphology, coupling behavior, and wettability. Moreover, the non-toxic and antibacterial behavior of the deposited MXene-coatings was confirmed by several tests. In our study, it was shown that the immobilization of MXenes on the SS316L surfaces has been successfully performed, and this method could be applied for creating adjusted surfaces for medical purposes.

Consolidation of ceramic permanent magnets by Radiation assisted sintering (RAS) in WC die

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Permanent magnets play an important role in modern devices and enabling technologies as they allow storing, delivering and converting energy. [1] Volume vise, Strontium and Barium Ferrites are one of the world's most-used permanent magnetic materials [2]. Although rare-earth magnets exhibit overwhelming superiority in performance the harmful environmental impact of their production, uneven distribution of raw materials and increasingly questionable supply chain force us to look for alternatives. One of the solutions can be found in the group of hexagonal ferrites [3], which do not contain critical raw materials [4].

In this study we explored Radiation assisted sintering (RAS) of strontium hexaferrite using tungsten carbide die in Spark Plazma Sintering (SPS) device , that enabled to isolate radiation as a sintering mechanism. Although ceramic magnets and radiation assisted sintering process have been known for some time, this is an less widely used approach to sintering this widely used magnetic material. This research focuses mostly on producing fully dense, bulk piece with satisfactory magnetic properties. Several parameters were tested, among which the sintering temperature and retention time at that temperature played the most important part. Density of the pellets produced was measured geometrically, the magnetic properties were determined using vibrating sample magnetometer (VSM). Based on the obtained data during sintering on the SPS apparatus we prepared a numerical simulation of sintering, where the heat was produced in tungsten carbide die by Joule heating and transferred to the sample via thermal radiation. Doing this we were able to better understand the course of events in the heating die. Compared to the conventional sintering radiation assisted sintering offers substantial energy savings, which would have a positive effect on the environment and at the same time reduces costs of production.

Key words: Strontium hexaferrite, magnets, WC die, sintering, RAS

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New fast routes for the synthesis of soft ferrites by microwave decomposition of hydrotalcites for functionalization of porcelain substrates

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Ferrites are iron compounds with magnetic properties and are widely used in technological applications such as sensors, information storage devices and catalysts [1]. The synthesis of ferrites from hydrotalcites using microwaves has been the subject of study in this work due to the advantages offered by this method compared to traditional synthesis processes. Hydrotalcite in its origin is a compound of aluminate and magnesium carbonate that is presented in the form of thin layers. It has been demonstrated that the decomposition of Ni-Zn-Fe hydrotalcites yields ferrites that significantly improve the magnetic and structural properties of the particles. In addition, the use of microwaves allows a faster and more efficient synthesis compared to traditional high-temperature heating processes. Particles with smaller diameters and higher purity are produced compared to traditional processes. [2]

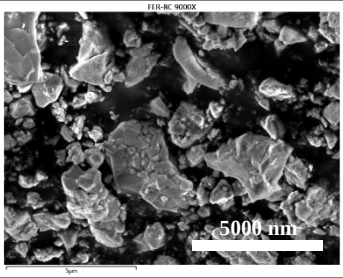
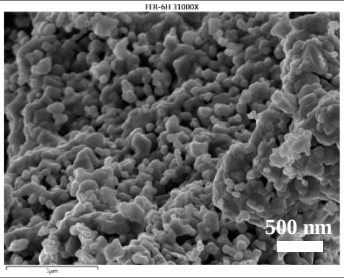
Ceramic method	Hydrotalcite decomposition
	

Table. 1: *SEM micrograph of ferrites synthesized by ceramic route and by decomposition of hydrotalcites.*

Regarding the application of these nanoparticles, in the present project, a modified ceramic support is developed. This ceramic material is suitable for electromagnetic induction plates that are traditionally made of glass-ceramic material. Materials for this type of applications are mainly characterized by their high resistance to thermal shock and by their high magnetic permeability coefficients that reduce the shielding of the magnetic field.[3]

In order to develop this ceramic plates, with the magnetic, thermal and mechanical properties required by this type of heating technology, a porcelain stoneware composition has been doped with synthesized particles of mixed Ni-Zn ferrites with low coercivity coefficients. [4]

By magnetizing the porcelain, eddy currents are established which heat the whole mass of the support by Joule effect. This implies a reduction of the thermal gradient inside the piece as opposed to a diamagnetic composition. [5]

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Material thermal extrusion of conductive 3D electrodes using highly loaded graphene and graphite colloidal feedstock

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Evolution in the energy storage field requires the development of more sustainable and efficient high-performance electrochemical storage devices (EESDs). Additive manufacturing (AM) opens unique opportunities to improve the efficiency and the electrochemical response by the design of 3D electrodes and the increasing of exposed active surface area impossible to achieve by other processing techniques. In other to reach high-performance EESDs, carbonaceous species are well-established as electroactive materials owing to their electronic properties, low cost, and sustainable origin. Although the materials have been widely investigated, there is a lack of feedstock development for the fabrication of EESDs by AM technologies. This work presents the fabrication of metals free light conductive filaments for material thermal extrusion (MTE) by formulating PLA-based composites with a high content of colloidal graphite and graphene (15-25 vol.%). By the colloidal characterization and surface modification of the carbonaceous species, an improved distribution and enhanced platelet/polymer bond were achieved, provoking the inorganic phase's orientation during the extrusion process. Prepared filaments were characterized to analyze the influence of the microstructural anisotropy on thermal, thermorheological, mechanical, and electrical properties. Subsequently, developed filaments were used to print complex electrodes by MTE, enhancing the electrochemical properties of the electrodes with superior control of the macrostructural design. The developed colloidal filaments with an oriented inorganic microstructure, not only allow a defined shaping of the outer structure of the electrodes but also let the design of completely different microstructures. Consequently, different microstructural configurations were printed increasing the number of conduction paths in the polymeric matrix to achieve high electric conductivities (22 S.m-1) in printed electrodes.

Obtaining superparamagnetic MnO nanoparticles as contrast agents

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In recent years, nanomedicine or the medical application of nanotechnology has established itself as an area of great potential for solving the major problems and challenges related to the diagnosis and treatment of such as cancer. Covering everything from the design of nanomaterials and nano-biosensors to the to the development of new marker agents and/or local drug distributors, nanomedicine encompasses nanomedicine encompasses a whole range of nanoscale technologies whose main objective is to revolutionise nano-scale technologies whose main objective is to revolutionise the world of disease through the monitoring, early diagnosis and personalised treatment. personalised treatment. Specifically, in the field of monitoring, there is a strong demand for new materials with optimised functional properties to replace current molecular counter agents, which have a number of weaknesses (not very sensitive and photo-stable) or those based on semiconductor quantum dots (much more specific and stable, but mostly toxic). Within this framework, the present work aims to implement new synthesis strategies with which to design new contrast agents for early detection of diseases by MRI. Specifically, the nanoparticles obtained so far are based on superparamagnetic inorganic MnO nanoparticles. These nanoparticles are also easily dispersible in aqueous solutions by using oleic acid as a surfactant.

Effective immobilization of TiO_2 nanoparticles for water remediation purposes

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Ceramic nanoparticles are emerging as a new class of pollutants. The increased use and diversity of products containing metal oxide nanoparticles implies that many will enter the sewerage system, eventually ending up in the aquatic environment. Actually, evidence of water contamination by inorganic nanoparticles has been already observed in raw sewage and wastewater effluents, for example revealing an anomalous occurrence of nanometric titanium dioxide as released from different commercial products and industrial processes. In this context, several alternatives are being considered to conventional processes of agglomeration, settling and filtration, since they are not efficient enough to isolate the suspended nanoparticles from aquatic bodies. This work presents a novel, bioinspired approach to water remediation in which poly-peptide molecules with specific affinity for non-biological inorganic materials are used to immobilize TiO_2 nanoparticles suspended in an aqueous batch. Based on the concept of molecular recognition, the proof of concept demonstrated here comprises a two-stage incubation methodology, whereby titanium oxide nanoparticles in very low concentrations (0.04 mg/ml and below) can be effectively removed from a water suspension after being recognized and selectively bound to a properly engineered inorganic-binding peptide. Operating all the time under extraordinary soft conditions of temperature and pH (T = 37 °C, pH 7). In addition, there is no evidence that the peptides are lixiviated throughout the process, further precluding any negative effects on the quality of the remediated water. The whole procedure is systematically monitored by FESEM, micro-Raman and UV–vis analytical measurements and reveal that the immobilization mechanism fits widely in Langmuir's isothermal adsorption model, with the biomolecule acting as a kind of molecular glue that ensures a stable adhesion with the noxious nanoparticles.

Additive Manufacturing of Ceramic Supports for Photocatalytic Degradation of Hydroxychloroquine

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Pollution of the aquatic ecosystems from medicines and pharmaceutical products poses a threat to environmental and global health. Consequently, it is necessary urgently to remove them from the environment. The aim of advanced oxidative processes (AOPs) is to produce highly reactive radicals such as hydroxyl radicals ($E°$=2.72 V) that can break down the structure of pollutant molecules to reduce organic contaminants in water. Heterogeneous photocatalysis, which involves light activation of semiconductor materials, stands out among these processes. Due to its great chemical stability, nontoxic, low cost, and strong photocatalytic activity, titanium dioxide (TiO_2) has the potential for use as an efficient photocatalyst for the removal of a wider number of pollutants present in the environment.

Catalysis processes are increasingly being influenced by additive manufacturing. Their connection is based on the need to intensify catalytic processes to make them more efficient and sustainable. Additive manufacturing can satisfy such a need, generating materials with an advanced design, easy production, and great adaptation, in addition to their high catalytic functionality. The immobilization of photocatalysts is considered as an advantage in wastewater treatment due to the durability and stability of immobilized photocatalytic materials compared to suspension form.

In this work, the TiO_2 was immobilized on the surface of the 3D-printed ceramic supports produced by the fuse filament fabrication technique. The post-processing technique of TiO_2 immobilization on the 3D printed supports using solvothermal synthesis in situ growth method was applied. The photocatalytic activity of immobilized TiO_2 was evaluated by the degradation of hydroxychloroquine from an aqueous solution under UV-Vis illumination. A comparison was made between impregnated 3D printed ceramic support and the powdered TiO_2. The prepared photocatalyst was characterized using X-ray diffractometry (XRD), scanning electron microscope (SEM), and energy dispersive spectroscopy (EDS). The obtained results of performed photocatalytic tests show the efficient degradation rate of hydroxychloroquine using the heterogeneous photocatalytic process.

Bioresorbable pyrophosphate ceramics produced by stereolithographic 3D printing

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The most promising materials for osteoplasty are bioresorbable materials, which can perform a supporting and guiding function at the initial stages of use. Calcium pyrophosphate belong to such materials, has good biocompatibility, bioactivity and optimal bioresorption rate. Also the implant must possess a system of macro- and micropores that promotes diffusion of ions and the sprouting of the forming bone. The method of stereolithographic 3D printing is used within the framework of this study to obtain porous materials, as traditional methods do not make it possible to control the final structure of the material.
We investigated the possibility of using a sintering additive of $CaNa_2P_2O_7$ to improve calcium pyrophosphate sintering. As a result, it was found out that the presence of a sintering additive allows to reduce significantly the temperature (700°C) required for sintering of the forming ceramics. The resulting material reaches a compressive strength of up to 140 ± 10 MPa and a density of 93%. However, the materials obtained this way may contain residual carbon, potentially exhibiting cytotoxic effects on body cells. Therefore, it was proposed to use powder mixtures to form ceramics based on calcium pyrophosphate at a higher temperature (1000°C), at which the burnout of residual carbon occurs. As a result, dense pyrophosphate ceramics with compressive strength of 154 ± 17 MPa and a density of 95% were obtained. We investigated the effect of the morphology and composition of powder precursors on sintering processes at a given temperature. Finally, fabrication technique of macroporous ceramic based on calcium pyrophosphate with a given architecture has been developed. Kelvin structures with a porosity of 70% and a compressive strength of up to 5 MPa were obtained. Chosen heat treatment mode ensures the absence of visible cracks and other defects in the material.

Development of waste-based colored glazes for stoneware

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Nowadays, the concept of sustainability holds tremendous significance in corporate discussions, owing to energy prices and the depletion of raw materials that directly impact production costs. Additionally, the instability of the energy market and growing concerns regarding CO_2 emissions further highlight the need for sustainable practices. This work stems from our concern for these issues and our commitment to a circular economy, whereby byproducts and residues are reused or repurposed rather than being sent to landfills. Our focus was on minimizing waste and maximizing resource efficiency to create a more sustainable and environmentally friendly future. The objective of this study is to develop a colored waste-based glaze for stoneware. Four byproducts/residues were studied: two from outside companies, and two from GRESTEL. First, they were characterized using DRX, FRX, SEM and DTA-TG techniques. Their fusibility and color (CIElab system) were also evaluated. New glaze formulations were prepared by incorporating of these byproducts/residues (1, 3, 10, 15% wt.). Pre-treatment methods such as milling and sieving were also evaluated. The resulting formulations were sorted and selected according to their aspect (transparency, opacity, etc..) and color. The selected waste-based glaze formulations were subjected to quality control tests, such as resistance to microwaves, ice, metal marking, food attack, leaching and cracking. Ultimately, the goal of this study is to create a sustainable and cost-effective solution for stoneware glazes that maximizes the use of byproducts/wastes.

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Study of WC-based inks for 3D printing

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Manufacturing of monolithic binder-less tungsten carbide (WC) parts presents many challenges since pressure-assisted sintering routes, like hot pressing or spark plasma sintering, are necessary for complete densification due to WC strong covalent bonds. As such, only simple shapes can be obtained. The additive manufacturing approach has been considered in recent years to reduce the machining costs and obtain structural ceramic components with complex geometry. Different 3D-printing methods have been proposed for ceramic materials, like VAT polymerization, powder-bed techniques (i.e. Selective Laser Sintering or Binder Jetting), or Direct Ink Writing (DIW). DIW was chosen in our study because it allows to obtain pastes with higher ceramic loadings compared with typical colloidal suspensions methods, like slip or tape casting, which would require instead high organic fractions to stabilize high density powders, like WC, and in view of its comparatively low cost and low production time with respect to the other additive manufacturing techniques. Here, we investigate the feasibility and report the rheological behaviour of gel-based binderless-WC-loaded inks for DIW. The ultimate goal is to obtain a meso-structured porous WC scaffold to be infiltrated with a metallic phase for application in impact resistant parts that combine compression resistance and fracture toughness. Therefore, preliminary diffusion couples between WC and a Ti-alloy are also presented to define the best infiltration temperature that allows complete filling of the scaffold and minimal carbo-reduction of the metal.

Ion Migration in Glassware and Crystal

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Lead glasses have been widely used due to their excellent properties, namely high refractive index and transparency, high density, and good sonority. However, the toxicity of the lead compounds has led to the establishment of lower maximum levels that can be released to products and the environment. Thus, there has been many attempts to formulate alternative glasses with the same properties. The present work, in partnership with the largest Portuguese producer of leaded glass (Vista Alegre Atlantis, S.A.), and following the development of a new lead-free formulation, aims to develop a model that allows the prediction of leaching/migration of chemical elements, through the study of leaching in simulated solution, according to ISO 7086, at room temperature and at higher temperatures.

Recovery of metallurgical industry waste in ceramic products

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The reuse of waste as a secondary raw material has been increasingly studied by the scientific community. The wire drawing process generates two types of solid wastes, CM and CR, with an iron content of > 90 wt.%, that are usually landfilled. In this sense, this work seeks to study a new application for these wastes: the development of colored ceramic pastes and glazes substituting the pigment used nowadays.
For this purpose, the influence of pre-treatments (sieving at 250 and 63 μm and milling + sieving at 63 μm) and incorporation levels (by addition) of 3, 5 and 10 wt.% in pastes and 1 and 3 wt.% in glazes; of both wastes were evaluated on the stoneware and glazes' characteristics. Preliminary results showed that the incorporation of this type of waste was not viable in glazes since all developed samples presented defects (pin-hole). Regarding ceramic bodies, two different colours were obtained, a dark grey and a reddish one, mainly due to the developed crystalline phases of the wastes during the sintering process. Samples' properties will be further evaluated, and their properties will be compared to those of the currently produced by GRESTEL.
This new added value application leads to a decrease in environmental impact and production costs since there is a reduction of the virgin raw materials consumption, namely, pigments. Furthermore, this solution contributes to the circular economy between two distinct sectors: the metallurgic and the ceramic industry.

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