Book of Abstracts

Young Ceramists Additive Manufacturing Forum

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Thermoplastic 3D printing of zirconia parts with varying infill strategies

Wednesday, 9th November - 12:25: A - MATERIAL JETTING - Oral

Ms. Ipeknaz Özden [1], Mr. Steven Weingarten [2], Dr. Aljaž Iveković [1], Mr. Uwe Scheithauer [2], Dr. Andraž Kocjan [1]

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Thermoplastic 3D printing (T3DP) is an additive manufacturing (AM) method which relies on the deposition of a thermoplastic feedstock in the form of droplets to form 3 dimensional objects. Deposited droplets are fused together with a certain fusion factor to create lines which are then fused further to create layers. Such a building concept allows changing the infill strategy which is known to have an effect on the final mechanical properties of the parts.

In this work, we aim to show the effect of varying infill strategies on the flaw types and final mechanical properties of the parts prepared by T3DP. A zirconia wax-based feedstock with 40 vol% solid loading was used to fabricate rectangular bending bars with 3 different infill strategies: 0°, 90° and a combination of two. Parts were wick debinded and sintered to reach high theoretical densities above 99%. Mechanical characterization was done by 4-point bending test, Weibull analysis and SEM imaging. 0° was found to be the best infill strategy of all, with a characteristic strength of 700 MPa and a Weibull modulus of 7.0 while the combination of two strategies resulted in a decrease in the characteristic strength and the Weibull modulus to 624 MPa and 6.0, respectively. Furthermore, microstructural analysis with SEM, phase characterization with XRD and examination of volumetric population of flaws present in sintered parts by X-ray CT analysis was done. The results showed that varying the infill strategy led to variations in the level of fusion of subsequent droplets and lines, that consequently resulted in different mechanical properties. Moreover, the results obtained with T3DP were comparable to those obtained by other AM methods which is encouraging for pursuing further research with this method such as multi-material fabrication.

Oral Presentations - Binder Jetting

Additive Manufacturing using the Layerwise Slurry Deposition in combination with binder jetting (LSD-Print)

Thursday, 10th November - 12:20: D - BINDER JETTING - Oral

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In the last decade the usage of additive manufacturing became more and more relevant. Beginning its evolution with the easier processable material of polymers, the possibility of more elaborate 3-dimensional components which promised lower material usage attracted the metal and ceramic disciplines as well.

One of the main manufacturing methods for ceramic additive manufacturing, the powder bed binder jetting, faces the challenge of the compaction and homogeneity of finer powders. In the process of the LSD-print the powder is dispersed into a slurry, which is deposited layer by layer by means of a doctor blade. This results in a higher packed powder bed and higher mechanical properties of the green body. Thereby a wider range of ceramic materials is enabled, even with before unprintable materials and particle sizes in standard powder-based binder jetting.

In this work the LSD-process will be introduced and the application ranging from silicate ceramics to high performance ceramics will be shown, with focus on silicon carbide ceramics.

Additionally recent developments and planned progression of the LSD technology in the next future will be talked about.

3D printing of lead-free KNN by binder jetting

Thursday, 10th November - 13:55: D - BINDER JETTING - Oral

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Binder Jetting of piezoceramic materials offers the possibility of producing application-oriented geometries while maintaining a high process throughput. This solution is attractive for lead-free ceramics, such as sodium potassium niobate (KNN), whose scarce performance with respect to PZT may be compensated for by specific design.

In this work, irregular and spray-dried granulated feedstocks were employed to produce simple and complex specimens. First, KNN stability in aqueous suspension with a polyanionic dispersant was quantified by ICP-OES and spray-drying conditions were investigated to produce spherical particles with a reduced average diameter. Secondly, printing parameters, in particular binder saturation, were optimised to achieve a homogeneous powder bed and to maximise the green bodies density. Then, heat treatments (debinding and sintering) were studied to promote diffusion mechanisms and obtain higher densification rate. Double sintering revealed to be necessary to achieve a sufficient mechanical strength, nevertheless all samples were affected by a large residual porosity (up to ~40%). SEM observations highlighted a preferential orientation for pores development along the interlayer directions, as already observed for ceramic shaped by binder jetting.

Finally, piezoelectric properties were measured both along the parallel and transverse direction with respect to the printing orientation to identify potential anisotropy induced by porosity. d_{33} values of 80-90 pC N^{-1} in both cases revealed a minimal influence of the building direction and optimal performances with respect to those of correspondent die-pressed components. The Figure of Merit for their employment in direct mode showed an improvement close to 100%, thus confirming the great potential of this manufacturing route for specific applications, such as in the field of sensors.

Synergy of powder bed 3D printing and RBSiC

Thursday, 10th November - 14:15: D - BINDER JETTING - Oral

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Powder bed 3D printing, also known as binder jetting, is one of the most efficient additive manufacturing technologies to create large and complex shaped ceramic parts. It enables the production of prototypes as well as final products, which may not be realized by established shaping techniques. One main drawback of the technology is the immanent porosity of printed green bodies, due to dry powder deposition methods. This usually prevents the creation of parts with material properties which are technically sufficient.

In contrast to the vast majority of technical ceramics, powder bed porosity is not an obstacle for the production of components made of reaction bonded silicon carbide (RBSiC). In fact, a porous network is a prerequisite for the liquid silicon infiltration (LSI) process which follows the creation of green bodies. Thus, it is feasible to manufacture complex and crack free parts with material properties that are comparable to conventionally processed RBSiC.

The presentation will give some insights into material and process development, which was key to enable the production of three dimensional printed RBSiC with excellent properties. Today we can exploit process-related advantages of additive manufacturing providing a new dimension of constructive design potentials and address demanding market segments of space, mechanical engineering, and thermal process technology.

Powder-bed binder jetting of cementitious materials with inorganic binders: parametric analysis of the effect of powder feedstock and fluid properties on the volume of residual voids using design of experiment

Thursday, 10th November - 14:35: D - BINDER JETTING - Oral

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The present work addresses powder-bed binder jetting by selective cement activation (SCA) to produce magnesium oxychloride-based components. Its focus is on the effect of powder-bed and fluid properties on the porosity of printed parts. Depending on the properties of the activator solution and of the powder bed and the kinetic energy of jetted fluid, droplets' impact leads to different consolidation mechanisms. In addition to the powder bed feedstock and fluid, cement additives (e.g., methylcellulose) modify powder-bed properties; printing strategies (e.g., voxel size and binder saturation level) change the kinetic energy of droplets, as well. In this study, a three-level general factorial design was employed to evaluate the effect of "water-cement ratio (w/c)", "fluid pressure" and "methylcellulose content" on the residual voids volume, using mercury intrusion porosimetry and μCT. The volume of residual voids quantifies the porosity and homogeneity of printed cubes. Analysis of variance (ANOVA) indicated that the studied factors are significant. Higher w/c ratios lead to smaller porosity which instead increases significantly with the addition of methylcellulose (MC). The optimum values of fluid pressure to have minimum voids are in the mid-levels. Methylcellulose content has an interaction with two other factors. It retains the solution in the dispensed locations for cement reaction, but if the liquid is sprayed at low pressures, the fluid migrates into the powder bed only after dissolving MC. Increasing fluid pressure leads to rearranging aggregates and leaves residual voids in the structure, while low pressures do not have enough kinetic energy to penetrate the deposited layer; both cases include a high volume of porosities. Quadratic interpolation modeled the volume of residual voids as a function of studied factors to analyze the powder-binder interaction at the micro level.

Large Scale Additive Manufacturing of Inorganic Components Using Binder Jetting

Thursday, 10th November - 14:55: D - BINDER JETTING - Oral

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AM with inorganics materials is an innovative, challenging research field enabling the production of parts with complex structures, and specific functional or structural properties.

In the current research, a large-scale binder jetting printer was employed to fabricate inorganic binder-based components. To achieve the target properties, the dry powder mix was comprised of specific aggregates, reactive binders and byproducts. The printing mix was activated by the selective deposition of water and solutions (e.g., alkaline solution). The reaction parameters were controlled in order to achieve an adequate setting time enabling rapid printing with suitable resolution, and the building up of a structure at the macro-scale (meter-size).

The production process and the printing parameters were simultaneously adapted to the specific conditions. The main chemical, physical, and mechanical characteristics of the printed parts were investigated.

Different fields of application (e.g., construction, fire resistance, marine protection, urban environment, art and architecture, etc) were studied and the specific advantages and the correlated functional properties were highlighted.

Mechanical response from room temperature up to 600 ºC at the submicrometric length scale of WC-Co produced by an AM route based on an inkjet printhead

Thursday, 10th November - 15:15: D - BINDER JETTING - Oral

Ms. Guiomar Riu [1]***, Dr. Joan Josep Roa Rovira*** [1]

1. *STEROS GPA INNOVATIVE, S.L.*

The present work is focused on dry-electropolished WC-Co. A systematic nanomechanical study of a 3D-printed WC-Co grade is investigated. In doing so, nanoindentation technique is implemented, while the main deformation or damage mechanisms induced at the submicrometric length scale are investigated at different temperatures, from room up to 600 ºC. In general, three different approaches are followed to accomplish this research: (1) assessment of intrinsic hardness values as a function of crystallographic orientation from a room temperature up to 600 ºC and (2) the determination of effective hardness and flow stress through the Tabor's equation of the metal cobalt binder. Finally, the elastic strain to break has been also determined for the main crystallographic orientations for the WC particles as a function of the temperature. The preliminary results highlight that the strength reduction with increasing temperature is attributed to metallic binder softening. On the other hand, the WC particles present an isotropic behavior when the testing temperature is over 500 ºC because, inside these particles, the dislocations and the stacking faults are the main deformation mechanisms induced at intermediate/high testing temperature.

Oral Presentations - Vat photopolymerization

Ceramic meta-material created via two-photon-polymerization for powder processing of yttria stabilized zirconia

Wednesday, 9th November - 14:35: B - VAT PHOTOPOLYMERIZATION - Oral

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Additive manufacturing of ceramics is of huge scientific interest. It brings ceramic properties into applications, where conventional ceramic manufacturing hits its borders. One of those is the creation of high resolution and lightweight ceramic objects. Filigree structures can be manufactured via two-photon-polymerization (2PP), a vat-polymerization technique operation with a non-linear light source and for that crucially requires transparent resins, which is a diametrical demand brought upon usually fully opaque ceramic resins and slurries.

In this study we present a transparent and photocurable resin with yttria stabilized zirconia particles, which are of such a small size, that scattering doesn't play a role any more at 800nm laser wavelength. The particles are well dispersed and can be stacked to weight fractions up to 80wt%. Sintered ceramic structures with a positive resolution of down to 500nm obtain filigree and overhanging features. The mechanical properties of those structures are dependent on the chosen geometry, infill pattern and sintering temperature varying from 800 to 1450°C. The resulting compressive strengths are close to monolithic yttria stabilized zirconia of a few thousand MPa, but at lower densities of 1-4g/cm^3. A ceramic meta material is born, where the mechanical properties of yttria stabilized zirconia are altered by changing geometrical parameters and creates a new ceramic material class.

Advanced photosensitive ceramic suspensions formulation and optimization through UV-rheology

Wednesday, 9th November - 14:55: B - VAT PHOTOPOLYMERIZATION - Oral

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1. *Universitat de Barcelona*

Rheology provides important information on the formulation and how the different ceramics photosensitive suspensions work for stereolithography (SL). For all the SL technologies a viscosity range is required for optimum production. Higher viscosity can lead to a lack of resin movement, causing an extremely long printing time or even making it impossible to print. On the contrary, a very low viscosity can lead to stability problems, which will lead to a sedimentation process that can cause inhomogeneities in the final pieces. Typically, SL resins have high viscosities related to large amounts of ceramic particles. These high amounts of ceramic are necessary to increase the densification of the final piece once the polymeric phase is burned to be able to obtain a ceramic piece as dense as possible. This high content of ceramic causes, in addition to changes in viscosity, changes in the photosensitive behaviour of the mixture, largely influenced by light scattering.
In this work, UV-rheology is employed to understand the photocuring behaviour of alumina suspensions in function of ceramic loading. UV-rheology enhances the possibility of determining the mechanical conversion in function of the time and intensity applied, being the perfect complement to DSC-UV. Different contents up to 80 wt% have been studied. The effect of the particle size and specific surface, of the alumina employed in the suspension, and its photosensitive and rheological behaviour are also studied.
Furthermore, the final formulation of these suspensions does not include only the resin and the ceramic, but also other additives. Those with a higher percentage are usually additives to improve the debinding of the piece. One of these additives used with good results is PEG. As they occupy an important percentage of the final formulation, their effect on the rheological behaviour and reactivity has also been determined.

A multi-physics approach to rationalize the properties of 3Y-TZP-based stereolithography pastes

Wednesday, 9th November - 15:15: B - VAT PHOTOPOLYMERIZATION - Oral

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Ceramic laser stereolithography (SLA) is an additive manufacturing technique with a high design freedom and resolution, required for producing complex and dense ceramic parts, notably useful for implants development. In this context, we aim to understand how formulation ingredients and mixing procedure could change paste characteristics influencing three SLA printing aspects of prime importance: I) the paste rheology, II) the photopolymerization and III) the debinding. To do so, we produced several SLA pastes containing 45 vol.% in ceramic particles functionalized with different dispersants , functionalization techniques and acrylate oligomers. We ran in-depth rheological and rheo-SAXS investigations with a rotational rheometer to provide quantitative parameters describing the paste “printability”, to compare our pastes and to understand particle restructuration under shear stresses.

Besides, solidification and polymerization of printed samples made from homemade pastes were investigated by FT-IR and low-field NMR relaxometry before to be compared to Jacob’s equation fit of single layer samples measurements. For a commercial paste, we observed that the evolution of polymerization depth, monomer conversion rate and solidification, regarding laser energy were similar regarding laser energy. Combining FT-IR and NMR probes further made possible to estimate absolute (average) values of the monomer conversion while varying the processing route and the paste formulation.

To this end, we were able to spot the differences brought by paste ingredients and process on monomer conversion and solidification and not only by sample shape and surface observation

Finally, thermal (TGA and DSC) as well as dynamic mechanical analyses (DMA) were performed on printed samples while heating, serving to clarify the phenomena occurring during the debinding step, allowing to reduce risks of fracture during the processing.

Innovative zirconia-based material shaped by SLA 3D printing

Wednesday, 9th November - 16:05: B - VAT PHOTOPOLYMERIZATION - Oral

Mr. Maxence BOURJOL [1], Mr. Christophe CHAPUT [1]

***1.** 3DCERAM Sinto*

Introduction

Today a strong need for high-performance ceramic materials combined with new shaping techniques appears on the market. 3D printing technology of ceramic objects is in strong development as it opens new perspectives. While ceramic materials are brittle and subject to catastrophic failure that is difficult to predict, ceria-stabilized zirconia-based composites can provide new ceramic materials with a plastic deformation domain before rupture, excellent resistance to processing flaws and a Weibull modulus approaching that of a steel.

In this study, we explored the influence of SLA stereolithography shaping on this new ceria-stabilized zirconia-based material with unique mechanical behavior.

Experimental

Firstly, a slurry compatible with the CERAMAKER process was developed. Then, the specific object of the study was to evolve the influence of the shaping parameters (layer thickness, lasing power, etc.) on the green part quality. Finally, the effect of the sintering temperature on the microstructure and mechanical properties of optimized SLA-printed material was also studied.

Conclusion

Through this study, 3DCERAM has been able to prove that this new material could be shaped by 3D SLA. Different sintering parameters have been studied to optimize densification and material properties. SEM observations show original layer structures and microstructure development attributable to the shaping process.

Additive manufacturing of zirconia-based high-performance ceramics with superior flexural strength and improved optical properties

Wednesday, 9th November - 16:25: B - VAT PHOTOPOLYMERIZATION - Oral

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Yttria-stabilized zirconia (YSZ) is a high-performing ceramic material for technical, as well as medical applications due to its extraordinary mechanical properties, chemical inertness and biocompatibility.

Compared to conventional ceramic shaping methods, Additive Manufacturing (AM) enables the production of zirconia parts for a wider range of complex designs. One of these AM methods is Lithography-based ceramic manufacturing (LCM), which is a vat photopolymerization-based technique to process slurries, consisting of ceramic powder dispersed in photosensitive resin, to green parts by selective exposure to light.

This contribution will focus on the development of YSZ systems for outstanding performance concerning mechanical properties as well as optical qualities.

With the LCM technology it is possible to manufacture parts from 3mol% yttria-stabilized zirconia with a 4-point-bending strength of >1000 MPa, which is already comparable to values achieved by conventional shaping methods. Further improvement is aimed at by evaluating the production parameters of the slurry as well as adjusting the thermal post processing routine.

Furthermore, the usage of different zirconia grades, e.g., powder containing a different yttria content, is considered to enhance the mechanical strength of the parts or change the translucency.

Another topic is the addition of pigments to zirconia to obtain coloured ceramic parts, mainly for aesthetical applications – such as dental or luxury - while maintaining adequate mechanical properties. The colouration was achieved either by direct addition of pigments or utilizing precursors which only develop the final colour during the sintering to not impede the initial printing process. The effects of these additives on the microstructure and the ensuing impact on the mechanical strength are also shown and discussed within this work.

Tailoring of 3D-printed ceramic microstructures through rapid sintering

Wednesday, 9th November - 16:45: B - VAT PHOTOPOLYMERIZATION - Oral

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Nowadays, numerous technologies for additive manufacturing (AM) ceramics are being developed, opening the path to fabricate ceramic components of high geometric accuracy and complexity. Stereolithographic 3D-printing technique is commonly used to print ceramics from slurries containing a relatively high amount of polymeric binder. As a result, debinding and further sintering of the printed parts are time and energy demanding processing steps, which may limit the mass production of certain ceramic products. In this regard, rapid sintering technologies, such as FAST or SPS have been recognized as a game-changer that can densify ceramics with fine-grained microstructures within a few minutes, raising the question on how the mechanical properties may be affected. Whereas the mentioned rapid sintering technologies are limited to simple geometries, in this work, a pressure-less rapid sintering process was used to obtain the ability of rapid sintering ceramic parts of complex shapes. In a hollow cylindrical graphite die, alumina ceramics were sintered through radiation heat transfer within less than 20 minutes. It was investigated, how temperature and dwelling time effects microstructural, physical and mechanical properties. Dense, pure alumina with a grain size below ~0.5 µm could be manufactured, resulting in a biaxal strength (σ 0 =860MPa) 30% higher than that of conventionally sintered alumina parts. This increase may be associated with the fine-grained microstructure and low internal micro-residual stresses, opening the path to sinter complex 3D-printed alumina parts in very short times.

Lithography-based Additive Manufacturing of Alkaline Niobate-based Piezoelectric Ceramics

Wednesday, 9th November - 17:05: B - VAT PHOTOPOLYMERIZATION - Oral

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Piezoelectric ceramics are of significant interest in a wide range of applications, from ultrasonic transducers to energy harvesters. Thanks to the augmented design flexibility and microstructure controllability, Additive Manufacturing (AM) is altering the way piezoelectric ceramics are processed. Here, we focus on lithography-based AM of potassium sodium niobate [(K, Na) NbO_3, KNN] that is a potential alternative to lead-based piezoceramics. Lithography-based AM of ceramics involves 1) designing of photopolymerizable ceramic suspensions, 2) selectively and layer-wise curing of suspension to fabricate green bodies through pattering the light in Digital Light Processing (DLP) technique, 3) heat treatment of green bodies to produce dense ceramics. So, the quality and performance of piezoelectric ceramics are correlated with several factors including suspension characteristics, e.g., solid loading, as well as heat treatment conditions, e.g., debinding thermal profile and sintering temperature and time. Herein, we strive to develop a highly loaded suspension of KNN nanoparticles with suitable rheological and optical properties for DLP-based AM technology. In addition, the rheology and stability of the suspensions with different solid loading and dispersant concentration are examined through viscosity and sedimentation testing. Also, the microstructure, density, as well as the piezoelectric performance of KNN piezoceramics made of suspensions with different solid loadings are investigated.

Oral Presentations - Material extrusion with ceramic filaments

Extrusion based Additive Manufacturing of ceramics: Current works about CerAM FFF at IKTS

Thursday, 10th November - 09:05: C - MATERIAL EXTRUSION WITH CERAMIC FILAMENTS - Oral

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Fused Filament Fabrication (FFF) is considered as an Additive Manufacturing (AM) process that can process not only pure thermoplastics but also particle-filled polymers into three-dimensional components via the powder metallurgy route currently. In particular, low acquisition costs of the equipment technology represent the opportunity for many companies to enter the field of AM of metal or even ceramic components. An increasing variety of filaments favors the accessibility to a wide range of different markets. The possibility to process all sinterable materials and even to combine them with each other is a special feature for additive processes. However, it is well known that the surface roughness caused by the layer buildup is often considered too high, which means that this technology is often used for prototyping only. By green machining the components, the surface quality can be significantly improved before sintering and fine structure sizes can be achieved. This tool offers great potential for improvement in the future. The presentation will give an overview of recent work in the field of FFF at Fraunhofer IKTS. Current materials as well as components will be presented and approaches to improve the surface roughness as well as to reduce distortion during heat treatment will be presented. Furthermore, examples will be shown how AM processes can be hybridized or individual parts can be joined to assemblies without using solders.

Additive manufacturing and ultra-fast high-temperature sintering (UHS) of zirconia ceramics

Thursday, 10th November - 09:25: C - MATERIAL EXTRUSION WITH CERAMIC FILAMENTS - Oral

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Doped zirconia is one of the most exploited high-performance ceramic materials owing to its intriguing mechanical, electrical and thermal properties. In the past decade, ultrafast sintering techniques have gained popularity as a way to reduce the processing time and energy consumption of the ceramic industry. However, most of the research study has focused on the field-assisted sintering of uniaxially or isostatically pressed powder compacts with simple shapes. Additive manufacturing (AM) allows fabricating complex geometries with fewer constraints compared to the conventional fabrication techniques such as HIP, extrusion, injection molding etc. Coupling AM with field-assisted sintering would be ideal to reduce production time from days to hours.

In this work, 3D printed cylindrical structures of 3 mol% yttria-stabilized zirconia (3YSZ) with gyroidal infill pattern were fabricated using the fused filament fabrication process (FFF). The used filaments were commercially produced from Nanoe. This research study examines the use of ultra-fast high-temperature sintering (UHS) to densify the complex 3D structure. A systematic study was done on the sintering behavior of the as-printed, chemically debinded and pre-sintered sample. Furthermore, current-controlled experiments were performed to study the densification behavior, extent of phase transformation and grain size. With optimized parameters, the 3D printed, chemically debinded samples could be fully densified in 60 to 120 seconds. There were no visible cracks on the surface of the samples. The hardness of the UHS-ed samples was comparable to that of conventionally sintered components. The work provides a first proof of concept on utilizing the ultrafast heating rates to sinter such complex geometries. Producing high-quality parts with complex geometries and adequate properties within hours using UHS approach would be quite appealing, also from an industrialization and prototyping perspective.

Preparation of Cu-based Colloidal Feedstocks for Material Extrusion with Filaments

Thursday, 10th November - 09:45: C - MATERIAL EXTRUSION WITH CERAMIC FILAMENTS - Oral

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The Additive manufacturing (AM) plays an important role in modern ceramic industry, and this role expands with each improvement in technology. With fused filament fabrication (FFF), it is possible to produce large and complex components quickly with high material efficiency.

The improvement in properties and the discovery of new functionalities are key goals that cannot be reached without a better understanding of the preparation, characterization, and manufacturing of novel functional formulations that offer excellent properties.

The availability of certain customized feedstocks for specific applications is still limited or nonexistent, which greatly impedes the production of desired structural or functional materials.

The combination of AM techniques with colloidal processing offers more freedom in terms of design while maintaining the necessary thermal and chemical properties. Colloids, with their stable biphasic nature, have tremendous potential to satisfy the requirements of various 3D printing methods owing to their tunable electrical, optical, mechanical, and rheological properties. This enables materials delivery and assembly across the multiple length scales required for multifunctionality.

In this work we propose the formulation of novel Cu-based colloidal feedstocks which will be used to produce functional filaments for material extrusion.

Firstly, a composition was successfully prepared from micronic Cu particles. After extrusion and 3D printing, the samples were sintered and calcined to transform Cu powder to CuO semiconductor, which displayed an advance structure with a mechanical characteristic like a scaffold ceramic.

The preparation of functional composites of PLA with incorporated CuO nanoparticles was also considered to perform specimens manufactured by fused filament fabrication (FFF) that are aimed to produce multi-purpose geometrically complex nanocomposite materials that could be employed in medical, food, and other sectors.

Colloidal feedstock with a high content of graphite particles for self-supporting 3D oriented conductive electrodes by FFF

Thursday, 10th November - 10:05: C - MATERIAL EXTRUSION WITH CERAMIC FILAMENTS - Oral

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1. *Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain,* ***2.*** *Instituto de Cerámica y Vidrio, CSIC*

Carbonaceous species have become an interesting issue in the energy storage field thanks to their electronic properties, low cost and sustainable origin. In this sense, the processing of graphite based conductive colloidal feedstock for Fused Filament Fabrication (FFF) requires physical union between particles hence the loading and, therefore, the dispersion of the electroactive inorganic particles in the polymer matrix becomes essential.The colloidal processing allows, following a more efficient and effective route than the classic hot mix routes, to maintain physical contact between the particles in the polymeric matrix through a high dispersion of the materials. This work presents the fabrication of conductive electrodes, with an oriented inorganic microstructure, by the formulation of conductive filaments with a high content of commercial Graphite and Graphene. Through the colloidal processing, well dispersed high inorganic loadings are obtained (15-30vol%) provoking the orientation of carbon nanoflakes during the extrusion process. The FFF approach enables a major control of the architecture and morphology of the electrode requiring less material. Therefore, rheological analysis of the melted filaments was studied, limiting the printing window in order to obtain highly defined different architectures and porosities. However, the developed colloidal filaments, with an oriented inorganic microstructure, not only allow a defined shaping of the outer structure of the electrode, but they also let the design of the completely different microstructures. Thus, by the improvement of the stacking of the conductive nanoflakes, the π unions remain intact keeping the mandatory electronic network that provides an electrical conductivity up to 22 S/m to the electrodes.

TiO2-based porous photocatalytic membranes by FFF from a colloidal feedstock

Thursday, 10th November - 10:25: C - MATERIAL EXTRUSION WITH CERAMIC FILAMENTS - Oral

Mr. Pablo Ortega Columbrans [1], Dr. Begoña Ferrari [1], Dr. Javier Sanchez-Herencia [1]

***1.** Instituto de Cerámica y Vidrio, CSIC*

In 2015, the United Nations General Assembly adopted the 2030 Agenda for Sustainable Development as a roadmap for international cooperation on sustainable development, with its economic, social, environmental and governance dimensions. Today, geopolitical developments make it essential to redouble efforts and address challenges for the rapid prototyping of advanced, environmentally friendly, low-cost and highly efficient photochemical devices.

One of the most promising technologies for water treatment today is photocatalytic membrane reactors (PMRs). They are considered a mature methodology for the immobilisation of semiconductors in photocatalytic processes, while their engineering is still unresolved. The challenge is related to the fabrication of photoactive membranes adapted to PMR configurations. In this sense, the project focuses on the creation of prototypes of photoactive membranes for their implementation in PMRs by means of fused filament fabrication (FFF), and the validation and demonstration of the additive manufacturing (AM) technology in a relevant environment.

In this regard, for water decontamination based on ceramic semiconductor (commercial TiO_2) we proposed a colloidal method to self-support nanoparticles in a polymeric matrix of polylactic acid (PLA). The fabrication of a photocatalysts material of thermoplastic nature made the composite suitable for processing by additive manufacturing techniques. The first step is surface modification using a polyelectrolyte to ensure homogeneous dispersion of the particles in the new medium. Then, after wet mixing of the matrix-particle system heat extrusion are used to process the final parts. Catalytic activity is evaluated as a function of the degradation of a chemical indicator over time. The results indicate that the geometries printed by FFF achieve degradation rates for methyl orange of 100% after an exposure time of 2h, reaching 50% degradation after one hour of testing.

Oral Presentations - Material extrusion with ceramic inks and pastes

Microstructural and Mechanical Properties of Translucent Zirconia Parts Produced by Direct Ink Writing

Friday, 11th November - 09:05: F - MATERIAL EXTRUSION WITH INKS AND PASTES - Oral

Ms. Mona Yarahmadi [1]***, Ms. Junhui Zhang*** [1]***, Mr. Marc Serra Fanals*** [1]***, Dr. Joan Josep Roa Rovira*** [1]***, Prof. Luis Miguel Llanes Pitarch*** [1]***, Dr. GEMMA FARGAS*** [1]

1. *Universitat Politècnica de Catalunya*

Translucent zirconia is the newest preference of zirconia-based ceramics which aimed to renewal the non-transparent Yttria-stabilized Tetragonal Zirconia Polycrystal (Y-TZP) in more demanding esthetic cases. As a result of advancements in 3D fabrication technology and increased demands of translucent zirconia, efforts made to achieve full-density 3D zirconia parts via Direct Ink Writing. In this work, partially-stabilized zirconia powder with 3 mol% Yttria (TZ-3YB) and high translucent zirconia grades: Zpex, Zpex-4 and Zpex-smile with 3, 4, 5 mol% Yttria respectively, were printed using an inverse-thermoresponsive hydrogel, Pluronic®F-127. Sintering was performed at 1450 ºC for two hours. Higher densities of printed samples were achieved at 73% of solid loading for TZ-3YB and 71% for high translucent zirconia grades. Sinterized speicmens displayed similar shrinkage which was higher for Z direction, 27±2%. The amount of yttria leaded to an increase of cubic phase: from 21±3% to 58±2% for TZ-3YB to Zpex-Smile, respectively. Nanoindentation tests pointed out that there is no significant differences regarding elastic modulus while hardness increased with increasing percentage of cubic phase: from 15.06±1.74 GPa for TZ-3YB to 16.92±1.65 GPa for Zpex-Smile. Fracture toughness was determined by means of indetation fracture. In this sense, grain size of printed samples plays an important role. Thus, TZ-3YB and Zpex with small grain size, around 0.28±0.1μm, exhibited higher fracture toughness, 5.4±1.45 MPa√m, than Zpex-4 and Zpex-Smile, with 0.65±0.2μm and 0.75±0.45μm respectively, where values decrease to 4.15±1.8 and 3.78±1.15 MPa√m.

Keywords: Translucent zirconia, Direct ink writing, Mechanical properties, Nanohardness, Fracture toughness

Robocasting of bioceramic scaffolds with different hollow strut geometries

Friday, 11th November - 09:25: F - MATERIAL EXTRUSION WITH INKS AND PASTES - Oral

Ms. Shumin Pang [1], Mr. Dongwei Wu [2], Mr. Franz Kamutzki [1], Prof. Jens Kurreck [2], Prof. Aleksander Gurlo [1], Dr. Dorian Hanaor [1]

__1.__ Technische Universität Berlin, Chair of Advanced Ceramic Materials, __2.__ Technische Universität Berlin, Chair of Applied Biochemistry

Additively manufactured hollow-strut bioceramic scaffolds present an attractive strategy for enhanced performance in patient-tailored bone tissue engineering (BTE). The channels in such scaffolds offer nutrient and cell transport pathways and facilitate effective osseointegration and vascularization [1, 2]. For extrusion-based 3D printing technologies, nearly all systems use a round-mouth nozzle geometry, from which the materials are extruded to be initially round filaments.

In this work, we describe the design and production of novel nozzles with various geometries via vat photopolymerization 3D printing. The novel extrusion geometries enable the fabrication of scaffolds with hollow struts of diverse cross-sectional geometries.

We synthesized copper substituted diopside, a promising material for BTE scaffolds combining good fracture toughness and bioactivity [3]. An optimized paste with favorable rheological behaviors was developed by employing sodium alginate and Pluronic F-127 as additives. Finally, scaffolds with hollow members of varied outer and inner geometries were printed by robocasting through specially designed extrusion nozzles, and their utility for bone repair was explored in terms of mechanical performance and bioactivity.

Surprisingly, mechanical testing and finite element analysis reveal that hollow-strut scaffolds with square cross-sections showed the best performances, exceeding those with round cross-sections or others. The hollow structure of these bioceramic scaffolds significantly improved cell attachment and proliferation. Moreover, hollow members can be infiltrated with a second phase, in a subsequent step or during printing, to produce high-performance biomimetic composites and self-healing materials.

Taken together, this work provides an easily controlled method for 3D printing of hollow-strut scaffolds with various cross-sections, which is a reference for the development of diversity and mechanical enhancement of printed objects in extrusion-based 3D printing.

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[2] C. Feng et al., ACS Biomater. Sci. Eng. 7(3) (2020) 872-880.

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Additive Manufacturing of Porous Ceramic Bodies by Extrusion of Capillary Suspensions

Friday, 11th November - 09:45: F - MATERIAL EXTRUSION WITH INKS AND PASTES - Oral

***Mr. Felipe Mello Rigon* [1] *, Dr. Norbert Willenbacher* [1]**

***1.** Karlsruher Institute für Technologie*

By employing direct ink writing (DIW) in combination with the capillary suspension concept, we are able to 3D print complex geometries with high open porosity > 60%. Capillary suspensions are ternary liquid-liquid-solid systems, in which the addition of a secondary fluid, immiscible to the main phase, changes the flow properties of the suspension drastically. This is due to the formation of a sample spanning particle network controlled by strong attractive capillary forces coming with the addition of the second immiscible liquid. This network structure reduces shrinkage and suppresses crack formation, making capillary suspensions ideal precursors for manufacturing of highly porous bodies, with the possibility to tune porosity and pore size, simply by varying particle size and volume fraction. Capillary suspensions display a high yield stress and strong shear thinning behavior, which makes them good candidates for extrusion-based 3D printing processes. The former guarantees a superior shape accuracy and enables large ratios of unsupported length over filament diameter ratios (»10) and printed feature sizes below 0.1 mm. In this study, we will present 3D printed structures to be used as filters, with geometries not achievable by traditional fabrication processes. Porosity and pore size will be analyzed and their influence on mechanical strength will also be discussed.

3D-printed biomimetic hydroxyapatite scaffolds with triply periodic minimal surface geometries: mechanical and biological properties

Friday, 11th November - 10:05: F - MATERIAL EXTRUSION WITH INKS AND PASTES - Oral

***Ms. Laura del Mazo Barbara** [1], **Prof. Morteza Aramesh** [2], **Prof. Cecilia Persson** [2], **Prof. Maria Pau Ginebra** [1]*

***1.** Biomaterials, Biomechanics and Tissue Engineering Group (BBT), Department of Materials Science and Engineering, Universitat Politècnica de Catalunya, Barcelona, Spain, **2.** Department of Materials Science and Engineering, Division of Biomedical Engineering, Uppsala University, 751 21 Uppsala, Sweden*

Direct Ink Writing (DIW) allows printing biomimetic hydroxyapatite scaffolds for bone regeneration with a precise control of both external shape and internal porosity. Triply periodic minimal surfaces (TPMS), minimal surfaces that are invariant under a rank-3 lattice of translations, have attracted great interest as nature-inspired models. Their limited surface curvature, similar to the trabecular structure, results in interconnectivities and concave pores that are favourable for cell growth, migration, and vascularization[1]. This study explores the feasibility of printing three types of TMPS (Gyroid, Diamond and Schwarz) through DIW of calcium phosphate inks and studies their mechanical and biological properties compared to a typical orthogonal-pattern.

Cylindrical TPMS models were created through Matlab codes and the ink was prepared by loading a 30wt.% Pluronic hydrogel with 70wt. % of α-tricalcium phosphate fine powder. After printing, the scaffolds were hardened by immersing them in water at 37ºC. After optimising the printing pattern, in terms of unit cells and porosity, the printed geometries were analysed by microcomputed tomography and tested mechanically by uniaxial compression. The effect on the blood permeability and the proliferation and metabolism of preosteoblasts cells were evaluated. Orthogonal-patterned scaffolds with the same porosity were used as control.

The Gyroid and Diamond geometries were successfully printed with a 20% porosity, resulting in concave, interconnected and relatively large pores compared to the convex and small pores from the control. Under compressive forces, the Gyroid exhibited a compaction effect, instead of the abrupt fracture observed in the orthogonal scaffolds, although the strength was reduced. Pore architecture had a strong effect on permeability, with TPMS structures, that had larger interconnectivities, showing a ten-fold increase compared to the control. Consequently, preosteoblasts proliferation and metabolism were affected. Therefore, Gyroid structure could be a biological properties enhancer, while keeping interesting mechanical properties.

1. H. Montazerian, *Acta Biomaterialia*, 96, 2019

Modelling the influence of the rod conductivity and the printing parameters on the effective thermal conductivity of 3D printed macro-porous structures

Friday, 11th November - 10:25: F - MATERIAL EXTRUSION WITH INKS AND PASTES - Oral

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The present work addresses the problem associated with the measurement of thermal transport properties of 3D porous macro-lattices developed by extrusion-based additive manufacturing (AM) technologies, which is a fundamental concern for their application in fields such as energy conversion and storage, catalysis or thermal management. The finite element method (FEM) is used to numerically analyze the most important parameters affecting thermal conductivity measurement by the transient plane source (TPS) technique. This technique stands out because allows determining the thermal conductivity, thermal diffusivity, and heat capacity in just one test, being suitable for a wide range of materials including highly porous 3D structures. The main concerns when evaluating thermal properties of 3D printed macro-lattices by TPS tackled by the developed FEM model are the interfacial thermal resistances associated with the imperfect contact between the testing probe and the rough scaffold surface, the gas presence inside the pores, and the anisotropy induced by the printing process, which complicate the straightforward design of additively manufactured components. In addition, the thermal characteristics of these lattices depend not only on the thermal properties of the strut material but highly on the geometrical parameters defining the structure like the shape, size, and the spatial distribution of the macro-pores. FEM modelling has also been performed for analyzing the effect of these parameters being a powerful tool to predict, tailor, and optimize the geometry and final properties of the 3D printed structures before the manufacturing stage, hence reducing the experimental workload. The FEM model has been experimentally validated in 3D printed structures of diverse ceramic materials.

Oral Presentations - Powder bed fusion

Influence of humidity thermal and ageing on the flow and packing behaviour of powders for additive manufacturing

Thursday, 10th November - 16:40: E - POWDER BED FUSION - Oral

Mr. Rafael Kleba-Ehrhardt [1], Mr. Leonell Juliano [1], Mrs. Anzhelika Gordei [2], Dr. Chrsitoph Heinze [3], Prof. Aleksander Gurlo [1], Dr. David Karl [1]

1. *Technische Universitaet Berlin, Chair of Advanced Ceramic Materials,* ***2.*** *Fraunhofer Institute for Production Systems and Design Technology,* ***3.*** *Siemens Energy Global GmBH*

Powder-based additive manufacturing (AM), such as laser powder bed fusion (L-PBF) or binder jetting (BJ), have high demands on the feedstock properties. They largely determine the process reliability and reproducibility of AM processes. While established measurement methods exist for the physical properties of powder particles (such as density, size and shape), there is currently no scientific consensus on characterizing the behaviour of powders for and in AM applications. [1] In particular, there are currently few detailed studies comparing the many different flowability measurement methods and discussing their specific suitability for AM. [2] In this work, five AM powder materials (metallic and ceramic), in the as-received condition, as well as modified powders, were investigated using a wide range of advanced powder flow characterization techniques.

An important consideration for carrying out such characterization methods are the humidity conditions of the lab environment in which the measurement takes place. To calibrate the equipment and learn about humidity influence, a detailed study on the influence of humidity on the flowability of powders was carried out. Furthermore, to learn about the recyclability of powders, the influence of thermal aging in L-PBF alloys on the powder properties was investigated. In detail for the powders, two nickel alloys, one iron alloy, one titanium aluminide and one tungsten carbide the most common flowability measurements, such as the measurement of flow time, Hausner-factor, angle of repose, bulk density, powder-rheology, avalanche angle and the ring shear cell test were carried out.

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Fabrication of dense SiC ceramics by a novel hybrid additive manufacturing process

Thursday, 10th November - 17:00: E - POWDER BED FUSION - Oral

Mr. Marco Pelanconi [1], Mr. Samuele Bottacin [1], Mr. Giovanni Bianchi [1], Prof. Alberto Ortona [1]

***1.** SUPSI*

The fabrication of Silicon Carbide (SiC) components by a novel hybrid additive manufacturing process was investigated. Selective laser sintering of polyamide powders was used to 3D print a polymeric preform with controlled relative density, which allows manufacturing geometrically complex parts with small features, such as cellular architectures. Preceramic polymer infiltration with a silicon carbide precursor followed by pyrolysis (PIP) was used to convert the preform into an amorphous SiC ceramic, and six PIP cycles were performed to increase the relative density of the part. The final densification was achieved via liquid silicon infiltration (LSI) at 1500°C, obtaining a SiSiC ceramic component without change of size and shape distortion. The crystallization of the previously generated SiC phase, with associated volume change, allowed to fully infiltrate the part leading to an almost fully dense material consisting of β-SiC and Si. The advantage of this approach is the possibility of manufacturing SiSiC ceramics directly from the preceramic precursor, without the need of adding ceramic powder to the infiltrating solution. This can be seen as an alternative AM approach to Binder Jetting and Direct Ink Writing for the production of templates to be further processed by silicon infiltration.

Property Tailoring using Powder Bed Fusion Studied by Multiphysics and Multiscale Phase-field Modeling

Thursday, 10th November - 17:20: E - POWDER BED FUSION - Oral

Mr. Yangyiwei Yang [1]***, Dr. Xiandong Zhou*** [1]***, Prof. Bai-Xiang Xu*** [1]

1. *TU Darmstadt*

As one of the most popular additive manufacturing techniques for inorganic materials, powder bed fusion (PBF) of alloy materials has shown industries flexibility and rapidness in manufacturing novel and complex geometries. Modeling and simulation of the PBF aim to complement the current time and cost-expensive trial-and-error principle with an efficient computational design tool. Nevertheless, it remains a great challenge due to the sophisticated and interactive nature of underlying physics, which covers a broad range of time and length scales and strongly depends on the processing parameters (incl. beam size and power, as well as scan speed). A unified modeling scenario considering scale effects as well as multiphysics coupling is thereby essential for reliable microstructure prediction.

In this work, we develop a non-isothermal phase-field modeling scenario coupled with multiphysics, such as fluid dynamics, heat transfer, phase transition, and magnetism, to recapitulate interesting phenomena from multiple time/length scales and reveal their interactions during the PBF, which are not accessible to the conventional isothermal model. Models are derived in a thermodynamically consistent way according to our latest work and numerically implemented by the finite element method (FEM). We further perform the parameter investigations of the PBF procedure. The influences of the processing parameters on the properties, incl., thermal, mechanical, and magnetic, are also discussed based on property homogenization.

Oral Presentations - Directed energy deposition

Oral Presentations - Emerging, hybrid and multimaterial AM technologies

Hybridization of materials and shaping technologies for the manufacturing of ceramic components with unprecedented combinations of properties

Friday, 11th November - 11:20: G - EMERGING, HYBRID AND MULTIMATERIAL - Oral

Mr. Uwe Scheithauer [1], Mr. Eric Schwarzer-Fischer [1], Mr. Johannes Abel [1], Mr. Steven Weingarten [1], Mrs. Hanna Elflein [1], Mrs. Stefanie Bode [1], Dr. Lars Rebenklau [1], Dr. Henry Barth [1], Mrs. Nadine Lorenz [1], Mrs. Lisa Gottlieb [1], Mrs. Maria Reichel [1]

1. *Fraunhofer Institute for Ceramic Technologies and Systems IKTS, 01277 Dresden, Germany*

The advent of additive manufacturing of ceramics (CerAM) has enabled unprecedented geometrical complexity of products: a “game changing” manufacturing technology in the processing of ceramics. As a result, complex ceramic components can be realized cost-effectively, with post-processing reduced or even completely unnecessary. Additively manufactured ceramics are now for the first time genuine alternatives to polymer and metal components, especially for applications in harsh conditions.

The potential of such CerAM components can be further increased by integrating additional functionalities, like incorporated electrically conductive 3D-tailored networks into the component.

The presentation gives an overview of the current development trends in the fields of material and process hybridisation, resulting in ceramic components with unprecedented combinations of properties. Using various application examples, e.g. from the aerospace, chemical and process engineering sectors, different manufacturing strategies (sequential and simultaneous manufacturing of multi-material or multi-functional components; hybridization of CerAM FFF and CerAM VPP to realize high-resoluted geometries in large components) as well as their possibilities and limitations will be presented and discussed.

Manufacturing Mixed Proton-Electron Conducting Ceramic Electrodes: 3D-Printing and Laser Post-processing

Friday, 11th November - 11:40: G - EMERGING, HYBRID AND MULTIMATERIAL - Oral

Ms. Joanna Pośpiech [1]***, Prof. Aleksandra Mielewczyk-Gryń*** [1]***, Prof. Maria Gazda*** [1]***, Dr. Sebastian Wachowski*** [1]

1. *Institute of Nanotechnology and Materials Engineering, Gdańsk University of Technology*

Additive manufacturing processes, such as 3D printing technologies, allows to fabricate materials with designed geometries and microstructure. Short prototyping time and low production costs make 3D printing appealing for industrial applications. Therefore, 3D printing attracts attention in the energy conversion sector due to the possibility of further advance development and commercialization of proton ceramic electrochemical cells. Additive manufacturing of protonic conductive fuel cell elements is quite a novel approach.

In our study, we present recent developments of 3D printing of the $Ba_{0.5}La_{0.5}Co_{1-x}Fe_xO_{3-\delta}$ ceramics as positrodes for protonic ceramic fuel cells. The main focus of this work was to integrate the micro-extrusion process with precise IR laser post-processing of deposited layers. The printer uses an extrusion-based printing head developed for Direct Ink Writing technology. The prepared printable inks consisted of ceramic powders dispersed in a polyvinyl alcohol (PVA) matrix. The PVA was later removed in the post-processing stage. Inks were deposited on the dense BZCY442 electrolyte to examine the interface adhesion of the future proton conducting fuel cell elements. The post-processing was performed by scanning the surface of the printout point-by-point with IR laser. This process allows the burnout of the organics with simultaneous sintering of the ceramic component. As a result, depending on the process parameters such as laser power, scanning speed, or laser working frequency, various microstructures were obtained from dense ceramics, through porous backbones with various pore sizes, to ceramic foams composed of large pores with the size of hundreds of micrometers with a few micron-thick walls. Various compositions and conditions were tested and the most appropriate parameters of the laser post-processing were chosen.

Acknowledgements

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Hybrid additive manufacturing for the fabrication of freeform transparent silica glass components

Friday, 11th November - 12:00: G - EMERGING, HYBRID AND MULTIMATERIAL - Oral

Mrs. Anna De Marzi [1], Dr. Giulio Giometti [2], Mr. Hannes Erler [3], Prof. Paolo Colombo [1], Dr. Giorgia Franchin [1]

***1.** Industrial Engineering Department, University of Padova,* ***2.** Isoclima Spa,* ***3.** Swarovski KG*

Additive manufacturing of ceramic and glass materials has been developed extensively over the past years, revealing strengths and limitations of the various technologies. Here, we present a hybrid extrusion-photopolymerization process developed to exploit the benefits of both approaches. Their combination allows for the fabrication of complex geometries with less rheological constraints and no influence of scattering and/or absorption. We demonstrated the capabilities of this method by fabricated transparent silica glass 3D honeycomb-like structures starting from a suspension that combines silica particles and a silicon alkoxide. We tailored the rheological behaviour and reactivity of the ink with an appropriate blend of acrylates as well as with the alkoxide addition. The latter also contributes to the final ceramic yield, allowing for faster thermal treatments despite a relatively low particle loading. The final components closely match the shape provided by the CAD model; their physical properties confirm the formation of a fully dense silica glass after the heat treatment. The setup can be mounted on a 6-axes robot arm, thus enabling additional degrees of freedom and control on the extrusion path.

Powder modifications and formulations for additive ceramic manufacturing via photonic sintering

Friday, 11th November - 12:20: G - EMERGING, HYBRID AND MULTIMATERIAL - Oral

***Dr. Alejandro Monton* [1], *Dr. Nicolas Somers* [1], *Prof. Mark Losego* [1]**

1. Georgia Institute of Technology

Additive Manufacturing (AM) of ceramics is a timely topic because it allows to produce functional ceramic parts with geometrical complexity, very useful in fields including electronics, medicine, aerospace, and sustainable energy. Nevertheless, the main limitation of most ceramic AM processes is the need of a thermal post treatment to remove organic materials as well as obtaining compact ceramic objects. This post treatment can be time consuming and generate cracks and deformations in the ceramic parts. This research project aims to study new powder modifications (coatings/grafting) and additives that could allow a single step AM of ceramic parts. Inorganic, preceramic polymers as well as sol gel precursors are investigated as potential additives to reach this objective. These new additives and modifications are tested by using different ceramic materials (alumina, zirconia, and silica) to enable single step AM of ceramic components at low substrate temperatures. Flash Lamp Annealing (FLA), an emerging technology which employs ultrashort (0.1-10 ms) pulses of light, is used to induce rapid transient heating and sintering of thin layers of ceramic materials. In addition to FLA, infrared heating is also studied to induce liquid phase sintering of ceramic thin layers with the use of light absorbent additives. These two light technologies are combined with AM processes, such as Direct Ink Writing/Robocasting and Aerosol Ink Printing, to deposit and treat very thin layers of ceramic suspensions in order to allow a single-step, low-temperature AM process for ceramic manufacturing without the need for post-process pyrolysis.

Two decades of Ceramic Additive Manufacturing: a bibliometric analysis

Friday, 11th November - 12:40: Closing - Oral

***Mrs. Esther Galindo Batanero* [1]*, Prof. Ana Inés Fernández Renna* [1]*, Prof. Jaume Valls Pasola* [1]*, Dr. Elena Xuriguera* [1]**

***1.** Universitat de Barcelona*

Nowadays, Additive Manufacturing (AM) is one of the most promising area of research in terms of manufacturing of three-dimensional objects. AM comprises a broad range of different techniques to build 3D objects layer-upon-layer, allowing to design and rapidly fabricate highly complex shapes with very few geometric limitations compared to traditional manufacturing processes.

Bibliometric analysis is a quantitative approach to identify, analyse and evaluate published research. Bibliometric is the science with the capability to present a precise, systematic and reproducible review process and thus improve the quality of reviews, leading the researcher to the most important studies and mapping the research field.

The *Web of Science Core Collection* database was used to search the most relevant scientific articles related to Ceramic Additive Manufacturing. The following main keywords were used for the search: "Additive Manufacturing" OR "3D printing" OR "rapid prototyping" and some ceramic keywords to delimited like: ceramic, alumina, glass, cement, zirconia and hydroxyapatite among many others. Only articles and reviews were considered in this analysis.

This study presents an overview of the history and the tendency of Ceramic AM research between 2001 and 2020. The results show that ceramic AM has been gaining a positive exponential growth in the last decades, especially since 2014, this trend is accordance with the expired patents between the years 2013 and 2015. Another relevant result comes from the analysis of ceramic AM research by country, showing China and United States of America (USA) as the main contributors to the knowledge development; nevertheless, when considering European Union as a whole, it accounts for a higher contribution than China or the USA. This reflects the strong efforts produced from the European initiatives towards AM technologies. Analysing the authors, P. Colombo is the most productive author publishing on the ceramic AM research topic, followed by D. Li.

Poster session I

Promoting the Bonding of Polyether-ether-ketone with Soft and Hard tissue by Additive Manufactured Scaffolds with Hydroxyapatite Filler

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Dr. Changning Sun [1]***, Prof. Kai Miao*** [2]

1. Xi'an Jiaotong University, 2. Xi'an

Additive manufactured (AM) Poly-ether-ether-ketone (PEEK) orthopaedic implants offer new opportunities for bone substitutes. However, the integration between Poly-ether-ether-ketone (PEEK) with hard and soft tissue represents a major challenge of PEEK orthopaedic implants owing to its chemical inertness. Here we investigated the influence of hydroxyapatite (HA) fillers and pore size of additive manufactured (AM) HA/PEEK composites scaffolds on the integration with bone and soft tissues through cellular experiments and animal experiments. The composite scaffolds were manufactured by fused filament fabrication. The cell experiment showed that the adhesion, proliferation, osteogenic differentiation and mineralization ability of mesenchymal stem cells (BMSCs) as well as the proliferation and adhesion of myofibroblasts on the PEEK/HA scaffolds were significantly improved. Ca2+ released by HA was proved to be the key in promoting cell behaviour on the surface of PEEK-based composites. The in-vivo experiment demonstrated significantly higher bone ingrowth and tighter adhesion of soft tissue in the HA/PEEK scaffolds. The shear bonding strength between bone tissue and the scaffolds showed that PEEK/HA composites scaffolds exhibited higher bonding strength than that of the pure PEEK scaffold. The maximum push-out force between the scaffold and bone tissue was 415.8 ± 43.9 N, exceeding the bonding force of the titanium alloy porous scaffolds with bone tissue. The macroscopic bonding force between soft tissue and scaffolds was dominated by the pore size of the scaffolds but was hardly affected by the HA content. The maximum bonding force of soft tissue was 5.61 ± 2.55 N, which was higher than that between natural bone and soft tissue of rabbits. The present study provides engineering-accessible design principles on material components and geometry of AM PEEK-based composites orthopaedic implants for improving the integration with the bone and soft tissue.

Dimensional accuracy, surface finish and porosity of 3D Printed Calcium Carbonate-filled polypropylene parts for medical applications

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Dr. Irene Buj Corral [1], Mr. Arnau Valls [2], Mrs. Núria Adell [2], Dr. Miquel Domingo [3]

***1.** Universitat Politècnica de Catalunya, **2.** Hospital Sant Joan de Déu, **3.** Eurecat*

3D printed replicas of bones are currently being used in some hospitals for surgical planning and clinical training, allowing the surgeons to simulate and prepare the procedure before the surgery. Fused Filament fabrication (FFF) technique allows obtaining models that mimic the properties of bones in an inexpensive and easy way, if low-cost machines are employed. However, these models are limited in their ability to mimic the mechanical behavior of bones in milling or drilling processes.

In the present work a new composite material was developed, consisting of calcium carbonate filled-polypropylene (PP) filament.

Tensile samples Type IA were printed according to ISO 527-2 standard to perform the study.

The samples were subjected to three different sterilization methods available at SJD Barcelona Children's Hospital: 134 Autoclave, 121 Autoclave and Peroxide HPO. 20 tensile samples were 3D printed: 5 copies for each sterilization method and 5 control specimens.

After this operation, the samples were characterized regarding dimensional accuracy, surface finish and porosity. In addition, functional tests were performed to the samples mimicking the operations performed during surgery such as milling or drilling.

The results showed that the sterilization processes did not significantly affect the performance of the samples, the dimensional accuracy, surface finish and porosity, showing that the proposed material could be used for 3D printed models of the bone and included in the operating rooms of the hospitals. In addition, it showed excellent performance in the machining tests. In future works, tensile strength tests will be performed to the samples.

Production of complex BCZY-GDC supports by 3D micro-extrusion

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

***Dr. Alex Sangiorgi* [1], *Mr. Andrea Bartoletti* [1], *Dr. Angela Gondolini* [1], *Dr. Elisa Mercadelli* [1], *Dr. Alessandra Sanson* [1]**

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Ceramic membranes operating at high temperatures are a key technology for hydrogen separation processes including advanced chemical reactors, power generation, CO_2 capture and hydrogen separation/purification from gas mixtures. Thanks to their 100% selectivity, high proton-electron conductivity, intrinsic lower costs if compared with Pt-based technologies and chemical and temperature stability, ceramic composites based on $BaCe_{0.65}Zr_{0.20}Y_{0.15}O_{3-\delta}$-$Gd_{0.2}Ce_{0.8}O_2$ (BCZY-GDC) have gained increasing attention as asymmetric membranes for H_2 purification. Nowadays, however, the hydrogen permeation fluxes obtained employing this technology are still not suitable for industrial applications. Asymmetric architectures are an effective way to improve both gas permeation and mechanical strength of the membrane, due to the combination between a dense and a porous ceramic layer that are commonly composed of the same materials to avoid thermal expansion mismatch between themes.

In order to improve the H_2 permeation, a lot of research is currently focused on optimizing the microstructure of the porous support aiming to increase the gas access and transport through it. In this work, complex-shaped BCZY-GDC composite supports were successfully fabricated for the first time by 3D micro-extrusion. Honeycomb-type geometries containing different size cavities were designed and produced to promote the support fluidics without affecting its mechanical stability. Different high solid loading water-based BCZY-GDC pastes were formulated finely tuning their viscosity. The latter was then micro-extruded in multilayer structures without nozzle clogging or other process issues. Aiming to obtain cracks-free green bodies, different drying process methods were considered and deeply investigated. Finally, the process optimization allowed the production of engineered porosity BCZY-GDC supports potentially employable in asymmetric membranes.

Investigation of additive manufacturing for electrical isolator via Ultraviolet-assisted low viscous Mica/silicone paste extrusion

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Mr. Minkyu Kang [1], Dr. Mathias Czasny [1], Mr. Till, Heinrich Emanuel Butzmann [1], Dr. Xifan Wang [1], Mr. Nishant Jain [1], Mr. Delf Kober [1], Prof. Aleksander Gurlo [1]

1. *Fachgebiet Keramische Werkstoffe / Chair of Advanced Ceramic Materials, Technische Universität Berlin, Berlin*

High voltage electrical devices like generators and motors are subject to extreme environments featuring high humidity, heat and dust pollution - conditions that can lead to electrical discharges, which damage the equipment through electric leakage and fires. In order to prevent this, it is essential to separate the electrical input and output signals to inhibit short circuit damages in parts of the devices with a high voltage isolation system. In this study, ceramic/polymer paste was printed to apply as an isolator for high voltage applications. The prepared printing models are cylindrical shells, square cubes and polyhedrons. The fabricated paste was degassed to remove air bubbles before printing to avoid defects. This work focuses on stacking layers of low viscous ceramic paste. The extrusion was controlled by an auger screw in the print head. The mechanical and high voltage electrical isolator properties were analyzed. The paste printing was assisted by ultraviolet (UV)-light exposure (λ= 380 nm – 390 nm) to cure the system and prevent issues with surface tension and gravity of the pastes. In addition, the wettability of the printing substrates was measured to determine the wettability of the stacking layer. The silicone resin was mixed with a UV curable agent and defined volume ratios of the mica reinforcement particles. Furthermore, 3d printed samples were compared to mold-cast samples having the same thickness. Analysis with FT-IR, TG/DTA, rheometer, optical microscope, SEM, and tensile tests were performed, changing mica content and curing conditions (UV/heat). In conclusion, this study shows the influence of material and processing parameters of mica reinforced silicone composite as a high voltage electrical isolator.

Optimizing Direct Ink Writing Parameters for High-Density Monolithic Alumina Toughened Zirconia Parts

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Ms. Berfu Goeksel [1], Prof. Jozef Vleugels [1], Prof. Annabel Braem [1]

***1.** KU Leuven*

Direct Ink Writing (DIW) is an extrusion-based additive manufacturing (AM) technique that allows the production of high-density ceramic parts with complex shapes. Parts are built in a layer-by-layer fashion by a continuous extrusion of a high solid loading viscoelastic paste through a narrow nozzle. A computer-aided design (CAD) model is used to control the movement of the nozzle to build the object with high precision.

The overall dimensional accuracy, roughness, and density of the printed and sintered parts are mostly dependent on the printing parameters. Deciding these parameters is also crucial for the mechanical properties since, it is not possible to remove printing defects in the subsequent drying-debinding-sintering steps. The resolution of the printed parts and surface quality is mostly dependent on nozzle shape and size while the dimensional accuracy is mostly related to the extrusion rate, i.e. pressure and nozzle speed. Moreover, carefully selecting the correct raster pattern, filament spacing and layer height is required for high-density parts with minimum defects.

The DIW printed objects are green bodies, requiring an appropriate drying-debinding-sintering route for densification. As the pastes studied are high solid loading aqueous pastes, controlled humidity drying is required to avoid drying cracks and warping. Drying is followed by debinding and sintering to remove the organic binder in the paste and to reach full densification.

To obtain the desired printing quality, optimization of printing parameters as well as the drying-debinding-sintering route is crucial. In this study, the aforementioned parameters were studied to produce high-density monolithic alumina toughened zirconia parts with high accuracy.

Development of Robocasting for Oxide/Non-Oxide Ceramics

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Dr. Elliot Douse [1]

1. *Lucideon*

Lucideon has developed a range of water-based ceramic pastes suitable for robocasting advanced ceramics such as alumina, sintered silicon carbide and nitride bonded silicon carbide. Paste formulations have been shown to produce high density complex parts with a range of geometric features using hardware designed for printing clay.

This presentation will highlight the properties of each material and show the types of geometry which can be manufactured from each paste. In addition, the development process will be explored, reviewing paste formulating, printing parameters, sintering and characterization. Applications for ceramics manufactured from robocasting will also be explored, assessing how geometric features only possible to create via additive manufacture can lead to novel applications and improved performance.

Structured NH3 sorbents fabricated by Direct Ink Writing of SrCl2-based composites

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

***Mr. Marco D'Agostini* [1], *Mr. Ali Ezzine* [1], *Mr. Nasir Shezad* [2], *Prof. Paolo Colombo* [1], *Prof. Farid Akhtar* [2], *Dr. Giorgia Franchin* [1]**

***1.** Department of Industrial Engineering, University of Padova, **2.** Department of Engineering Sciences and Mathematics, Luleå University of Technology (LTU)*

Selective catalytic reduction (SCR) of NO_X is an effective strategy for pollution abatement in internal combustion engines: by using NH_3 as a reducing agent over a suitable catalyst, NO_X can be converted to N_2 and H_2O resulting in a significant decrease of harmful emissions. Commercial application is however complicated by the fact that NH_3 is a flammable and corrosive gas, posing substantial risks when stored in pressurised vessels: commercial SCR units instead employ urea as reducing agent, which can be stored and dosed more safely but also presents the disadvantage of releasing CO_2 as a byproduct of the reaction. Alkali earth metal halides (AEMHs) display the ability to readily absorb and release NH_3 by forming ammine complexes and thus offer a safe solid-state storage alternative which operates near ambient pressure, but the significant volumetric expansion associated with the formation of the ammine complexes makes their use impractical. Such limitation can however be mitigated through the design possibilities of Additive Manufacturing.

The authors present here a novel approach to shape $SrCl_2$ into a structured sorbent for NH_3 storage in SCR units by Direct Ink Writing (DIW) of $SrCl_2$-bentonite slurries. The porous lattice design of the 3D-printed component improves access of NH_3 to the salt and more easily accommodates the volumetric expansion of $SrCl_2$ (over 400%) to preserve the structural integrity of the component. The use of bentonite allows the printed monoliths to achieve good mechanical properties after drying without the need for heat treatments, which are problematic due to the poor stability of $SrCl_2$ at high temperature. The $SrCl_2$-bentonite monoliths were tested for NH_3 absorption where they displayed comparable storage capacity to that of the pure salt and the ability to withstand numerous absorption-desorption cycles without loss of performance or structural integrity.

3D Printing of Alumina by Direct Ink Writing (DIW) from Inorganic Salt Precursors

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

***Dr. José Antonio Padilla Sánchez* [1], *Mr. Pol Barcelona* [1], *Mr. Adrià Urban Palomar* [1], *Dr. Mònica Martínez* [1], *Dr. Mercè Segarra* [1], *Dr. Elena Xuriguera* [1]**

1. *Universitat de Barcelona*

Direct Ink Writing (DIW) is an additive manufacturing (AM) extrusion-based 3D-printing technique in which a filament of paste (known as an “ink”) is extruded from a small nozzle while the nozzle’s position is controlled by a computer in accordance with a CAD model. An ink for robocasting must be highly shear thinning behaviour to allow extrusion through fine nozzles and retain a degree of strength and stiffness to be self-supporting following printing. There are several options to get these rheological conditions, being the use of inks based on a hydrogel as carrier one of them.

Usually, inks with a high solid load of ceramics powder are required, commonly above 40 vol%. The high content entails the use of additives that prevent agglomeration and/or sedimentation, ensuring the homogeneity and stability of the suspension, with a proper viscosity and workability. An alternative strategy to ceramic powder is the use of ceramic precursors in the form of soluble salts. This type of chemical compounds allows to obtain small size pieces with micro details due to the large shrinkage generated and porosity.

The present work shows the preparation of inks based on ceramic precursors to obtaining Al_2O_3 ceramic pieces specifically inorganic aluminium salts. Inks are based water and the gelling agent is Pluronic F-127. A complete rheological characterization of the inks has been carried out to ensure printability for DIW, TGA/DTA experiments were performed to study the thermal decomposition and determine the parameters for the thermal treatments need it to obtain alumina after the printing. The evolution of the crystalline phases was studied by XRD and microstructural characterization by SEM.

γ-alumina coating on α-alumina parts fabricated by Direct Ink Writing for catalytic applications

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Mr. SEYED ALI RAZAVI [1], Dr. Miguel Morales Comas [1], Ms. Junhui Zhang [1], Ms. Mona Yarahmadi [1], Mr. Otman Moumen [2], Prof. Luis Miguel Llanes Pitarch [1], Dr. GEMMA FARGAS [1]

1. *1.CIEFMA-Department of Materials Science, Universitat Politècnica de Catalunya, Barcelona-Tech/ 2.Centre for Research in Multiscale Engineering of Barcelona, Universitat Politècnica de Catalunya, Barcelona-Tech,* ***2.*** *CIEFMA-Department of Materials Science, Universitat Politècnica de Catalunya, Barcelona-Tech*

Alumina is widely used as catalytic support because of its high chemical inertness, strength, and hardness. As a polymorphic material, has seven different crystalline structures; of which alpha(α) and gamma(γ) are the most interesting phases for catalytic applications. γ-alumina possesses excellent surface area owing to the small particle size, which results in a high activity of the surface for catalyst support, while α-alumina is characterized to display better mechanical properties. In this sense, this work was focused on producing α-alumina as a core coated with γ-alumina. In doing so, the Direct Ink Writing technique was used to print α-alumina parts. The sintering process has been carried out in a range of temperatures between 600 and 1450 ºC to determine the optimal conditions in roughness to enhance the adhesion of γ-alumina coating. The coating procedure was performed by immersing printed samples in a γ-alumina suspension at different times (10-20 s). Cross-sections of resulted samples were analyzed by Raman spectroscopy to identify the presence of α and γ-alumina phases at the surface. Adhesion of the coating was determined by scratch tests. Results revealed that roughness has a significant effect on the adhesion of γ-alumina unlike immersion time in the coating procedure. Raman spectroscopy analysis evidenced the formation of a γ-alumina coating on the α-alumina substrate for all studied conditions. Coating adhesion was higher for supports sintered at lower temperatures.

Thermal transfer and effective properties of pre-heated powder bed in laser additive manufacturing: a phase-field investigation

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Mr. Prasanth Bondi [1], Mr. Yangyiwei Yang [1], Prof. Bai-Xiang Xu [1]
***1.** TU Darmstadt*

In laser additive manufacturing (LAM), understanding the parameter dependence on the properties of interest is the key to designing and producing consistently good quality products[1]. Melt pool size proved crucial in building cellular lattice structures with good quality using laser additive manufacturing (LAM) process[1,2]. Such structures are of particular interest due to their unique mechanical properties[2]. Meanwhile, pre-heating in LAM is an essential step owing to improved structural stability and reduction in internal stresses and distortion. An improvement in dimensional accuracy in the finished products produced using LAM process is also reported[3]. Effects of powder size distribution on the effective thermal conductivity of powder bed are evident from the previous studies[4], which attributes to the relative density of the powder bed rather than the material property itself. However, there are not many studies available on how powder size characteristics affect the thermal conductivity of a pre-heated powder bed. It is of great interest to predict the melt pool width using our data-driven phenomenological LAM model[1], since it's an essential factor affecting the melt pool width as per our previous study.

In this work, we investigated several powder beds of stainless steel 316L with varying powder size distributions to evaluate the dependence of powder size characteristics on the thermal conductivity of pre-heated powder beds. Spherical particles with distinct size distribution were pre-heated under 600 ^{o}C. Isothermal phase-field pre-heating simulations were performed to present the microstructure evolution during pre-heating, where the neck size is adopted to quantify the degree of pre-heating. Effective thermal properties based on transient microstructure were then evaluated by diffuse-interface based thermal homogenization. Data-driven analysis was also performed to investigate further the correlation and the sensitivity of the powder bed conductivity to factors of interest, incl. the powder size distribution, packing density, and degree of pre-heating.

Towards a single-step ceramic additive manufacturing process by promoting liquid phase sintering at low temperature

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

***Dr. Nicolas Somers* [1]*, Dr. Alejandro Monton* [1]*, Prof. Mark Losego* [1]**

***1.** Georgia Institute of Technology*

Additive Manufacturing (AM) of ceramics is currently limited by the necessary and long post treatment after printing to remove organics and densify the ceramic body. The grail of ceramics AM would be to create a single-step, low-temperature AM process without the need for post-process pyrolysis. This work aims to reach this goal by using low melting points additives or “filler” particles that become viscous at modest temperatures or under illumination. Formulations and methodologies for binding together ceramic powders of arbitrary composition are optimized based on inorganic condensation reactions, sol-gel chemistry and liquid phase sintering. By making these binding chemical reactions light-activated, an excitation process where each 3D printed layer is then exposed to some external energy source to drive densification is developed. Layer by layer, a mechanically stable green body can be created by photo-assisted aerosol jet printing or photo-assisted Direct Ink Writing.

SLA-DLP of 3D ceramic packaging for high power electronics applications (GaN, SiC)

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

***Ms. Charlotte Meuly* [1], *Mr. Loïc Teisserenc* [1], *Ms. Marie Beaujard* [1], *Dr. Lucile Mage* [1], *Dr. Regis Delsol* [1], *Dr. Emmanuel Marcault* [1]**

1. CEA Tech Occitanie

This work focuses on the development of 3D ceramic packaging to reach requirements of future high power devices (gallium-nitride and silicon carbide semi-conductors). New designs of ceramic hoods are developed to ensure efficient cooling, optimize power density and improve compactness of those new electronic devices.
Initially, thermal modeling is performed with Ansys Mechanical to optimize 3D packaging designs and identify ceramic materials that will ensure good heat dissipation. Those designs are shaped by stereolithography (SLA-DLP). Alumina hoods with good geometry and tolerances are obtained.
In order to improve heat dissipation, it is necessary to get a good interface between the hood and the ship. A specific PCB is developed and an interface (TIM, Thermal Interface Material) between the hood and the chip is added. A specific tooling and gluing process is developed to ensure a good alignment of parts and ensure a good adhesion of the TIM.
Finally, first result on a packaging 100% ceramic will be shared.

Strategies for the fabrication of carbide components by coupling sol-gel chemistry and photopolymerization-based additive manufacturing

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

***Ms. Alice Zanini* [1], *Ms. May Yam Moshkovitz* [2], *Dr. Sara Maria Carturan* [3], *Dr. Stefano Corradetti* [3], *Prof. Paolo Colombo* [1], *Prof. Shlomo Magdassi* [2], *Dr. Giorgia Franchin* [1]**

***1.** Industrial Engineering Department, University of Padova, **2.** Hebrew University of Jerusalem, **3.** INFN – Laboratori Nazionali di Legnaro, Legnaro, PD, Italy*

Photopolymerization-based additive manufacturing has gained worldwide attention in recent years as a proven and flexible technique for the fabrication of an enormous variety of materials, thus opening up novel pathways for utterly complex architectures over different length scales. To meet the growing demand of finely customized materials, the coupling of photopolymerization-based additive manufacturing and sol-gel chemistry has been receiving considerable interest and provides a unique tool to explore a new generation of materials with advanced performances; particular focus is devoted to hybrid organic-inorganic precursors for the synthesis of ceramics. Sol-gel-based feedstocks rely on the assembly of the molecular building blocks into dual or multiple interlocking networks, with the photopolymerization-derived network in addition to the one derived from hydrolysis and condensation. The interplay between the inorganic and organic species plays a significant role in the arrangement of the structuring units, therefore holding particular importance for the final properties. Research endeavours are still ongoing to unravel the several acting mechanisms and this work has been pursued aiming at developing a versatile and feasible protocol for the fabrication of titanium carbide components by means of sol-gel processing combined with stereolithography. A bottom-up approach has been therefore designed to achieve a control over the organization and positioning of the structural units of the hybrid network, and to offer a broad range of possibilities to synthesize tailor-made components with tunable properties, with particular focus on nanoscale porosity. Indeed, this synthetic strategy takes advantage of its inherent flexibility, which allows for a large variety of modifications by easily playing with the choice of the network-forming structural motifs, and can be potentially extended to the fabrication of different carbide-based materials.

3D-printing of SiC by Digital Light Processing using submicron SiC-powder and HDDA (1,6-hexanediol diacrylate)

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

***Ms. Maria Mykland* [1]*, Prof. Kjell Wiik* [2]*, Prof. Mari-Ann Einarsrud* [2]*, Mr. Vidar Johannessen* [3]**

***1.** Department of Materials Science and Engineering, NTNU Norwegian University of Science and Technology, 7491 Trondheim & Fiven Norge AS, 4790 Lillesand, **2.** Department of Materials Science and Engineering, NTNU Norwegian University of Science and Technology, 7491 Trondheim, **3.** Fiven Norge AS, 4790 Lillesand*

SiC is a ceramic with a high refractoriness and decomposes to volatile Si-species and graphite at temperatures above 2300^{o}C. Forming and densifying SiC is a challenge, and green machining and post-sinter treatment is often necessary to obtain the final geometry for a given application. 3D-printing of green bodies based on alumina using Digital Light Processing (DLP) has been accomplished giving high surface finish and accurate geometry within a short process-time. Our aim is to develop a DLP-technique for SiC to produce sinterable green bodies. However, to utilize DLP-printing with UV-triggered polymerization, optical properties like transmittance, absorbance and reflection of the ceramic particles are crucial to the penetration depth of the incoming light into the dispersions. First part of the investigation will report optical properties of SiC-particles with varying purities (99,0-99,99 %) and sizes (500-700 nm) in dispersions with the photomonomer, 1,6-hexanediol diacrylate.
Second part of the investigation presents of stability and rheology of the SiC-dispersions as a function of the solid loading. Dispersions are prepared by mixing photomonomer, surfactants and 2-propanol prior to adding SiC-particles. Solid loading is pivotal to the sinterability of the green body and should be as high as possible.
Results from 3D-printing of optimized dispersions will be presented and discussed with respect to rheology, optical properties, and penetration depth.

Acknowledgements
Financial support from the Research Council of Norway (project number ES 690878) and Fiven Norge AS is gratefully acknowledged

Design and fabrication of 3D-printed triply periodic minimal surface optically transparent/translucent scaffolds for sensing applications

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Dr. Arish Dasan [1], Dr. Jozef Kraxner [2], Prof. Dušan Galusek [3], Prof. Enrico Bernardo [4]

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The design and fabrication of optically transparent glass systems shaped in 3D form features facilitate applications in various areas such as sensing, optics and photonics. Despite the advent of 3D glass printing methods, many issues still remain, including limited materials availability, resolution, and surface roughness of printed parts. This study reports on the fabrication of transparent/translucent glass objects with complex shapes (triply periodic minimal surface) using stereolithography. To use this technology, the glass powder, obtained from the crushing of crystal glass (RONA glass, Slovakia), was dispersed in a photosensitive suspension with a solid loading of at least 55 wt%. The green parts were printed and later converted into sintered objects via controlled heat treatment. The role of viscous flow sintering is discussed and the mechanical performance of the sintered materials is studied. The parts produced with the use of glass powders demonstrate the advantages and current limitations of the approach. Direct doping of rare-earth (RE)-based oxyfluoride nanocrystals with glass powders can be used for optical thermometry sensing applications.

Digital Light Processing of preceramic polymers for the preparation of bioactive modified SiOC ceramics

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Dr. Piotr Jeleń [1], Dr. Jakub Marchewka [2], Mr. Patryk Bezkosty [2], Mrs. Justyna Grygierek [1], Prof. Maciej Sitarz [2]

__1.__ Akademia Górniczo-Hutnicza w Krakowie, __2.__ Faculty of Material Science and Ceramics, AGH University of Science and Technology, al. Mickiewicza 30, 30-059 Kraków, Poland

The Polymer Derived Ceramics (PDCs) are a variety of materials obtained from polymeric precursors. The use of such a type of materials allows for their relatively simple and effective preparation for a specific application, including bioactive materials and materials for catalysis, etc. In addition, this simplicity allows for easy modification and optimization of the processes of obtaining and printing such materials. Black glasses, or silicon oxycarbides, are such materials.

Due to the use of organosilicon polymers as preceramic materials it is possible to control the Si:C and Si:O ratio of final materials. Also the amount and form of the free carbon phase can be controlled. Additional modifications can also be introduced in the form of cationic substitutions enhancing desired properties of the final product.

The aim of this study was to introduce into the photocurable polysilsesquioxanes matrix, containing methacrylate groups, the additives in the form of nitrates like cerium or copper nitrate to enhance bioactivity of the PDCs material. In order to fabricate desired structures Digital Light Processing 3D printing using Cellink Lumen X+ was applied.

Detailed spectroscopic research (FT-IR and Raman) were employed during preparation process of Ce and Cu modified photocurable polysilsesquioxanes materials to study the reaction mechanisms. Obtained 3D printed preceramic samples were subjected to thermal processing and extensive structural (FT-IR, Raman) as well as microstructural (SEM, BET) studies.

This project was supported by National Science Center grant number 2019/35/B/ST5/00338 “New biocompatible coatings on metallic substrates based on materials from the Si-O-C system”

3D silicon oxycarbide structures prepared by Digital Light Processing

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Dr. Jakub Marchewka [1], Mr. Patryk Bezkosty [1], Ms. Izabela Rutkowska [1], Prof. Maciej Sitarz [1]

1. *Faculty of Material Science and Ceramics, AGH University of Science and Technology, al. Mickiewicza 30, 30-059 Kraków, Poland*

Preceramic polymers used for the preparation of Polymer-derived Ceramics (PDCs) are the new materials extensively developed in the recent years. Their main advantage is the ability of processing using the methods typical for polymers. With their application, the samples with complex geometry which are subsequently converted to ceramics may be obtained. It provides more possibilities for the preparation of materials with a specific microstructure compared to the direct processing of ceramics. Therefore, this concept is very attractive in the preparation of new materials.
Silicon oxycarbide (SiOC) is one of the most interesting silicon-based ceramics because of its physicochemical properties which originate from the characteristic structure. Typical amorphous structure of silica is modified by the introduction of carbon as a substituent of some oxygen as well as a separate phase. The total content of carbon and the ratio of its two forms determine the final properties of the product.
The research involved the development of new procedure for the preparation of 3D SiOC structures using Digital Light Processing 3D printing. This included the following steps:
(1) sol-gel synthesis of photocurable polysilsesquioxanes with the introduction of methacrylate groups designed to obtain a specific amount of carbon in the final material,
(2) optimization of the 3D printing process using Cellink Lumen X+ including the selection of the photoinitiator system to provide the efficient photopolymerization process and the preparation of the 3D samples which fully reproduce the predesigned virtual models,
(3) thermal processing of the printed samples to obtain the final 3D SiOC structures.
The products were characterized in terms of their microstructure (Scanning Electron Microscopy) and structure (FT-IR and Raman spectroscopy).
This research was funded by the National Science Centre, Poland, grant no. 2020/37/B/ST8/02859 and supported by the program „Excellence initiative – research university" for the AGH University of Science and Technology.

Design and manufacturing of self-supported electrolytes for solid oxide fuel cells application by Stereolithography

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Dr. Anastasiia Novokhatska [1], Dr. Arish Dasan [1], Dr. Jozef Kraxner [1], Prof. Dušan Galusek [2]

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Solid oxide fuel cells (SOFCs) directly convert the chemical energy of fuels into electricity. They have attracted great interest worldwide due to their high efficiency and excellent fuel flexibility. Despite these advantages, commercialization of SOFC technology has been restricted by its high-cost and complicated procedure of cell fabrication. Stereolithography has widened the scope for designing more performing microstructures for SOFCs. Topological modifications of electrolytes facilitate ion transport and increase the electrochemical performance of the cell. In this work new approaches of 3D modeling were explored with the aim of thickness reduction and increase of surface area of yttria-stabilized zirconia self-supported electrolytes. Planar and wave-shaped electrolytes with different thicknesses were printed and sintered at up to 1400°C in air and vacuum. The shrinkage, mechanical properties and density of sintered ceramics were investigated. Furthermore, ceramics microstructure and phase stability of yttria-stabilized zirconia were examined.

Comparison of accuracy of additively manufactured zirconia four-unit fixed dental prostheses

Wednesday, 9th November - 10:50: Poster session I and Coffee break - Poster

Mr. Jörg Lüchtenborg [1], Dr. Evita Willems [2], Mr. Fei Zhang [2], Mr. Christian Wesemann [1], Mr. Florian Weiss [1], Mr. Julian Nold [1], Mr. Jinxing Sun [3], Mr. Fabien Sandra [4], Mr. Jiaming Bai [5], Dr. Helen Reveron [4], Prof. Jérôme Chevalier [6], Dr. Benedikt Spies [7]

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In recent years, Additive Manufacturing (AM) has become an integral part of various dental applications. Nowadays, in dentistry, the focus is on AM of polymers and metals, producing a variety of parts with different technologies for the use in dental clinics. The increasing demand for metal-free restorations results in a higher need for ceramic material like zirconia. This poster discusses the manufacturing accuracy of zirconia four-unit fixed dental prostheses (FDPs) fabricated by three different additive manufacturing technologies compared with substractive manufacturing. The accuracy of the manufactured parts is of significant importance for the clinical application. Inaccurate marginal fit can be responsible for plaque retention, micro-leakage and cement breakdown. Poor internal accuracy can increase the thickness of the cement and thus influence the mechanical stability of zirconia-based restorations. Parts produced by stereolithography, material jetting and digital light processing were digitized and the accuracy of three Regions Of Interest (inner/outer shell, margin) is presented here.

Poster session II

Ceramic-based 3D scaffolds for biomedical application by FFF using a colloidal feedstock

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Mr. Alvaro Eguiluz [1], Dr. Ana Ferrández Montero [1], Mr. Pablo Ortega Columbrans [1], Dr. Javier Sanchez-Herencia [1], Dr. Begoña Ferrari [1]

1. *Instituto de Cerámica y Vidrio, CSIC*

This work is aimed to develop an additive manufacturing (AM) method to process porous hydroxyapatite-based scaffolds with high potential in the field of medicine. Over recent years, AM techniques have gained a special attention in order to process complex structures and patient-customized pieces for biomedical application. One of the most known technique is Fused Filament Fabrication (FFF), which highlight as an economic, simple and fast method in comparison with similar other AM technologies. However, FFF is limited to process thermoplastic materials, therefore it is restricted to polymer feedstock or polymer-based composites with low amount of inorganic load dispersed into the organic matrix. In this work, a new route to process composite thermoplastic composites with a high ceramic content has been developed. In addition, it is presented a procedure to measure the oscillatory rheology of the melt composites in terms of the parameters related with the 3D printing.

In this work, filaments and feedstock with high inorganic content of Hydroxyapatite-based (HA) have been developed, using polylactic acid (PLA) and polyethilenglycol (PEG) as processing agents to provide the thermoplasticity necessary for FFF technique. Melt composites are characterized through oscillatory rheology in order to determine the main parameters associated to the extrusion and printing processes. These results provide understanding to what happens to the HA filaments during printing, and also how the inorganic content conditions the printability and affects to the obtaining of a final full ceramic 3D object of HA.

Fused Deposition Modelling of Fibre Reinforced Ceramic Matrix Composites

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

***Ms. Daorong Ye* [1]*, Prof. Jon Binner* [1]**

***1.** University of Birmingham*

Ceramic matrix composites, CMCs, are finding many uses in the challenging environments that are becoming increasingly common in modern society. Fibre-reinforced CMCs have a higher toughness compared to the monolithic ceramics and can also be tailored to offer a wide range of other properties too, e.g., carbon fibre-reinforced ultra-high temperature CMCs can be used in extreme environments where temperatures can exceed 2500oC.

This work, which has just started, focuses on using additive manufacturing for the fabrication of fibre reinforced ceramics. The ultimate goal is to achieve the simultaneous fabrication of both the reinforcement and the matrix. The approach being investigated involves the accurate controlled use of fused deposition modelling (FDM), which it is hoped will enable flexibility in the structural and material design of fibre-reinforced CMCs, whilst being capable of producing parts of complex shape and good surface finish, potentially without a need for final machining. The influence of filament composition and FDM parameters are currently being investigated. The research aims to improve the thermo-mechanical properties of fibre-reinforced CMCs via multiple approaches, reducing the demand for subsequent post-processing for densification.

Characterization of 3D Printed Calcium Carbonate and Zirconia-filled Polylactic Acid parts

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

***Mr. Daniel Vidal* [1], *Dr. Irene Buj Corral* [1], *Mrs. Nohila Belkourchia* [2]**

***1.** Universitat Politècnica de Catalunya, **2.** Polytech Montpellier*

Additive manufacturing (AM) processes are nowadays well introduced in different segments: medical, aeronautics, packaging, industrial, electronics, optical,… An important application for 3D printing is the manufacture of prostheses, which usually have complex shapes with porous structures.
Specifically, Fused filament fabrication (FFF) 3D printing technology was used for the printings in this work. Main advantages of the technique are the easiness of use and the possibility to manufacture complex shapes.
The main aim in the present work is 3D printing and characterizing Calcium Carbonate ($CaCO_3$) and Zirconia-filled Polylactic Acid (PLA) parts for use in prostheses. Cylindrical samples were defined in two different sizes: diameter 6,35 mm x 12,70 mm height, according to ASTM C1424 - 15 Standard for ceramics, and diameter 12,7 mm x 25,4 mm height, according to ASTM D695 Standard for plastics. Six replicates were obtained of each experiment. The purpose is to compare the performance of the samples printed in different sizes.
After 3D printing, the main parameters for the characterization were: surface roughness, dimensional error, density and total porosity.
Similar results were obtaining regardless of the size of the samples, showing the option to manufacture samples in different sizes, depending on the standard considered, either for ceramic or for plastic materials.
Keywords: additive manufacturing; 3D printing; prostheses; porous structures; Polylactic Acid (PLA); Calcium Carbonate ($CaCO_3$); Zirconia; ASTM; surface roughness; dimensional error; density; total porosity; Fused filament fabrication (FFF)

PREDICTION OF MECHANICAL BEHAVIOR OF 3D PRINTED CNT-ZnO BASED STRUCTURES

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Ms. Maria- Eliza Puscasu [1], Ms. Stefania Chiriac [1], Dr. Laura Madalina Cursaru [2], Dr. Radu Robert Piticescu [1], Ms. Lidia Licu [1], Ms. Anca Elena Slobozeanu [1]

***1.** National RD Institute for Non-ferrous and Rare Metals (IMNR), **2.** National RD Institute for Non-ferrous and Rare Metals*

In their interaction, composite materials based on carbon nanotubes (CNT) and zinc oxide (ZnO) presents properties with tremendous potential in a variety of domains. By combining the capacity to absorb a wide range of contaminants and large specific surface area of CNT with the environmental-friendly ZnO, a material with promising efficiency in wastewater treatment could be obtained. In order to process CNT-ZnO based structures with a geometry that will facilitate a better filtration of the water, the additive manufacturing technology is needed. The obtained 3D structure has to present a suitable mechanical behavior in order to withstand the filtration process. Thus, it is required that the printed geometry to undergo mechanical examination.

In this study, the CNT-ZnO 3D structures obtained via robocasting were designed considering the previously used printing parameters. The CAD model was subjected to a static simulation in order to make a prediction of the mechanical behavior of the CNT-ZnO based structures.

ZnO-CNT nanocomposite powder prepared by hydrothermal synthesis was used to obtain 3D structures via robocasting technique. Considering the input parameters of the 3D structures, a CAD model was designed using the software SolidWorks 2019. The designed structure was subjected to static pressure tests at 0.1 MPa, 0.5 MPa and 1 MPa. The minimum and maximum values for Von Mises stress and dislocation were determined under static conditions.

The results of the simulation process indicated that the structures will not fracture under the action of mechanical stress. Furthermore, under the action of applied pressures, the CAD structure shows a predictable displacement that is likely to occur mainly in the center of the structure. The results obtained in the simulation are a satisfactory indicator of the mechanical potential of the obtained architectures.

Effect of geometry on mechanical properties of 3D printed dense zirconia ceramics parts produced by robocasting

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Ms. Junhui Zhang [1], Ms. Mona Yarahmadi [1], Mr. Marc Serra Fanals [1], Mr. Sergio Alberto Elizalde Huitrón [1], Prof. Luis Miguel Llanes Pitarch [1], Dr. GEMMA FARGAS [1]

1. Universitat Politècnica de Catalunya

Advanced ceramic materials have attracted large interest in various modern industrial and practical engineering applications due to their excellent mechanical and physical properties. Additive manufacturing offers new opportunities to manufacture advanced ceramic materials with increased geometry complexity and reduced waste material. Among these additive manufacturing techniques, robocasting is often considered to perform fine and dense ceramic structures with geometrically complex morphology and high strength, thanks to the high solid loading in the feedstock. The main goal of this work is to develop dense zirconia ceramics by evaluating the effects of different geometries on mechanical performances of sintered parts. In this work, three-dimensional 8 mol.% yttria-stabilized zirconia (8Y-ZrO_2) ceramic parts with rectilinear (0/90º and 45º) and cylindric geometries were printed via robocasting with a 100 % infill. A printable ink was developed using an inverse-thermoresponsive hydrogel, Pluronic® F-127, and 8Y-ZrO_2 ceramic powder with a solid loading of 70 wt.%. Volumetric shrinkage and relative density together with mechanical properties were determined. Results revealed that cylindric printed samples presented higher volumetric shrinkage and density while rectilinear ones with filaments oriented at 45º displayed the highest compression strength.

ADDITIVE MANUFACTURING OF ZnO-CNT STRUCTURES WITH POTENTIAL APPLICATION IN ENERGY STORAGE OR WASTEWATER TREATMENT

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Ms. Stefania Chiriac [1], Ms. Maria- Eliza Puscasu [1], Dr. Laura Madalina Cursaru [2], Dr. Radu Robert Piticescu [1], Ms. Lidia Licu [1]

1. National RD Institute for Non-ferrous and Rare Metals (IMNR), 2. National RD Institute for Non-ferrous and Rare Metals

Composite materials based on carbon nanotubes and metal oxides (CNT / MO) combine the unique properties of CNTs with those of metal oxides and have extraordinary properties resulting from their interaction. Zinc oxide nanoparticles had shown great promise in the area of water and wastewater treatment due to its high BET surface area, strong oxidation ability and good photocatalytic capability. Carbon nanotubes can be easily modified by functionalization of the material surface and it presents large specific surface area, high chemical stability, the capability to adsorb a great variety of contaminants and high electrical conductivity. Therefore, reinforcing ZnO with CNT could be an interesting strategy in order to obtain a composite material with better properties with potential application in different fields such as energy storage and wastewater treatment. Over the past five years, additive manufacturing (AM) has shifted from a tool used exclusively in the industry for rapid prototyping to a new, far-reaching approach to the development of high-value products. The researchers also explore the potential of using 3D printing techniques in composite printing, which is why the main purpose of this study was to obtain three-dimensional structures based on ZnO-CNT composite powder by robocasting. During this work, 3D parts with different printing characteristics were designed and manufactured based on ZnO-CNT hydrothermally synthesized powder. The 3D bodies thus obtained were characterized from a morphological point of view with the help of the scanning electron microscope. Some of the 3D structures were impregnated in sodium nitrate (with the role of a phase change material) using the solvothermal method process at high pressure in order to evaluate their potential in energy storage application. Also, in order to evaluate the potential of these structures in wastewater treatment applications, preliminary tests for retaining Cd ions from standard solutions prepared in the laboratory were performed.

Characterization of porous zirconia-based ceramics with complex geometries produced by robocasting

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Mrs. Ludmila Hodasova [1], Mrs. Paula Pou [1], Ms. Mona Yarahmadi [1], Prof. Elaine Armelin [2], Prof. Luis Miguel Llanes Pitarch [1], Dr. GEMMA FARGAS [1]

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3 mol% Yttria-Stabilized Zirconia Polycrystal (3Y-TZP) exhibits an excellent combination of mechanical strength, fracture toughness, biocompatibility, and bioactivity. This is one of the reasons why 3Y-TZP can be used in the biomedical field, which requires complex shapes and geometries together with mechanical resilience and in the case of dental applications also aesthetics. This complexity cannot be achieved by traditional manufacturing processes, therefore additive manufacturing is a suitable alternative. With these requirements, multiple porous geometries, including rectilinear at 0/90 and 45^{o}, triangular, concentric, and cylindrical were produced by the robocasting technique using 3Y-TZP/Pluronic hydrogel paste as printing material and an 840 mm nozzle for extrusion. After sintering at 1450 ^{o}C for 2 hours, samples were analyzed by micro-computerized tomography (m-CT) to observe filament placement and pore distribution in the cross-sections and Field Emission Scanning Electron Microscopy (FESEM) to follow microcracks or manufacturing faults of different shapes. Compression tests were carried out to determine the stress-strain curve for each condition and to elucidate differences between them. Results revealed that rectilinear geometry at 45^{o}displayed higher strength in contrast to concentric and cylindrical ones.

Design and Fabrication of Calcium Phosphate Scaffolds with Concave Surfaces by Direct Ink Writing

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Dr. Irene Lodoso [1]***, Mr. Ethan Segura*** [1]***, Mr. Oriol Romeu*** [1]***, Prof. Maria Pau Ginebra*** [1]

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Osteoinductive biomaterials are those capable to induce the osteogenesis process, by stimulating pluripotent stem cells to differentiate into bone-forming cells[1]. Such biomaterials hold great potential in bone regeneration applications. Recent studies have shown that osteoinduction is highly benefited from the presence of concave surfaces[2]. This represents a challenge for the application of extrusion-based 3D printing techniques in the fabrication of synthetic bone grafts, due to the convex surface of the extruded filaments. The aim of this project was to overcome this limitation by developing a novel method that relies on the fabrication of calcium phosphate scaffolds with concave surfaces by the infiltration of sacrificial polymeric moulds with self-setting calcium phosphate slurries and their subsequent dissolution. The goal was to obtain ceramic scaffolds with concave porosity which were the negative of the polymeric mould printed by direct ink writing. Printing parameters such as infill density, pattern and layer height were modified and their effect on scaffold porosity and mechanical properties was analysed. Infill density did not affect the pores' geometry or dimensions but increasing infill densities of the printed moulds increased porosity and decreased the compressive strength. Three infill patterns were studied; perpendicular, gyroid and crossed, of which the perpendicular pattern had the highest compressive strength for the same porosity values. Layer height affected pore size and their geometry, as oval pores were obtained for smaller layer heights. This translated into lower porosity values and, as a consequence, lower compressive strengths. Overall, infill percentage and infill pattern had the highest influence on mechanical properties and infill percentage on porosity. We concluded that the use of sacrificial moulds is a promising approach to obtain ceramic scaffolds with concave porosities that could promote osteoinduction.

[1]Albrektsson & Johansson, Eur. Spine J. 10 (2001)

[2]Barba, et al. ACS Appl. Mater. Interfaces , 9, 48 (2017)

Direct ink writing of porous geopolymers for thermochemical energy storage

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Ms. Camille Zoude [1], Dr. Laurent Gremillard [1], Dr. Elodie Prud'homme [1], Dr. Kevyn Johannes [2]

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Due to the depletion of fossil resources and to the rising ecological awareness, energy storage has become a major issue in our society. Thermochemical storage, based on hygroscopic salts, seems promising to meet these new challenges. However, in order to optimize the thermal properties of these systems, it is necessary to associate the salt with a host material. For our application, this host material must be affordable, resistant and as porous as possible. To meet these requirements, we have chosen to design porous geopolymers. In addition, an energy efficient shaping technology was also chosen: Direct Ink Writing, in order to create a multi-scale porosity.

Different additives were added to an initial geopolymer ink formulation to firstly increase their porosity in order to store more salt, and secondly make their rheology adequate for the printing process.

On a small scale (parts < 8 cm^3), a robocasting machine provided by 3D Inks LLC was used. For parts larger than 8 cm^3, we used a WASP 2040, accessible to the general public, on which we made some technical modifications to allow geopolymer printing.

As a result, structures have been successfully printed and show good fidelity to the model for the two printers. However, even if the samples are more porous (50% against 4.5% without additives), the porosity is not sufficient for our application. Moreover, it is lower than that of the molded samples, which suggests that the 3D printing process modifies the internal structure of the foam.

To improve these results, other strategies are currently considered, such as adjusting the formulation, a new technical modification of the printer or the use of other foaming agents.

Printing microbatteries by Robocasting

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

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The integration of microbatteries in increasingly complex (e.g. flexible or wearable electronics) and numerous electronic devices requires a paradigm shift in their fabrication, towards greater design and fabrication flexibility, lower costs, as well as a reduced environmental footprint. Additive manufacturing can meet this challenge, while allowing innovative internal structuring. This work aims to demonstrate the feasibility of microbatteries printed by Direct Ink Writing [1]. This device allows printing with a current resolution of the order of 200 µm of anode, cathode, and even solid electrolyte materials. By optimizing both the materials and the design of their architecture a better manufacturing flexibility is targeted and a decrease in financial and environmental costs. The optimization of the formulation and printing conditions has allowed to reach a printable and rheologically adapted anode ink at around 50% of dry extract with minimized CMC and carbon black contents and more than 90% of graphite increasing the nominal capacity of the anode. The anode is cycle-tested in a lithium-faced Swagelok with a liquid electrolyte. In parallel with the printing and electrochemical cycling stages, tomography is proving to be a good characterization technique to judge the mechanical strength of a printed ink and, in the long term, to monitor the printed structure over the battery charge/discharge cycles.
For the cathode, two materials are considered: LiFePO4, an inorganic polyanionic material, and Mg(Li2)-p-DHT [2], an organic material which is elaborated by means of a low-energy synthesis chemistry. Finally, the electrolyte must be able to fit into the pores of the electrodes while providing a real electronic barrier between them; photo-ionogels [3] are considered as good candidates. All the components of the batteries could then be printed in complex shapes (interdigitated).

[1] Tabard et al., **2021;** Coffigniez et al., **2021**.
[2] Jouhara et al., **2018**.
[3] Aidoud et al., **2016**.

Study of the properties of ceramic parts printed by stereolithography on an open source machine

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Ms. Cristina Fabuel [1], Dr. Maria Pilar Gómez Tena [1], Mr. Esteban Garcia [1], Dr. Alejandro Saburit [1]

1. ITC-AICE

High-performance ceramic materials have been widely used in the aerospace, biomedical, automotive and electronics industries due to their exceptional properties, such as high strength and hardness, resistance to high temperatures and corrosion.
Traditional manufacturing methods, such as moulding or machining, still face difficulties in manufacturing complex ceramic geometric shapes.
Additive manufacturing technologies offer an opportunity to alter and complement traditional manufacturing paradigms. The ability to directly 3D print parts from digital designs drastically alters design and prototyping workflows. Key technologies contributing to the growth of the 3D printing market include the light curing segment, which accounted for 21% of the 3D systems market in 2021.
Stereolithography makes it possible to print very dense parts and also very small details thanks to the high resolution provided by its thin layers. Therefore, SLA 3D printers are an excellent choice for jewellery applications, dentistry and other types of high-precision functions.
That said, this ceramic additive manufacturing technology requires a high investment that not all companies can afford; the cost of a technical ceramic SLA 3D printer can start at around $150,000 and go up to over $500,000.
3D printing with ceramic resin is also a relatively complex operation involving long preparation times and delicate post-processing steps, such as washing off excess resin, curing, and sintering.

The aim of this project, financed by Instituto Valenciano de Competitividad Empresarial, is to study the properties of ceramic parts printed on an open source machine, specially the influence of the particle size distribution on the density of the resulting parts.

Basic characterization of aluminum oxide 3D structures printed using Digital Light Processing

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Ms. Izabela Rutkowska [1], Dr. Jakub Marchewka [1], Mr. Patryk Bezkosty [1], Prof. Maciej Sitarz [1]

1. *Faculty of Material Science and Ceramics, AGH University of Science and Technology, al. Mickiewicza 30, 30-059 Kraków, Poland*

There is potential to influence the ceramic industry by offering new opportunities to manufacture advanced ceramic components without the need for expensive tooling, thereby reducing production costs and increasing design freedom. Application of Additive Manufacturing technology makes it possible to obtain 3D parts by adding material, layer-by-layer, directly based on virtual 3D model.

The aim of this research is an evaluation of Digital Light Processing (DLP) 3D printing process of aluminum oxide based structures. In order to obtain a printing material, sol-gel synthesis of aluminum sol is provided with introducing photocurable compound. The solid printed structure is made out of preceramic polymer which after thermal treatment turns into ceramic. Materials obtained by this route are called Polymer Derived Ceramics (PDCs). It is the group of materials which offers an interesting way for the preparation of ceramic materials, since preceramic polymers may be processed using standard methods applied for typical polymers. The combination of DLP and PDCs gives an opportunity to obtain ceramic products with strictly predesigned architectures. For the sake of the research, the optimalization of printing parameters were provided. In order to characterize designed structures basic microstructural (SEM) and structural (FTIR, XRD) tests are performed.

This research was funded by the National Science Centre, Poland, grant no. 2020/37/B/ST8/02859.

Sol-gel synthesis of novel photocurable preceramic polymers

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Mr. Patryk Bezkosty [1], Ms. Izabela Rutkowska [1], Dr. Jakub Marchewka [1], Prof. Maciej Sitarz [1]

1. *Faculty of Material Science and Ceramics, AGH University of Science and Technology, al. Mickiewicza 30, 30-059 Kraków, Poland*

3D printing technologies based on the photo-curing mechanism have been developing very quickly in recent years. The most known and used technologies in today's science and industry are Stereolitography (SLA) and Digital Light Processing (DLP). Photosensitive resins used in these methods should have low viscosity or adequate fluidity. Low viscosity resins are of small molecular weights, which result in a high degree of cross-linking of photo-curable materials. As a consequence of this is the high hardness and brittleness of the final product. On the other hand, if the molecular weight of the photosensitive resin is large, the viscosity is too high for printing. Then, a large amount of monomer is needed to make the material suitable for printing, which causes the loss of good performance of the resin. Therefore, for the further development of 3D printing methods based on photopolymerization, it is extremely important to develop a photosensitive resin with low viscosity, good fluidity and high efficiency.

This work presents the preparation of photo-curable resins in the sol-gel process based on polysiloxanes, which belong to the preceramic polymers. Novel materials from the SiOC system have been received with the use of precursors containing methacrylic groups in their structure. This allows the use of such materials in 3D printing technology by the SLA or DLP method. The structure of the synthesized product was determined and the first samples were printed using the DLP method.

Acknowledgment

This research was funded by the National Science Centre, Poland, grant no. 2019/35/B/ST5/00338 and supported by the program „Excellence initiative – research university" for the AGH University of Science and Technology.

Preparation of ceramic suspensions for blue-light stereolithography

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Dr. Přemysl Šťastný [1]***, Dr. Daniel Drdlík*** [1]***, Dr. Pavlína Šárfy*** [1]***, Dr. Klára Částková*** [1]

1. *Advanced Ceramic Materials CEITEC*

The poster describes the development of photosensitive ceramic suspensions cured by a blue light (λ=455 nm). The backbone of the suspension is based on a mixture of 1,6-hexanediol diacrylate and trimethylolpropane triacrylate. The photoinitiator – co-photoinitiator mixture of camphorquinone and 2-(dimethylamino ethyl methacrylate) was used. The premix was loaded with alumina (Al_2O_3) ceramic powder with a specific surface area of 11.85 $m^2.g^{-1}$ up to solid loading of 83 wt%. The optimal solid loading and dispersant content were studied. The prepared suspensions were test-printed, and the microstructural quality of 3D-printed parts was evaluated.

Strategies to enhance properties of 3D-printed ceramics

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Ms. Anna-Katharina Hofer [1], Mr. Josef Schlacher [1], Dr. Martin Schwentenwein [2], Prof. Raul Bermejo [1]

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Technologies of additive manufacturing (AM) impress with the ability of fabricating ceramic parts of high precision and complexity. However, their mechanical performance may be affected by printing process-related parameters as compared to conventionally processed ceramics. To improve strength and damage tolerance of 3D-printed alumina-based ceramics, two different approaches have been investigated, focusing on microstructural and architectural design.
(i) High strength alumina based on a multilayer design: A-B-A laminates of (A) alumina and (B) alumina-zirconia materials were additive manufactured using the 2K-lithography- based ceramic manufacturing (LCM) technology. Through mismatching thermal expansions of the different materials, compressive residual stresses were induced into the surface alumina layers during cooling after sintering. A biaxial strength of 1 GPa was obtained, in comparison to 650 MPa on 3D-printed bulk alumina.
(ii) Damage tolerance alumina: Textured alumina ceramics were 3D-printed by applying the method of templated grain growth. Through shear forces, occurring during the printing process, aligned high aspect ratio templates grew due to the dissolution and precipitation of surrounding submicron-sized powder particles. As a result, anisotropic crystallographic properties as well as the morphology of the textured grains led to a biaxial strength of 670 MPa, compared to 570 MPa measured on equiaxed alumina sintered under the same conditions. Additionally, toughening mechanisms as crack deflection, bifurcation and even crack arrest could be observed, leading to an enhanced damage tolerance.
These two strategies may be applied to other 3D-printed ceramic materials and systems of more complex geometry to enhance their structural properties.

High-energy ball milling of bismuth telluride powder to prepare colloidal suspensions for the deposition of thermoelectric thin films by aerosol jet printing

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

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Nowadays energy and pollution crisis raised interest towards alternative ways to harvest energy, like thermoelectric conversion of waste heat into electrical energy (i.e., thermoelectric generators, TEGs). The most interesting aspect is the direct conversion of energy by employing lightweight and quiet devices, without moving parts, therefore at zero green-house gases emissions. Since their thermoelectric properties, the most used and studied materials are bismuth telluride and its alloys (p-type $Bi_xSb_{2-x}Te_3$ and n type $Bi_2Se_{3-x}Te_x$). Aerosol jet printing (AJP) is based on atomization and deposition of a colloidal suspension of a powder with a particles size (d_{50}) lower than 500 nm. As preliminary tests, as-received bismuth telluride (Bi_2Te_3) powder (d_{50} = 2.26 µm) from Sigma Aldrich was ball milled (high energy planetary ball milling Retsch PM400) to reduce the particle size. A submicrometric mean particle size was achieved by dry ball milling, by testing different milling parameters (e.g., higher rotation speed, lower spheres diameter…). The lowest d_{50} value was about 840 nm, using a multiple steps ball milling process. The next steps consisted in the preparation of a stable colloidal suspension based on ball milled bismuth telluride powder in a dispersant based on ethanol, ethylene glycol and glycerol, as formulated by Hollar et al [1,2]. The stable suspensions enabled the deposition of uniform thin films by AJP.

1. Hollar, C. et al. High-Performance Flexible Bismuth Telluride Thin Film from Solution Processed Colloidal Nanoplates. Adv. Mater. Technol. 5, 1–8 (2020).

2. Varghese, T. V. Additive Manufacturing of High Performance Flexible Thermoelectric Generators Using Nanoparticle Inks. Boise State Univ. 1–97 (2019).

Additive manufacturing of calcium carbonate parts through photopolymerization

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Mr. Mateus Mota Morais [1], Prof. Italo Leite de Camargo [1], Prof. Paolo Colombo [2], Prof. Carlos Alberto Fortulan [1]

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Calcium carbonate, a raw material naturally found in coral, limestone, and marble, is an essential feedstock for several applications. Although it is more commonly used in powder form, bulk or structural parts could have exciting applications, such as bone scaffolds, artificial corals, and synthetic rocks. Additive manufacturing, especially vat-photopolymerization, is a promising strategy for producing complex geometries for such purposes. However, two challenges arise: 1) Producing a $CaCO_3$ suspension suitable for vat-photopolymerization additive manufacturing with high ceramic solid loading and low viscosity; 2) Sintering the $CaCO_3$ without causing its thermal decomposition into CaO above 600°C. The first challenge was addressed by following a systematic formulation strategy for the ceramic suspension. First, the photocurable monomer was selected considering the viscosity and curing thickness of mixtures containing different percentages of 2-Hydroxyethyl methacrylate and poly(ethylene glycol) diacrylate 250. Then, eight dispersants were compared, and the one which resulted in the lowest suspension viscosity was selected. Finally, the volumetric loading of the calcium carbonate suspension was increased until a compromise with the viscosity and printability was reached. Preliminary sintering tests at 850°C were carried out in a controlled atmosphere, and the resultant crystalline phases were evaluated using X-ray diffraction. As a result, a printable calcium carbonate suspension was obtained with 35 vol% of $CaCO_3$ and low viscosity (282 mPa.s at $30s^{-1}$). The resin was successfully used to print scaffolds and artificial corals using an ordinary digital light processing (DLP) 3D printer. The developed sintering method revealed that it is possible to remove organics and sinter CaCO3 without thermal decomposition by using the proper atmosphere. The presented results are an important advance for fabricating calcium carbonate parts with complex geometries that can be used as bone scaffolds, artificial rocks, and other applications.

Quality-by-design driven development and up-scaling of a medical device for the guided regeneration of Bone Defects

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

Dr. Mathilde Maillard [1], Prof. Paola Aprile [1], Prof. Rachida Aid [1], Prof. Soraya Lanouar [1], Prof. Laurent Bidault [2], Prof. Teresa Simon-Yarza [1], Prof. Didier Letourneur [1]

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Guided Bone Regeneration (GBR) therapy has proven successful in repairing large bone defects for both dental and orthopedic applications. Collagen-based medical devices (MDs) are often used for GBR application however, their fast degradation rate can jeopardize the clinical outcome. Therefore, the aim of this work was to develop and up-scale the production of an alternative to collagen-based MDs for GBR application by integrating the QbD strategy at the early developmental stage of the product design.

The MD was produced by adapting a published protocol. The mechanical properties were evaluated by rheometry (Discovery HR-2) and nanoindentation (Piuma). Crosslinker quantification and spatial distribution was studied by an in-house biochemical assay. For *in vitro* biocompatibility and efficacy testing, murine and human primary fibroblasts were used to assess material toxicity (Live/Dead, CCK-8 and LDH assay) and cell penetration (immunolocalization and DNA quantification (PicoGreen)). Material degradation was assessed *in vitro* by enzymatic digestion and *in vivo* by subcutaneous implantation in mice. Gamma sterilization was performed by IONISOS. Statistical analyses were performed using GraphPad Prism software and results, are presented as mean ±SD. A level of $p<0.05$ was considered significant.

In order to facilitate the design and up-scaling of our MD, it was necessary to apply a risk assessment cycle. As such, critical material attributes (CMA) and process parameters (CPP) were initially identified accordingly to their influence on the MD's critical quality attributes (CQA).

Crosslinking the material at 50°C led to a slower degradation time *in vivo* which was correlated to higher mechanical properties. Although *in vitro* the gamma irradiated MD degraded faster than the UV sterilized, *in vivo* it showed a comparable performance to a commercial gold standard (collagen-based MD, data not shown).

Linking rheology and mechanical properties of dense ceramic for extrusion-based additive manufacturing

Thursday, 10th November - 10:45: Poster session II and Coffee break - Poster

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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 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, agglomerates and bubbles should be removed and a 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. 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 particular with a work on the optimum amount of dispersant and gelling agent, and an optimization of mixing sequence.

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 SEM and X-Ray Tomography at each stage of the process (after printing, debinding and sintering). It is very important to optimize printing parameters and rheological properties at the same time because cannot print all geometries with one rheology. These studies will allow to better understand links between printing parameters, rheological properties and mechanical properties.

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