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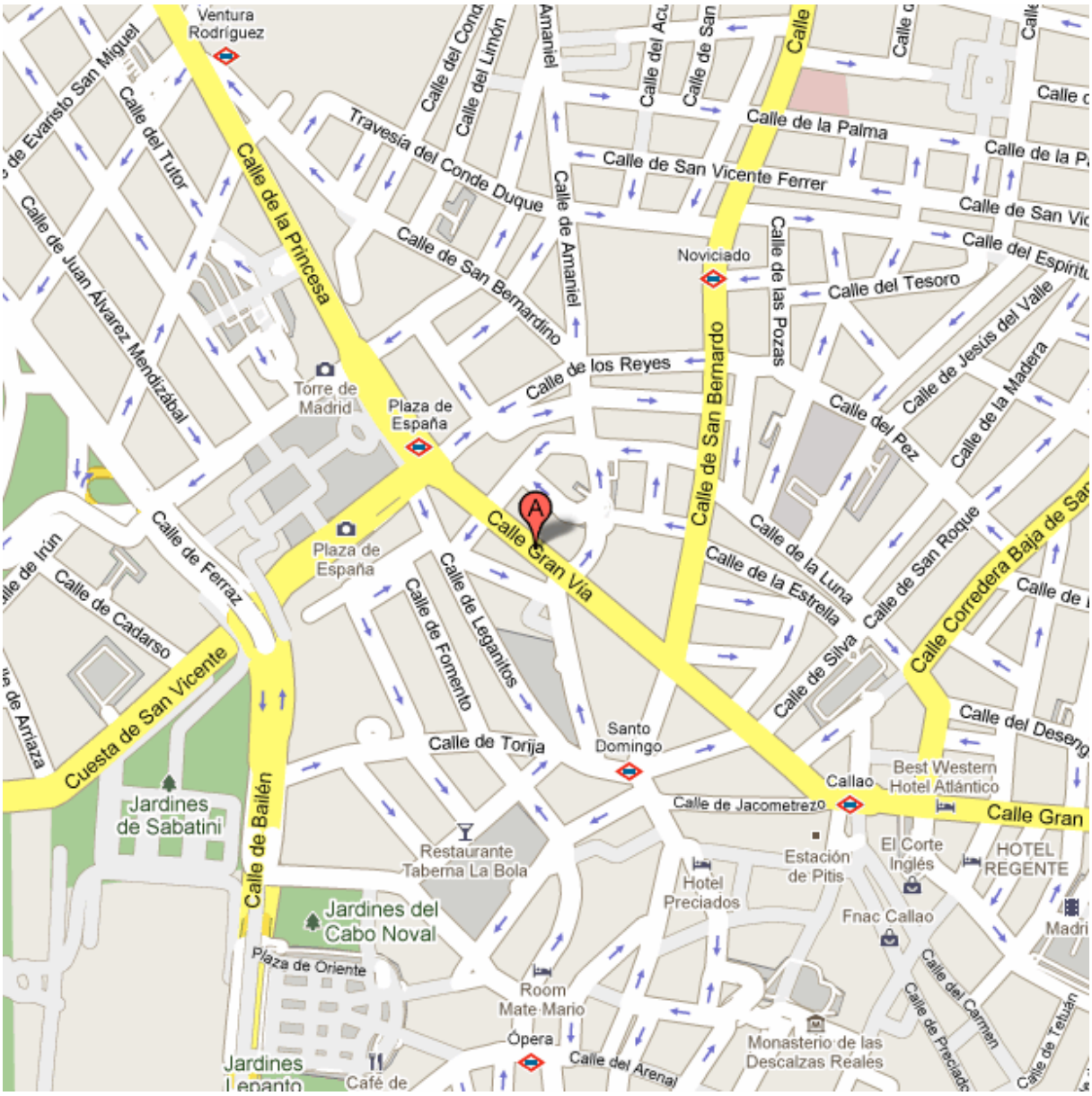

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PROGRAM AT A GLANCE

SUNDAY 15th November	
16:00 - 18:00 h	Registration at Conference site
18:00 - 19:00 h	Opening session and Plenary Lecture (PL)
19:00 - 21:00 h	Welcome Cocktail
MONDAY 16th November	
08:30 - 11:00	Session 1 (S1) Chair persons: J. Halloran & P.J. Sánchez-Soto
11:00 - 11:30 h	Coffee Break
11:30 - 13:30 h	Session 2 (S2) Chair persons: F. Rossignol & M.I. Nieto
13:30 - 15:00 h	Lunch
15:00 - 17:30 h	Invited Lecture (IL4) and Poster Session-1 (P1)
17:00 - 17:15 h	Coffee Break
17:15 - 19:00 h	Student Contest (SC1) Chair persons: A. Segadães &. C. Baudín
TUESDAY 17th November	
08:30 - 11:00	Session 3 (S3) Chair persons: J.P. Eraw &. I. Santacruz
11:00 - 11:30	Coffee Break
11:30 - 13:30	Session 4 (S4) Chair persons: P. Colombo &. M.T. Colomer
15:00 - 17:00	Session 5 (S5) Chair persons: G.L. Messing &. A.J. Sánchez-Herencia
17:00 - 17:15	Coffee Break
17:15 - 19:00	Student Contest (SC2) Chair persons: C. Galassi &. C. Baudín
21:00 - 23:30	Conference Dinner Restaurante El Espejo.

WEDNESDAY 18th November	
08:30 - 11:00	Session 6 (S6) Chair persons: K. Uematsu &. E. Sánchez-Vilches
11:00 - 11:30	Coffee Break
11:30 - 13:30	Session 7 (S7) Chair persons: J. Heinrich &. A.C. Caballero
13:30 - 15:00	Lunch
15:00 - 17:15	Session 8 (S8) Chair persons: A. Boccaccini &. B. Ferrari
17:15 - 18:45	Poster Session-2 (P2)
18:45 - 20:15	Closure and Cocktail

A complete final programme is located at the end of the this book

Sunday 15th November.

Plenary Lecture

COLLOIDAL APPROACH TO RAPID SHAPE FORMING

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Approximately 20 years ago, the whitewares industry made a major advance in the automated forming of large, complex shapes by filter pressing. Plaster molds were abandoned. Clay, small particles of hydrous aluminum silicate minerals, such as kaolin, is the common ingredient, when mixed with water, that imparts plasticity to various whiteware compositions that include fillers (e.g., quartz, alumina, etc.) and high temperature fluxes (e.g., feldspar). With the simple hypothesis that the plastic phenomenon imparted by the clay is primarily due to the attractive forces between the small clay particles, researchers have demonstrated that interparticle forces can be manipulated to produce clay-like behavior and new shape forming technologies to many advanced ceramic powders.

Without repulsive shrouds, particles attract one another to from touching, strongly cohesive networks because of their pervasive, attractive van der Waals forces. The attractive van der Waals forces can be either fully or partially overcome by 'shrouding' the particles with either ion clouds or molecular brushes to produce either fully repulsive or weakly attractive particle networks. The separation distance needed to produce significant repulsion can be controlled by the 'thickness' of the shroud. When the repulsive interaction is made to occur at very short separation distances, the van der Waals force first causes the particle to be attractive, and then, repulsive before the particles touch. For this case, the particles sit in a potential well and form a weakly attractive, but non-touching network. Because forces are required to pull apart the attractive particles, these particle networks have a yield stress that is required for shape forming. The yield stress will depend on the magnitude of the interparticle force, and the number of particles per unit volume. Rheological methods have been developed to measure the 'strength' of a particle network. These methods are not only used to characterize the interparticle forces, but also to judge the shape forming ability of a consolidated slurry.

With these basic ideas, a new shape forming method has been developed called Colloidal IsoPressing. By developing the 'just-right' interparticle force, low volume fraction slurries can be filter pressed to remove much of the water. After filter pressing, the consolidated body is liquefied via shear-rate thinning, and extruded into a rubber mold. The filled mold is rapidly isopressed to remove a small fraction of water to produce a fully saturated, elastic powder compact that retains the shape of the rubber mold. Because the powder was consolidated via isopressing at pressures larger than a capillary pressure developed during drying, the body will not shrink during drying. Thus, it can be directly placed in a furnace and desified without concern of cracking during drying, prior to densification. Filter presses and isopresses are common capital equipment in a modern whiteware factory. Knowing how to manipulate interparticle forces in advanced ceramic powders enables Colloidal IsoPressing, and thus, the rapid shape forming of reliable ceramic components for advanced applications.

Invited Lectures and Session 1

Chair persons: J. Halloran & P.J. Sanchez-Soto

PROCESSING AND PROPERTIES OF NANOSTRUCTURED YSZ CERAMICS

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The processing of yttria partially stabilised zirconia (YSZ) nanopowders into components has been investigated via a series of research projects, each focusing on a different stage of the manufacturing route. The processing work described will focus mainly on the slip casting and die pressing of green bodies with densities as high as 55% of theoretical. For slip casting, it has been possible to concentrate the as-received, aqueous nanosuspension from its original solids content of ~5 vol% up to ~30 vol% without the viscosity exceeding approximately 0.2 Pa s. This and the controlled drying of the slip cast bodies has permitted components measuring up to 60 mm in diameter to be formed without cracking. However, processing is of necessity quite slow and hence effort has also been directed towards learning how to process the same nanosuspension into green bodies via die pressing. This has involved spray freeze drying the concentrated suspension to form flowable granules; the incorporation of 1 – 2% of freon yields granules that also crush readily at industry-standard pressures. Both types of green body can be sintered to >99.5% of theoretical density whilst controlling the final grain size via use of a 2 stage sintering technique. The use of hybrid microwave – conventional (radiant) heating yields final average grain sizes as fine as ~65 nm, whilst purely radiant heating generally yields a coarser microstructure with average grain sizes up to ~200 nm (0.2 μm) in size.

The second half of the presentation will focus on the resulting properties of the bodies; this will include not only measurements of strength, toughness and wear resistance as a function of both yttria content and grain size, but also an examination of the hydrothermal resistance of these materials. With respect to the latter, it is known that submicron 3 mol% yttria partially stabilised zirconia is readily attacked by water; it survives < 1 hour at 250^{o}C and 4 bar pressure in the presenceof moisture before being converted back to powder via the tetragonal to monoclinic transformation. In contrast, the nanostructured version of the same material can survive 2 full weeks under the same conditions with no indication of any transformation at all from the tetragonal to monoclinic phase. It is also hoped to be able to provide results of the ionic conductivity of these materials, also as a function of yttria content and grain size; these measurements have started and should be finished prior to the conference.

Acknowledgement:
The authors gratefully acknowledge the support of the EPSRC and TSB in the UK as well as that of MEL Chemicals Ltd, Dynamic Ceramic Ltd, CERAM Research and RRFCS Ltd.

MONO- AND OLIGOSACCHARIDES IN DEFLOCCULATION PROCESS OF NANOCERAMIC POWDERS

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The results of studies on application of selected mono-, di- and oligosaccharides in the deflocculation process of nanometric alumina and zirconia slurries are presented. In the research nanometric alumina powder of average grain size 47 nm and specific surface area measured by BET method S_{BET} = 35 m^2/g was used. In the research of nanometric zirconia powders, two kinds of powders were used: average grain size 17 and 44 nm with specific surface area measured by BET method S_{BET} = 35 m^2/g and 65 m^2/g respectively. To explain the influence of the individual hydroxyl groups of saccharide on the adsorption process on the n-Al_2O_3 and n-ZrO_2 surface and the reduction process of the particle agglomeration were performed. The influence of D-fructose, D-glucose, L-sorbose; and disaccharides: maltose and saccharose on nanometric alumina and zirconia slurry have been carried out. The results obtained with mono- and disaccharides have been compared with the results on inulin. Inulin is oligosaccharide which consists of fructose units connected in chain (about 12 units) and ended by a glucose molecule from one side. It has been found that not only the amount of hydroxyl groups but also their orientation in saccharides molecule and their orientation to the surface of nanopowder are the main factors which decide about the amount of water connected with the nanometric alumina and zirconia particles and directly influenced oh the rheological properties of alumina slurry. In comparison to alumina nanopowder where the viscosity decrease with an increasing amount of saccharides addition, rheological investigations demonstrated that monosaccharides increase the viscosity of ceramic slurries made of nano-ZrO_2 with an average particle size of 44 nm (Inframat Advances Materials) Explanation of such unexpected phenomenon requires some additional investigations.

Acknowledgement:
The work was supported by Warsaw University of Technology

CONTROL OF MICROSTRUCTURE IN CERAMICS BY SLIP CASTING UNDER A STRONG MAGNETIC FIELD

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Tailoring the crystallographic orientation in ceramics is one way of effectively improving their properties, such as electrical, piezoelectric and mechanical properties. Many studies have been reported for the production of textured ceramics, such as by the Templated Grain Growth method and hot forging.

On the other hand, recently, superconducting magnet technologies have been developed and used for various applications, and we reported that the successful control of the development of a textured microstructure in diamagnetic ceramics, such as alumina, titania, α-silicon carbide and aluminum nitride, was achieved by a colloidal processing in a strong magnetic field. When we use the crystals with asymmetric unit cells, these crystals has anisotropic susceptibility. The ceramic particles become rotated to an angle that minimizes the system energy by a magnetic torque generated from the interaction between the magnetic anisotropy and the applied magnetic field. However, it is generally difficult to apply a magnetic field effectively in order to rotate fine diamagnetic particles, even if a strong magnetic field is used, since this anisotropic susceptibility is extremely small in diamagnetic ceramics. Accordingly, the dispersion of particles in a suspension is necessary for the effective utilization of the magnetic field, because a strong attractive interaction between particles in a suspension prevents each particle from rotating under the application of a magnetic field. In order to rotate the particles, colloidal processing was used in this study because this processing is a powerful technique for controlling the stability of particles in a suspension. It makes use of repulsive surface forces in order to avoid agglomeration. And the other conditions in the consolidation under the magnetic field are also important for controlling the orientation. Furthermore, the crystallographic orientation is seemed to be inseparably related to the microstructure after sintering.

In this presentation, we report the relationship between the development of the crystallographic orientation during consolidation and the microstructure in ceramics prepared by consolidation of colloidal suspensions in a strong magnetic field.

Acknowledgement:
This study was partially supported by the Budget for Nuclear Research from the MEXT, Japan and also Grant-in-Aid for Scientific Research from the JSPS of Japan. And the authors thank the World Premier International Research Center Initiative (WPI Initiative) on Materials Nanoarchitectonics, MEXT, Japan.

CRITICAL PARTICLE SIZE FOR SHAPING DENSE CERAMIC BODIES BY SLIP CASTING

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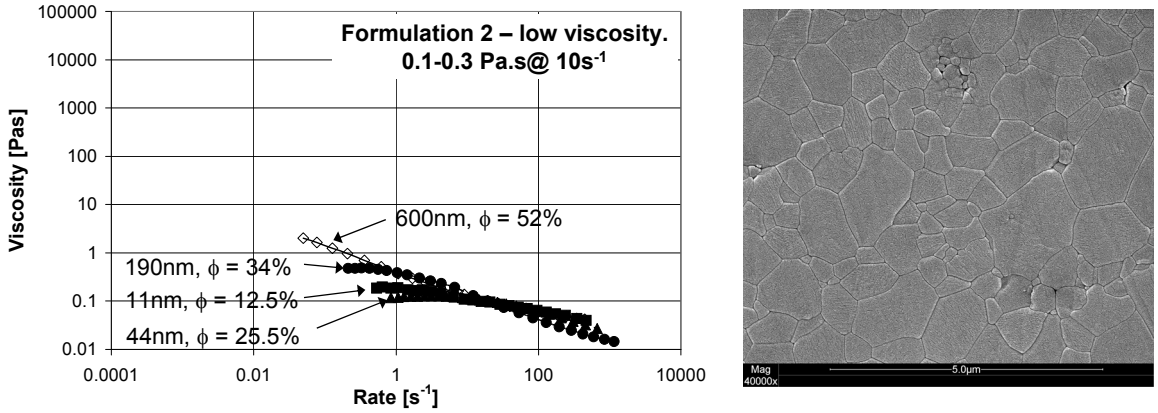
The use of nanoparticles for different applications has been extensively studied using a wide range of techniques and approaches. Ceramic nanopowders are expected to produce promising materials in terms of strength, hardness and wear if the appropriate microstructure may be obtained after sintering. Unfortunately, the handling and manipulation of nanopowders is often very difficult. So the shaping method selection will be restricted to those size particles which can be processed adequately. Importantly, suspensions must be produced which have low viscosity for good shaping and also produce high density green bodies for good sintering (at low temperature to avoid excessive grain growth). The work presented is a study of the effect of the nanometric-ranged particle size of the starting powder on a simple and well-established shaping method, slip-casting. Several alumina suspensions with the same viscosity (but different solid content suspensions) are produced using different particle sizes (11, 44, 190 and 600 nm). They were shaped into dense bodies by slip casting and sintered. The green and sintered densities ranged between 30-67% and 63-99% of the theoretical value, respectively. These values, together with the microstructure observations reveal the effect of the solid content and size of the ceramic powders. The result is an optimal particle size of alumina particles that allows the preparation of concentrated suspensions with low viscosity and sintered bodies with density close to the theoretical value.

a) b)



a) Viscosity curves for different alumina particle size suspensions; b) SEM image of the sintered and polished surface of the dense body obtained using the 190 nm alumina powder.

HYBRIDIZATION OF TEXTURE INDUCING PROCESSES

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Microstructural texture is defined as orientational distribution of crystallites in a polycrystalline material. There are various processes, used actively in the industry and research, which induce texture in the material. It is possible to classify these processes in several ways. One way to classify them is based on the type of force or gradient which is used to create texture; this gradient can be either mechanical or magnetic. In case of ceramic powder processing mechanical methods depend almost always on the anisometric particles. Oriented consolidation of anisometric particles and templated grain growth can be given as examples of such methods. Since industry standard methods such as extrusion, uniaxial pressing and tape casting are employed for production of textured materials with these techniques they are quite suitable for mass production. One drawback of mechanical methods is the dependence of texture induction on anisometric particles. This is quite an important issue due to lack of efficient production of such particles. On the other hand, in methods where magnetic forces are applied on colloidal powder systems for orientation of particles with respect to the direction of applied magnetic field, presence of anisometric particles is not obligatory. These methods exploit the anisotropic magnetic susceptibility of crystallites. However, for some crystal systems may not exhibit magnetic susceptibility anisotropy, and hence it is not possible to orient their crystallites in a certain direction by magnetic methods. As a result both systems have their advantages and disadvantages.

One important problem in textured materials is the anisotropic shrinkage which introduces residual stresses and complicates dimensional control. Anisotropic shrinkage may develop in both mechanical and magnetic methods either due several reasons such as presence of anisometric templates which are larger than matrix powder, networking of these rigid inclusions, processing defects such as voids and heterogeneity or sintering anisotropy caused by different growth rate of different crystal planes (grain boundary energy anisotropy) during sintering.

Several engineering problems may be overcome by combining different approaches. This may also be valid when different texture inducing processes are combined to eliminate the need for high amounts of anisometric particles and the effect of anisotropic shrinkage. For this purpose 0, 2, 5 and 10 wt % rod like anisometric ZnO particles were added to fine ZnO powder to prepare colloidal suspensions. These suspensions were than slip cast under 12 T rotating magnetic field. The green body is cut in directions parallel and perpendicular to the slip casting direction and thermo mechanical analysis (TMA) was employed on these specimens to study sintering behavior. Sintered c-axis specimens were analyzed by x-ray diffraction for qualitative texture analysis and electron backscatter diffraction (EBSD) method for quantitative texture analysis. For quantitative analysis data obtained from EBSD pole figures were fit with March-Dollase equation.

TMA results showed that anisotropic shrinkage was existent even in the sample without rods and texture. This is attributed to processing defects such as density gradients. Sample which is slip cast without rods also exhibited texture and anisotropic shrinkage. Different amounts of templates resulted in different sintering behavior. This showed that by controlling rod-like particle content anisotropic shrinkage behavior can be controlled. XRD and EBSD results indicate that the presence of anisometric particles cause a reduction in the strength and quality of crystallographic orientation under magnetic field.

Acknowledgement:
This work is supported by Turkish Academy of Sciences (TÜBA) via Outstanding Young Investigator Award and National Institute for Materials Science (NIMS) of Japan via NIMS internship program.

POWDER PROCESSING FOR TRANSPARENT POLYCRYSTALLINE ALUMINA

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Production of transparent polycrystalline alumina (PCA) for sapphire replacement has been a hot topic over the last decade. Due to the high sensitivity of the optical properties on defects (i.e. cracks, pores inhomogeneous/excessive grain growth) transparent PCA is prone to powder and processing variations.

The influence of green body fabrication by uniaxial, isostatic, filter-pressing, slip- and gelcasting methods (e.g. homogeneity, porosity, defects) on the sintering behavior of alumina has already previously been reported from other studies[1-3]. Filter-pressing has always proven to give good performance in terms of both green body characteristics and industrial viability. Eventual processing issues during filter-pressing related to the reduction of the powder sizes (e.g. pressure, time, segregation) can be solved by different flocculation routes[4, 5].

In this study the influence of the green body fabrication method on the real in-line transmittance (RIT) of PCA sintered in low pressure atmosphere followed by post-HIP is being addressed. Focus is put on filter-pressing and powder granulation techniques with and without additives such as dispersants, binders and flocculation agents. The results are compared to the RIT of PCA obtained by Spark-Plasma-Sintering (SPS) to highlight the main advantages of both sintering procedures.

Acknowledgement:
The authors want to thank the Swiss National Science Foundation for funding the research (n° 200021-122288).

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DEVELOPMENT OF GRAIN ORIENTED TUNGSTEN BRONZE CERAMICS WITH MAGNETIC FIELD

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Fine particles of tungsten bronze were highly dispersed in a distilled water with a dispersant and placed in a rotating-magnetic field to direct their crystalline axes into a specified direction. Particle oriented green compact formed by removing the water was sintered to obtain ceramics of oriented crystalline axes. Orientation was found to improve the piezo-electricity and other properties, significantly. Fabrication method, properties and microstructure will be presented in detail.

REALISATION OF CERAMIC HOLLOW FIBER GAS SEPARATION MEMBRANES BY SPINNING WITH PHASE INVERSION

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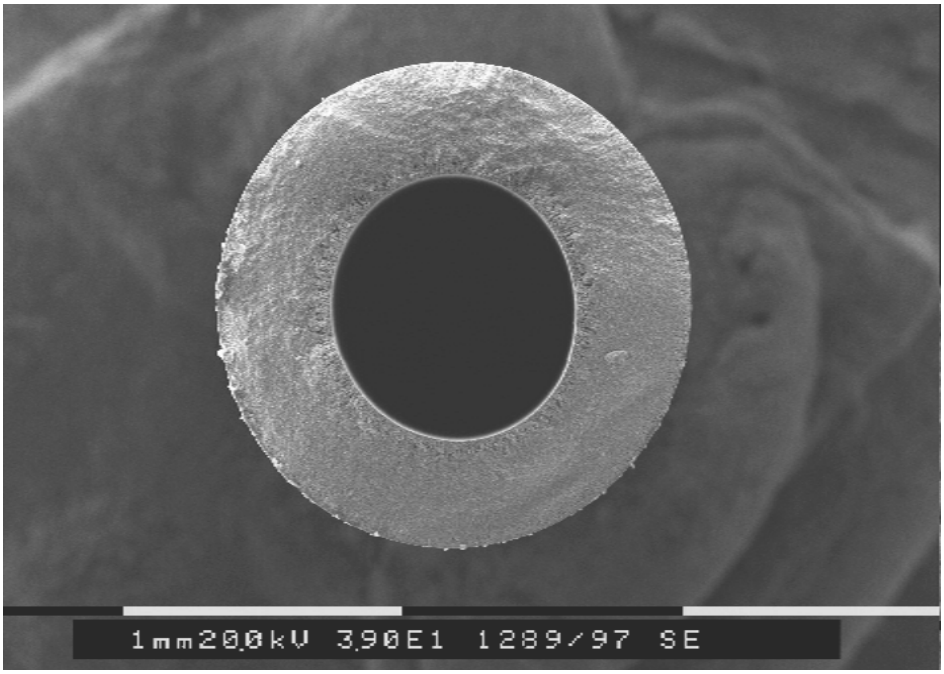
Technologies for high-purity oxygen separation from air and partial oxidation of light hydrocarbons using dense ceramic membranes with mixed oxygen-ionic and electronic conductivity have significant potential for the sustainable production of energy. Shaping of membranes in the form of hollow fibers offers interesting advantages over other membrane designs, eg the application of the hollow fibers in membrane module allows to strongly enhance the ratio of the membrane surface area with respect to the volume of the module. In this light, the MEMBRAIN-project considers amongst others ceramic hollow fiber gas separation membranes.

$Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ (BSCF) oxide ceramic hollow fibers were produced via the spinning technique, based on phase inversion. The technique of spinning combined with phase inversion is commonly used for the fabrication of polymeric membranes, but it has been successfully adapted to dense ceramic hollow fiber membranes by using a starting suspension loaded with a certain fraction of ceramic powder. The composition of solvent/non-solvent system, rheology of the spinning suspension, flow rate of both suspension and bore liquid and further parameters were investigated and adapted in order to obtain dense, crack-free and gas-tight LSCF and BSCF hollow fibers.



Cross-section of a hollow fiber

Invited Lecture and Session 2

Chair persons: F. Rossignol & M.I. Nieto

ELABORATION OF TAILORED MILLIMETRIC POROUS CERAMIC SPHERES BY COLLOIDAL WAY

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Based on diluted systems, a new process of powder granulation is proposed to elaborate ceramic spheres directly in suspension. Powder agglomeration takes place via the electrostatic interactions occurring between the particles. The first step consists to form “primary agglomerates” (2-3 μm) which are then mixed under a continuous rotational movement. If locally opposite charges exist, their coalescence occurs to lead to “secondary agglomerates” the size of which is about one millimetre, nearly this of the final sphere. Finally, the mutual friction of the objects makes them round and smoothens the surface. Formulations developed for this granulation method are based on aqueous oxide suspensions. The phenomena occurring during this process take place when the oxide surface carries both positive and negative charges, and can be explained on simple basis involving surface chemistry.

Several raw materials (Al_2O_3, TiO_2, ZrO_2) can be granulated by this method. But formulations containing latex spheres and mineral grains can be also agglomerated by addition of nanofilaments carrying a high density of positive charges. By selecting the suitable ratios for each raw material, the structure of primary agglomerates consists of core (latex)/ shell (mineral monolayer) type.

The control of the surface chemistry of powders, which in turn determines the details of interactions between the solid phases, allow to produce cohesive objects with a spherical form. After calcination, the spheres obtained exhibit a hierarchical multiscale porosity (fig.1). The process can be extended to produce catalysts, filters…

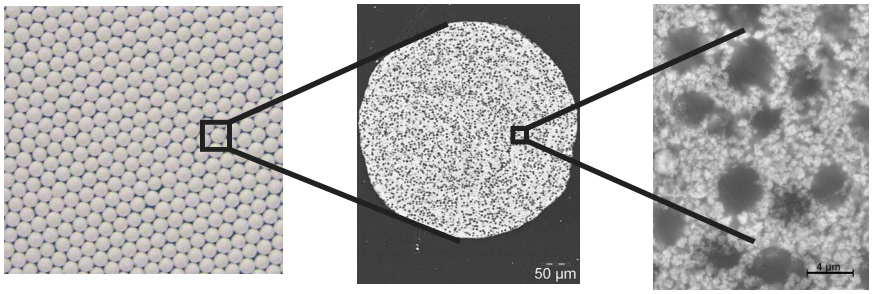

Figure 1. From a mixed colloidal suspension of organic (latex) and mineral particles, spherical millimetric objects can be produced by granulation.

PARTICLE PACKING IN PARAFFIN-WAX SUSPENSIONS USED FOR LPIM

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Low-pressure injection moulding (LPIM) is used for forming of near-net shaped ceramic green parts with complex geometries. Its success relies primarily on the rheological properties of the ceramic-powder paraffin-wax suspension. It is desirable that the slurry contains a high loading of homogeneously dispersed particles in a liquid carrier to ensure suitable moulding characteristics. These slurries are weakly flocculated because the surfactant, which is usually stearic acid, is effective only in reducing the van der Waals attraction but is insufficient to provide complete stabilization. When the overall interaction potential in the suspension is attractive, a continuous network is formed above a certain volume fraction of solids and a certain shear stress is needed to overcome the interparticle forces in order to induce flow. Since the feedstocks used in LPIM are above critical volume fraction, the attractive interpaticle forces play a crucial role in obtaining high solids loading and appropriate rheological characteristics.

In the present study we focus on the shear rate dependent behavior of the ceramic-powder paraffin-wax slurries and their effect on the packing of particles during LPIM forming. The effect of particle size of the powders, the solids loading and the chain length of surfactants on the shear rate dependent behavior of paraffin-wax slurries for several oxide ceramic materials will be addressed.

POWDER-BINDER-SEPARATION IN INJECTION MOULDED GREEN PARTS

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For powder injection moulding (PIM) the ceramic powder is mixed with a thermoplastic binder system to achieve an injectable feedstock. During the mould filling process shear forces act on the blend that might cause separations of powder particles and binder. In this way polymer films are created at the mould surface and at internal interfaces which induce microstructural defects in the debinded part. In particular for multi-component parts this effect is critical since binder films in the joining zone weaken the bonding strength between the two components that might even lead to delamination.

To detect binder separations within the injection moulded bulk material and at joining zones of 2-component parts the microstructure of green samples was studied. Since conventional mircosecting techniques like grinding and polishing modify the original structure, e.g. when particles are pulled out of the matrix and binder smears onto the surface, a special ceramographic method for the preparation of cross sections was applied. This approach bases on broad ion beam techniques and enables the simultaneous polishing of hard ceramic particles and soft polymer molecules without destroying the structure. In the analyzed samples binder accumulations were found along flow lines, at weld lines, at boundaries of dead water regions and at the interface of 2-component parts.

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AQUEOUS DISPERSION OF TUNGSTEN POWDER FOR INKJET PRINTING PROCESS

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Inkjet printing process is very promising for the production of 3D fine-scale multifunctional devices.

Thanks to its ease of use and flexibility (DAO), inkjet printing reveals to be an economical and powerful process, all the more as the use of a multi-nozzle system enables to design 3D multifunctional microelectronic devices integrating connection network, by depositing successively different suspensions of various nature and composition.

This technique can in particular be applied to the manufacturing of interconnection multilayer HTCC modules (High Temperature Co-fired Ceramic), by depositing conductive lines on insulating ceramic layers. Furthermore, such a process enables to create metallic vias to connect two conductive levels.

The present work focuses on the elaboration of specific aqueous tungsten inks compatible with inkjet printing process, concerning the grain size (<1µm), the surface tension, the viscosity (only few mPa.s) and the stability over time.

It reveals to be a challenge to disperse tungsten in water considering its metallic nature, its high density (18-20g/cm3), and the coarse grain size distribution of the commercial powder: preliminary steps of aqueous dispersion and milling are thus necessary. At this stage, inorganic additives as fine alumina were added to the primary W suspensions in order to guarantee the W inks adhesion on the ceramic substrate and improve the shrinkage compatibility, without significant decrease of the electrical conductivity.

First, various dispersants were screened to select the most efficient in regards to the physic-chemical stability of W powder suspension, then, the optimum dispersant concentration was determined through Zeta potential, sedimentation rate, and viscosity measurements.

The optimization of the mixed W-Alumina suspensions preparation via milling was carried out thanks to a fractional factorial design of experiments. This latter enables to compare the effect of each factor i.e. duration, vol% powder, inorganic ratio, and dispersant concentration on the grain size distribution, the stability, the surface charge, as well as the stability of the milled suspensions, and also leads to the optimal conditions to use to prepare further inks.

To formulate inks compatible with IJP process, organic additives are necessary, to adjust the viscosity, the surface tension as well as the drying behavior and the green mechanical strength of the deposit. At this step, it is important to well control the powder dispersion state as well as physical parameters of aqueous medium in order to these additional components not destabilize the dispersion, and that is why preliminary tests were carried out to select compatible organic additives. To study the main effect of each component on the stability, the physical characteristics (pH, grain size, viscosity, surface tension) and the ability to ejection, a design of experiments is in progress, considering as factors the nature and concentration of each constituent. This could also highlight potential unfavorable interactions between some ink components.

SOLID FREEFORM FABRICATION OF AL_2O_3/TIO_2 GRADIENT MATERIALS

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The fabrication of Al_2O_3/TiO_2 laminates with multilayer gradient structures by Lamination of Object Manufacturing (LOM) was investigated. The influence of the different laminate configurations (layer stacking of Al_2O_3 to TiO_2) on the mechanical and thermal properties of the final materials was verified. Green tapes of Al_2O_3 and TiO_2 were produced by aqueous tape casting. Optimised parameters already published in previous work [1, 2] were applied to cut and laminate the green tapes. A cw-CO_2-laser LOM system working at a line energy of 336 J/m was used. After lamination, a one step heat treatment up to 1300°C was carried out for debinding and sintering of the laminates. The laminate shrinkage was measured in two different directions e.g. along the layer direction (in-plane) and perpendicular to the layer (out of plane). Laminates up to 20 layers in complex geometries [Fig. 1] were also fabricated.

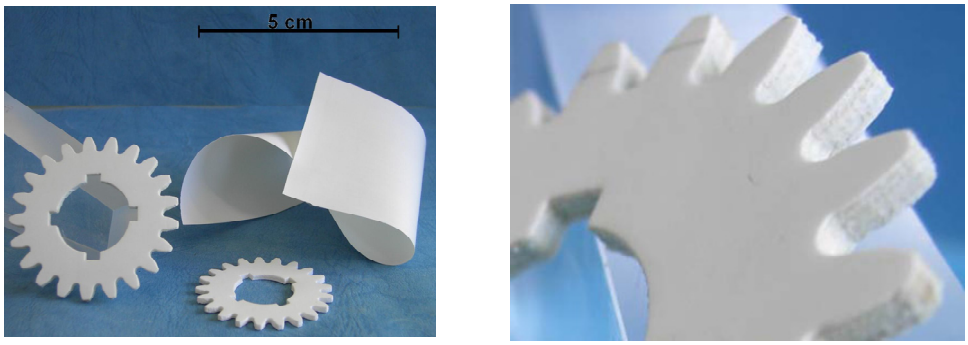

Fig 1. Ceramic laminates produced by LOM.

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NEW FRONTIERS IN CERAMIC MICRO SHAPING TECHNOLOGIES

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A novel process capable of producing simple and microstructured ceramic and polymer/ceramic nanocomposite fibres has been developed recently. This broad technology is based on continuous “on the fly” curing of UV curable colloidal ceramic dispersions structured through a micro-extrusion die. Due to the decoupling of the shaping and setting viscosities, the viscosity of the extrusion feedstock can be orders of magnitude lower than in conventional extrusion. This enables fabrication of continuous fibres with correspondingly smaller dimensions. Due to the special feedstock and wide choice of monomers, the elasticity of the nanocomposite fibres can be tailored for various applications including woven products. Other novel shapes such as microspheres and cellular articles produced from the colloidal ceramic dispersions via rapid prototyping techniques are also discussed.

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De Hazan Y., Heinecke J., Weber A., Graule T., High Solids Loading Ceramic Colloidal Dispersions in UV Curable Media via Comb-polyelectrolyte Surfactants, J. Colloid Interface Sci, accepted for publication (May 2009)

PHOTOPOLYMERIZATION OF CERAMIC SUSPENSIONS

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The photopolymerization of suspensions of ceramic powders in monomer solutions is the fundamental step in several rapid prototyping forming techniques based on photocuring. Exposure to an UV dose of energy E causes the monomer to be polymerized to a cure depth C_d, which are related by $C_d = D_p \ln(E/E_c)$, where D_p is the resin sensitivity and E_c is the critical energy. We present simple models for resin sensitivity D_p in terms of the attenuation of the UV beam by absorption and scattering, and demonstrate the applicability of the model with experimental data for UV lamps, a UV laser, photorheology and photo DSC, and direct photospectrometry. We also present a model for the critical energy E_c in terms of the relative number of photogenerated radicals vs. the concentration of inhibitors.

Invited Lecture and Posters Session 1

INNOVATIVE PRODUCTION PROCESSES FOR CERAMIC MEMS/NEMS

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The fabrication of micro components made from ceramic materials is becoming more and more important in various branches of technology because of their outstanding chemical stability. Different replication methods like low or high pressure ceramic injection moulding of ceramic feedstocks have been established in the industrial process. In Micro and Nano-Electromechanical Systems (MEMS/NEMS) the combination and interaction of different materials and the accurate mounting of several parts is of major importance. Routes to shape ceramic materials are needed that fit common MEMS-processes like different lithographic methods or printing techniques.

The manufacturing of miniaturized ceramic components using the powder route, i.e. pressing or micro powder injection moulding is limited, however. In general, producing original moulds needed to shape micro parts is very complex and therefore expensive.

The direct shaping of sintered ceramics using mechanical methods like milling, cutting or grinding is critical since the machining tools suffer from the hardness of the material. Shaping micro parts in the green state before sintering is of no avail when it comes to smallest features because it is difficult to produce tools with dimensions smaller then 0.1 mm. Moreover, strong efforts are necessary in order to accomplish the accuracy requirements of Microsystem Technologies.

Two main approaches to the fabrication of ceramic MEMS/NEMS are presented in this review. In so called nano-imprint- (NIL) or soft-lithography methods the microstructures are initially prepared on a duroplastic master for derivative moulding or printing processes. A direct method is the structuring and cross linking of ceramic powder filled polymers using lithographic energy sources like UV, X-ray, E-beam and laser. Amorphous bulk ceramics derived from the thermal pyrolysis of liquid organic precursors, known as polymer-derived ceramics (PDCs) provide interesting material properties. Favourable creep, thermal shock and high oxidation resistance have been reported. The nanostructure of PDCs can be adjusted by tailoring the ceramic precursor polymer and their processing to alter specific properties. For example the formation of nanocarbon during pyrolysis of SiCN precursors allow the use in electronic devices. By using Si-(B)-C-N based PDC the application of MEMS/NEMS in harsh environments with high temperature under oxidizing conditions becomes possible. Smallest features below one micron have already been realized.

Various efficient microfabrication techniques for ceramic MEMS/NEMS are presented. Application in electrical actuators and efficient heaters in harsh environments with high temperature under oxidizing conditions becomes possible. Micro grippers, photonic crystals and micro fluidic components have been tested and are a promising origin to new applications. Smallest features below one micron have already been realized. The methods presented are also applicable to precursor polymers for a variety of functional and structural ceramics. One challenge is to adapt the processes and materials to common MEMS technologies. For direct lithographic applications suited precursor polymers are needed that provide side chains that can be photochemically activated.

CONDUCTING ALUMINA PARTICLES: EFFECT OF IONIC STRENGTH AND pH ON ZETA POTENTIAL

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Technological improvement continuously demands for the development of new materials that exhibit better properties or lower production costs, but most ceramic components are still being produced by powder consolidation and sintering. The final properties required by the particular application are determined by the material's microstructure. The sintering stage is generally concerned with unwanted microstructure changes, such as excessive grain growth and/or shrinkage, and there is a rather limited manoeuvring range for the correction of possible particle packing defects at this stage. Thus, it can be said that the final microstructure is irrevocably established during the powder consolidation stage.

Novel techniques of wet processing of ceramic powders, like freeze and gel casting, temperature induced forming (TIF), direct coagulation casting (DCC), solid freeform fabrication (SFF), rely on the comprehension of properties and stability of concentrated suspensions, namely on how the individual powder particles are spatially arranged within the suspension, for how long that arrangement is kept (i.e. colloidal stability), and how it propagates through the consolidation methods into the green body, after removal of the suspending liquid.

To understand all of the above, it is necessary to start at the particle level and the electrical charges that develop at the particle surface when it is immersed in a polar liquid. Interactions between charged particle pairs can be explained by the DLVO theory, as the balance of the repulsive potential due to the electric charges present in the particle's electrical double layer and the attractive potential due to the ever present long distance van der Waals forces. Such Interactions are mostly controlled by the diffuse layer of the particle's electrical double layer, hence, strongly dependent on the ion concentration in the suspending liquid. In practice, the diffuse layer potential cannot be directly measured and Zeta potential is used instead, given that it can be accessed through electrokinetic measurements. This work was aimed at clarifying the mechanisms of electrical conductivity observed in aqueous suspensions of commercial alumina powders by studying the particles dynamic electrokinetic mobility through measurement of the electrokinetic sonic amplitude (ESA) and relating it to the electrolyte ion concentration and pH.

To separate the particles and electrolyte contributions to the suspension conductivity, aged suspensions were first subjected to dialysis to remove the great majority of alien ions until a very low ionic strength was reached (~0.0005mol/l). The ionic strength was then manipulated by adding KCl and the pH was adjusted with HCl. A calibration curve was constructed. The particles electrical conductivity was calculated using Maxwell's model.

This suspension preparation methodology enabled the identification of three different stages of Zeta potential changes. Initially, for low electrolyte conductivity (K_L<100μS/cm), Zeta potential decreases towards a minimum value. Then, as the electrical conductivity increases, Zeta potential increases, goes through a maximum (K_L=277μS/cm) and decreases again. These observations can be related to the ion adsorption sequence during the establishment of the Helmholtz inner and outer planes due to changes in the electrical conductivity of the alumina particles. The particles maximum conductivity, $K_{P,Max}$, is reached when the Stern layer is complete; electrolyte conductivities higher than the particles maximum conductivity (K_L>$K_{P,Max}$) promote the shrinkage of the diffuse layer and Zeta potential falls exponentially, as reported in the literature. In this way, the effect of co- and counter-ions on the particles dynamic electrokinetic mobility and Zeta potential was evidenced and interpreted.

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CHEMICAL STABILITY OF AQUEOUS/NONAQUEOUS (V,Zr)SiO_4 SUSPENSIONS

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The ceramic pigments with particle size in the nanoscale have a massive potential market, because of their high surface area that provides higher surface coverage, higher number of reflectance points and hence improved scattering. Ceramic pigments do not dissolve in medium due to the deterioration of color intensity and quality. For this reason, the stability of inks and colloidal properties are affected by physico-chemical interactions between pigment particle and solvent should be well known. To achieve this aim, industrially available blue (V,Zr)SiO_4 (V-zircon pigment) pigments' chemical stability in aqueous/nonaqueous suspensions at different pH values (pH=7,9 and 11) was investigated. 5 wt % pigment particles were dispersed in water and/or in diethylene glycol (DEG) in order to specify the effect of the medium. Afterwards, the interaction between pigment particles and medium (water/DEG) was investigated by using inductively coupled plazma optic emission spectrometer (ICP-OES). The ICP-OES results show that cations of (V,Zr)SiO_4 pigments dissolve in aqueous/non aqueous system. In this presentation, dissolution mechanism of pigments will be discussed. In addition, some approaches will be presented to eliminate the dissolution of cations and subsequently to maintain the colour quality.

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IMPROVEMENT OF THE DISPERSION OF THE MWCNT IN A ZIRCONIA MATRIX BY THE ADDITION OF PARTIALLY COATED MWCNT AND COLLOIDAL PROCESSING

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Ceramic materials can show improved properties when carbon nanotubes are well dispersed in the matrix. Colloidal processing offers the possibility of obtaining homogeneous complex samples with well dispersed second phases in the ceramic matrix. In this work, we compare the colloidal behaviour and final properties of as-received multi wall carbon nanotubes (ar-MWCNT) and nanozirconia partially coated MWCNT (pc-MWCNT) in a nanozirconia matrix. pc-MWCNT showed a better wettability than ar-MWCNT in an aqueous medium due to a chemical bonding between the zirconia coating and CNT; this fact facilitates the preparation of more homogeneous aqueous CNT/ceramic suspensions and hence better bodies, where the coated nanotubes resulted to be fully immersed in the ceramic matrix. Finally, sintered samples with higher density, lower grain size, and improved toughness and hardness under the same sintering cycle were obtained

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KINETICS AND SYNTHESIS MECHANISM OF CORDIERITE BY KAOLIN/TALC/ALUMINA MIXTURE IN SLIP CASTED BODIES

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The effects of mechanical activation by intensive ball milling of a kaolin/talc/alumina mixture in stechiometric proportions, time and temperature of treatment on the kinetics and synthesis mechanism of cordierite (*$2MgO.2Al_2O_3.5SiO_2$) have been studied*. Raw materials were initially characterized by chemical and X-ray diffraction (XRD) analyses, measures of specific superficial area, laser-diffraction granolumetric analyses and helium picnometry. Previous work studied the rheological behavior and slip casting performance of 40 vol.% slips of kaolin/talc/alumina formulation with relative weight ratios of 40.0/43.8/16.2 of both milled and unmilled conditions. In this work the study of kinetics and synthesis mechanisms of cordierite formation were carried trough XRD, differential thermal analysis (DTA), dilatometry tests and SEM analyses. The overall mechanism of phase formation was studied as a function of temperature (1000 to 1400 ^oC), thermochemical treatment time (0 to 4 h) and formulation milling time (0 to 45 min). The milling process reduced the formation temperature of cordierite in approximately 50 °C and eliminated a concurrent reaction that produced undesired phase saphirine. This reaction was preferential at lower temperatures for unmilled slips. In order to assure the cordierite formation, the optimum thermochemical treatment conditions were defined as consisting of milling during 45 min followed by subsequent heat-treating at 1280^oC during 1 h.

PRESSURE FILTERING AND DENSIFICATION OF FINE GRAINED MAGNESIUM ALUMINATE SPINEL

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Ceramic materials such as magnesium aluminate spinel is a strong candidate for transparent armours and for special optical applications. However, transparent ceramics require grain sizes of dense microstructures below 500 nm, porosities close to 0,05% and pore sizes below 20 nm. To this end, advanced shaping processes are needed to improve the particle coordination of green bodies and enable a decrease of the sintering temperature. Consolidating powder compacts to a uniform and high particle packing density is a central aspect of ceramic processing. Advanced densification heat treatment technologies are also required to avoid grain growth.

It is the objective of the present investigation to study the influence of pressure filtering variables on the homogeneity of particle coordination of green bodies and consequently on the sintering of fine grained transparent magnesium aluminate spinel. Experiments on the formation of powder compacts from slurries of differing properties and pressure filtered under different conditions are presented. The influence of (1) pressure, (2) solid content of the slurry, (3) slurry viscosity (4) type of solvent and (4) dispersants are discussed. Different particles arrangements are evaluated on their effects on the resulting pore size distribution, the associated sintering densification and grain growth. Densification was performed by a two step process using rate control sintering at lower temperatures to closed porosity and spark plasma spray (SPS) or hot pressing (HP) at final step densification.

A quantitative assessment of the homogeneity of particle coordination in green bodies is achieved by porosity measurements. A quantitative description of the rate at which the particles are deposited from the slips in the pressure filtering process is also given. Uniform and non_ uniform theories of cake growth are checked. Fundamental cake growth properties such as density, uniformity, permeability behaviour and yield curve are also tackled.

Preliminary results show that homogeneity of particle coordination of the green bodies, reduced sintering temperatures and microstructures with low grain growth can be obtained using a controlled pressure filtering process

FORMULATION OF DIELECTRIC INK FOR FABRICATION OF HIGH POWER CERAMIC CAPACITORS BY INK-JET PRINTING PROCESS

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Devices of boarding power electronics are used to convert and manage electrical power with flexibility and effectiveness. There is an increasing demand for applications ranging from a few Watts (food for wandering systems, house automation, car...) up to tens of MW (heavy industry, maritime propulsion, etc). The objective of this study consists on the manufacturing of ceramic capacitors with a colossal capacity in the field of railway transport. Ink-jet printing process is very promising for such a capacitor. Indeed, this technique exhibits the capability to deposit in one stage, via a multi printing-head system, 3D complex architectures integrating dielectric layers and the electric connection network. In this work, the perovskite $CaCu_3Ti_4O_{12}$ (CCTO) has been selected as dielectric material because of its giant dielectric constant. In fact, CCTO sintered pellets exhibit a relative permittivity $\varepsilon_r \approx 1.4 \times 10^5$ with dielectric losses tan $\delta \approx 0.16$ at 1 kHz at room temperature. The aqueous dielectric ink based on CCTO powders should be compatible with ink-jet printing process, in particular in terms of granulometry, rheology, surface tension and stability. The first step of our study has consisted in the adjustment of the dielectric ink formulation. Therefore, a preliminary step of milling has been carried out to disaggregate CCTO powder and to guarantee a maximum diameter of the powder below one micrometer to avoid the blocking of the nozzle (52µm). The efficiency of ammonium salt of polyacrylic acid (PAA-NH_4) as dispersant was demonstrated by measuring the Zeta potential values of CCTO particles (around -31 mV at pH = 9) using an Electrokinetic Sonic Amplitude (ESA) measurement technique. The dispersant ratio was optimized for minimum viscosity and maximum surface charge. The stability of the suspensions was demonstrated via sedimentation tests. An experimental design is in progress to adjust ink formulation in terms of nature and content of the different inorganic additives (binders, surfactant…).

INKJET PRINTING OF FUNCTIONAL MATERIALS FOR CERAMIC ELECTRONIC APPLICATIONS

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Inkjet printing (IJP) process is a cost efficient prototyping process which allows ideal resolution for material deposition without any contact with the substrate. New emerging applications for inkjet printing, especially in ceramic electronic industry, need both ceramic ($BaTiO_3$, TiO_2, Al_2O_3, PZT) as well as precious metals inks (Ag, Pd, Au, Pt). For these applications, reliability of drop placement is essential to print material layers with a good homogeneity and an accurate geometry regarding to the CAD model. Indeed, materials cost can be prohibitive if development time is too long. The printing equipment must be able to adapt itself both to the inks and to the components design.

Last three years, CERADROP has developed an important know-how based on micro-positioning technology to correct static and dynamical following errors of printheads. In the present review we describe several 3D printers (Figure 5) (with printheads angular setting).The understandings of the major errors of droplet placement have been investigated. Moreover, several simulation algorithms have been computed to predict the filling of the CAD components design by the splats of the different materials with different parameters (splats micro-design, splats overlapping…).

This work illustrates the production of 3D fine scale PZT micro-pillar arrays (figure1) for 1-3 piezoelectric ceramic polymer composites for medical imaging probes, whose final electromechanical properties are similar to those of commercial piezoelectric composites made by dice and fill technique. Furthermore, we demonstrate the feasibility of high resolution nanosilver contacts printed on ceramic, silicon, and glass substrates, for thick film and photovoltaic applications (Figures 2,3). Processing conditions, and ink formulation have been optimised to allow the printing of fine lines with variable width and thickness. Finally we also report the investigations on the fabrication by IJP of 3D multimaterial components such as complex Multilayer Ceramic Capacitor device (Figure 4)

Key words: inkjet printing, ceramic, 3D multimaterial components, accurate droplet placement

Acknowledgement:
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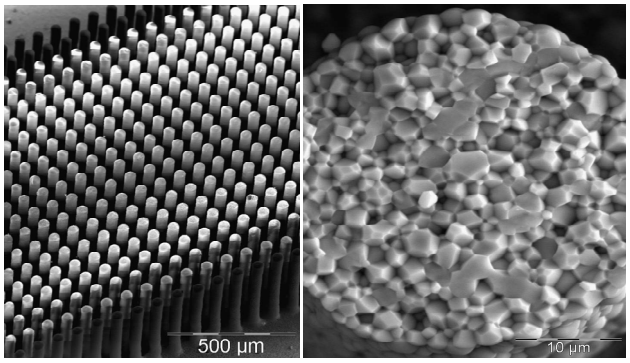

Figure 1: Reproducible and highly dense PZT micropillars array.

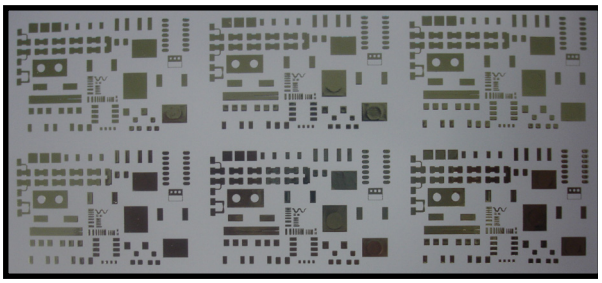

Figure 2: Inkjet printed of electronic test pattern with nanosilver ink.

RAPID PROTOTYPING TECHNIQUE FOR CERAMIC MINI-DEVICES CONTAINING INTERNAL CHANNELS WITH ASYMMETRICAL CONTOUR

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The electrophoretic deposition (EPD) was used to serve the function of the rapid prototyping technology (RPT) regarding to manufacturing ceramic mini-devices containing internal channels with asymmetrical contour, of which case is generally not easily obtainable by the conventional ceramic forming techniques such as slip casting, CIP, tape casting, extrusion or injection molding. The spacing and geometry of such internal channels, either liner, curve-linear, symmetrical or asymmetrical, can be designed via the preliminary stage of computer-aided design to prepare the fugitive inactive inserts such as graphite, paper or polymer, which are positioned with a certain distance in front of working electrode in the EPD working environment. Requirements of such inserts include no undesirable chemical reaction with the solvent of the EPD suspension and easy elimination from EPD-formed ceramic green body from subsequent pyrolysis or acid dissolution.

Ferroelectric ceramic like $(Ba_{1-x}Sr_x)TiO_3$ can be deposited via EPD route unto the conducting electrode with the insert buried in the ceramic laminates, which is removed after adequate drying process. It is feasible to duplicate the mini-devices in terms of formation of desirable array patterns. A great number of mini-device components can be obtained by dicing the pattern array of green state or sintered ceramic laminate. Preparation of EPD suspension also plays a key factor to achieve a good forming performance by EPD in terms of type of organic solvents and their mixture ratio, different milling process, and addition of pH adjusting agent and surface charge modifier in the EPD suspension. Various type of electrodes (Pt, graphite, carbon fiber cloth) were investigated regarding the EPD efficiency, adhesion of ceramic green form, and the RPT performance. Effects of EPD conditions on shape integrity of internal channel in the mini-devices were examined. The results of our study offer some interesting applications when designing the prototype of electrical ceramic components such as mini-actuators, filters, waveguide, and dielectric resonators.

Acknowledgement:
The authors would thank for the financial supports of this work from the National Science Council of the Republic of China (Taiwan) under project contracts No. NSC-97-2221-E-035-002. Also, the grant No. FCU-08G27202 and technical supports from R&D Office and Precision Instrumentation Center, Feng Chia University (Taiwan) are also highly appreciated.

MICRO POWDER INJECTION MOULDING OF ALUMINA DENTAL BRACKETS.

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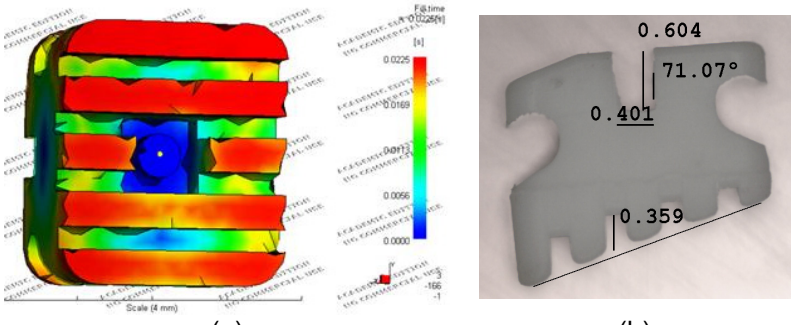
Micropowder injection moulding is an economical mass production technology of microparts. It is specifically suitable for shaping ceramic materials since final net shape can be obtained without the need of machining. Such microparts hold enormous potential for the important area of electronic, automotion, medicine, orthodontics, etc. In this experimental work micropowder injection moulding of alumina dental brackets was studied. Alumina feedstock with a binder system based on high density polyethylene (HDPE) and paraffin wax (PW) was prepared. Feedstock homogeneity was evaluated through pycnometer density measurements of different portions of the same batch. In order to study the suitability of the feedstock for the process a rheological study was performed. Pseudoplastic behaviour was exhibited by all feedstock formulations with viscosity values suitable for injection moulding. Injection stage was carried out in a Battenfeld Microsystem 50 injection moulding machine. Suitable processing parameters including temperature, volume and injection speed were selected to ensure complete mould filling. Injection moulding stage was also analyzed using the Moldflow package following measurement of key feedstock material properties. Binder removal was performed by solvent debinding followed by thermal debinding. Parts were immersed in heptane at 60ºC during different times to determine the optimum leaching time. It was observed that weight loss increase up to 20 minutes of immersion time, longer times did not produce further paraffin loss. A 90% paraffin wax removal was reach. Thermogravimetrical analysis of solvent debound part allowed designing thermal debinding schedule. Final parts reached high densities (99.8%) after sintering at 1600ºC and exhibited good shape retention. However a slight distribution of dimensions in the retention zone of the bracket was observed Moldflow modelling allowed establishing a possible correlation between the mould filling behaviour and this dimensional scatter of the sintered parts (figure1).



(a) (b)

Figure 1.-Injection moulded bracket: (a) Mould filling behaviour and (b) sintered part.

WATER DEBINDING KINETICS OF CERAMIC INJECTION MOULDING FEEDSTOCKS

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Ceramic Injection Moulding (CIM) is an attractive method to produce small parts with relative complex shape. Besides the "classical" thermal debinding process, a couple of alternative methods have been developed over the years that alone or in combination with the former allow to master what is usually considered as the most critical step of the process : catalyzed debinding, super-critical fluid debinding, solvent or water debinding. Water debinding has two main advantages - it is both cost effective and environmental friendly - and has accordingly gained increased interest. Other claimed advantage is the higher debinding rates: that of the water debinding stage itself as well as that of the subsequent, still necessary, pyrolysis step.

In this presentation, the water debinding kinetics of two commercial CIM feedstocks will be reported. The dependence of the evolution of the mass and porosity of the tested specimens on the water temperature (in the range 25-75°C), resident time (up to 48 h) and ceramic solid loading will be shown. The progress of the debinding front under these varying conditions will also been assessed by scanning electron microscopy observation of the partly debinded specimens cross section. Finally, the experimental results will be compared with available models from the literature (e.g. shrinking core model).

Acknowledgement:
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ELECTROHYDRODYNAMIC FORMING OF CERAMIC COMPONENTS FROM A PRECERAMIC POLYMER

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Electrohydrodynamic spraying of a solution of a preceramic polymer (polysiloxane) dissolved in ethanol led to the formation of a great variety of miniaturized preceramic structures (bubbles, capsules, nanofibre mats, micro-tubes, patterned microchannels) with controlled surface characteristics. To achieve these structures, processing parameters such as the voltage difference of the applied electric field, the concentration of the solution and the flow rate were varied. Furthermore, by employing an advanced processing setup comprised of a set of co-axially arranged micro-needles (up to 3 needles at present), the features (shape and surface characteristics) of the components could be further extended.

The samples, varying in size from a few hundred nanometers to a few millimeters, were ceramized, after post-forming cross-linking, by heat treatment in inert atmosphere, leading to SiOC ceramic components (see for instance Figure 1). They could be used in a variety of applications, including as lightweight fillers for syntactic foams, implantable capsules for drug delivery, components for tissue engineering, advanced filters and micro-fluidic devices.

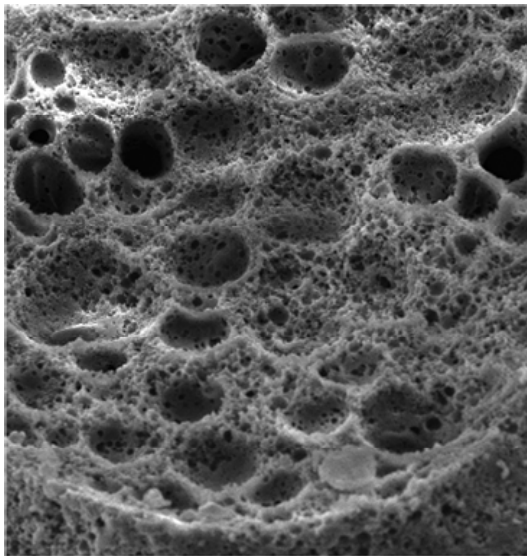

Figure 1. Porous ceramic capsule from electrospraying of a silicone resin preceramic polymer

PROCESSING OF YTTRIA BY GEL-CASTING

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The recent increased interest for gel-casting technique is motivated by its simplicity, capability of forming near net shapes, low concentration of organics in comparison with high-pressure injection molding[1],[2]. Yttria was selected for its excellent physical and chemical properties that make it ideal as host material for solid state laser. On the other hand the low amount of near net shape processes applied to yttria encouraged this study to obtain complex shapes that improve laser performances. The research is now focused on the development of "green" gel-casting method in water, employing polysaccharides as gelling agents. Thermogelling polysaccharides (agaroids) are soluble in water at high temperature and transform into a gel at temperature below their Tg (around 40-50°C). This study was focused to the optimization of thermogel-casting process of yttria slurries induced by polysaccharides (agarose and carrageenan). The high density of yttria (5 g/cm^3) and its tendency to flocculate even at high negative zeta potential (aging effect)[3] needed a deep study in order to achieve stable suspension with the higher solid concentration required to obtain full dense transparent materials for laser applications. Different dispersants were tested. Zeta potential measurements with acoustosizer technique allowed the characterization of the dispersed state in terms of the identification of the best amount of dispersant, pH, particle size distribution. DLS measurements on the suspension well evidenced the aging effect, and the role played by polyacrylate dispersants in comparison with dispersant not containing polymeric chains. Rheological measurements identified the exact Tg temperatures of gelling agents. The yttria suspensions were ball milled with dispersant for 24h and heated at the temperature selected for casting (65°C). At the same time the gelling agents were solubilized at 90°C and added to the ceramic suspension. The resulting mixture was de-aired under vacuum and casted into PE or silicon molds (Fig. 1). The casted bodies were dewatered in a controlled humidity atmosphere, debonded at 500°C and sintered in air or under vacuum at 1600-1700°C. The microstructure of the sintered bodies with final relative density between 90-95% were analyzed by SEM. An homogeneous distribution of spherical pores was intercalated by fully dense zones that showed intragranular fracture. The vacuum sintering reduced the porosity but fully dense material was not obtained probably due to the low density of the green body. Further investigations are in progress in order to increase the slurries solid content and therefore the green density.

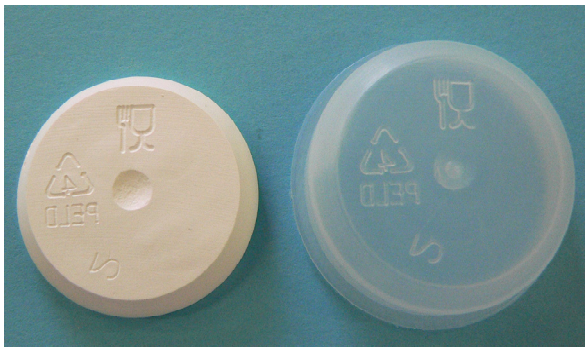

Fig. 1 Green body of yttria thermo gel-casted on a PE cap

[1] G. Tari, "Gelcasting ceramics: A review", *American Ceramic Society Bulletin*, (**2003**) 82:44, 43-46
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FABRICATION OF INTERCONNECTED POROUS CERAMIC PARTS BY SELECTIVE LASER GELLING

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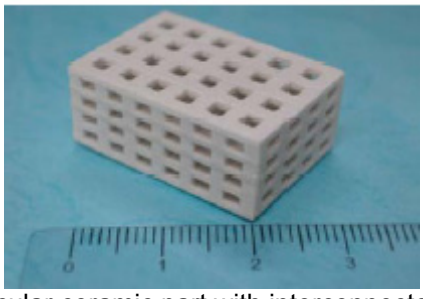
Ceramics have been adopted widely in Rapid Prototyping (RP) technologies in recent years due to their appealing properties. In this paper, a selective laser gelling (SLG) process for fabricating silica parts with **an interconnected porous structure** is presented. The forming procedures in laser scanning of a single line, a single layer, and multi-layers of a ceramic RP part were investigated in detail to unveil their effects on the part accuracy. A strategy for generating **multi-length pillared support** was also proposed to reduce a sagged deflection of the overhang. The proposed technique was verified by fabricating a complex-shaped part with inner channel structures. Furthermore, a number of critical issues including **ease of building porous features and thinner layers, ability to obtain high accuracy and surface finish, and saving material expenses had been investigated.** The effects of process parameters on the gelled line overlap, layer over-gel, dimensional accuracy, and surface finish of the ceramic parts were explored and a set of appropriate parameters were obtained.



A 3D reticular ceramic part with interconnected porous structures made by selective laser gelling.

OPTIMIZATION OF FABRICATION PARAMETERS OF CELLULAR ALUMINA STRUCTURES TO BE USED AS FILTERS

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Cellular alumina structures were fabricated by the replication method following a Taguchi experimental design in order to obtain a structure suited as a diesel soot filter. The replication method consists on the impregnation of a polymeric reticulated foam with a ceramic suspension composed of ceramic powder and additives. The structure is heated in order to eliminate the polymer and consolidate the ceramic as a replica of the original structure.

In this work, alumina powder was used. Prior to the fabrication of the structures, a study of the effect of milling time on the size, shape and agglomeration state of alumina powders was performed. It revealed the necessary milling time to obtain adequate characteristics of the powder (small particles, few agglomerations) in order to promote thixotropic behavior of the suspension and diffusion processes during sinterization. Later on, structures were fabricated using polyurethane foams that were impregnated with a suspension made of 60%wt alumina powder, 5%wt acrylic binder, 1%wt ammonium polymethacrylate and 34%wt water. By controlled compression of the foam the excess suspension was removed and the structure was left to dry. Afterwards it was heated using a heating rate of 0.5ºC/min in order to eliminate the polymer without creating residual stresses. This rate was maintained until 450ºC, since a thermo-gravimetric analysis showed that at this temperature the polymer has been completely removed. A rate of 10ºC/min was then used until 1550ºC. By the replication method it is difficult to obtain structures with small pores (45-60 pores per inch) since the ceramic slurry tends to cover the windows generating clogged pores. However, soot filters must have small open pores in order to retain soot. For this reason a new stage was introduced in the process, which consists in blowing the structure once the excess slurry has been removed. This way the structures produced are highly reticulated (Fig. 1 and 2).

In order to determine the best combination of fabrication parameters a Taguchi experimental design was implemented. The parameters that were varied are: cell density (45 and 60ppi), percentage of compression (40, 50 and 60%), blowing pressure (5, 10 and 15psi) and time sustained at the sintering temperature (30, 60 and 90min). The Taguchi design proved to be useful in maximizing the mechanical resistance of the structures. The optimized structure, fabricated with a 60ppi polymer, 40% compression, blowing pressure of 10psi and 90min sustained at the sintering temperature, presented a Darcyan permeability of 2.11 x 10^{-10} m^2, mechanical resistance of 1.10 MPa and 83% porosity, values that suit the range commonly reported in the soot filtration application.

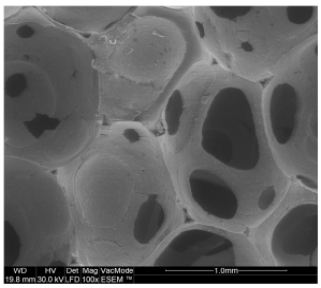

Figure 1: SEM 100x, alumina structure fabricated by the traditional replication method.

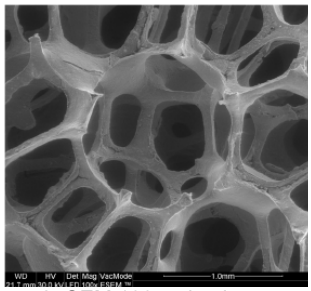

Figure 2: SEM 100x, alumina structure fabricated by the replica method with the implementation of a blowing stage.

COLLOIDAL PROCESSING OF MAGNESIUM ALUMINATE SPINEL DENSE BODIES

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Magnesium aluminate spinel ($MgAl_2O_4$) samples were processed by colloidal techniques, namely slip casting, direct coagulation casting and centrifugal casting. X-ray diffraction analysis and scanning electron microscopy were used to access the characteristics of the starting material (commercial spinel powder). The specific surface area was determined by adsorption of N_2 by the multipoint BET isotherm method and particle size distribution was evaluated by laser diffraction. Zeta potential and viscosity measurements allowed the identification of the pH range of aqueous suspensions stability. High solids loading slurries were prepared and used to process bodies by means of the three different colloidal techniques. For the direct coagulation casting, it was used the enzymatic decomposition of urea catalyzed by urease at room temperature. Compressive strength measurements were performed on cylindrical wet coagulated bodies with different enzyme concentration and decomposition time. The processed samples were characterized by density measurements, dilatometric analysis and SEM observation before and after sintering at 1600ºC during 30 minutes. Results have shown that direct coagulation and centrifugal casting allowed to obtain denser bodies, before and after sintering. Moreover, centrifuged bodies have shown a very homogeneous microstructure with 80% of theoretical density (TD) after casting and 97% TD after sintering.

OPTIMIZATION OF CUPRATE BARIUM POWDER SLIP FOR TAPE CASTING

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One of the processes to produce $YBa_2Cu_3O_{7-\delta}$ superconducting ceramics is the recombination of Y_2BaCuO_5 and barium cuprate rich liquid phases at temperatures above the peritectic melting point, followed by slow cooling. In this work high quality tapes of barium cuprate have been obtained from homogeneous and stable suspensions of $Cu_5Ba_3O_8$ powders in non-aqueous medium.

The $Cu_5Ba_3O_8$ precursor powders were fabricated through repeating calcinations and grinding of stoichiometric mixtures of high-purity $BaCO_3$ and CuO at 850–900 ºC. Suspensions with solids loadings of 3.5 vol.% were prepared in polar and non-polar solvents, such as ethanol, toluene, etc. for sedimentation tests. Two types of dispersants were used, Glycerol Trioleate (GTO) and a polyester/polyamine copolymer (KD-1). The first one is considered as a non-ionic surfactant, while the last one acts via steric hindrance. Concentrations of deflocculant ranged between 0.3 and 0.9 wt.%, and 0.3 and 2.1 wt.% for GTO and KD1, respectively.

The stability of the suspensions was studied through sedimentation tests using diluted suspensions, and the determination of the rheological behaviour of the concentrated slurries with a rotational rheometer operated at controlled shear rate and controlled shear stress modes. The rheological behaviour of the slurries was described using the Cross model and the influence of the solid fraction on the slurry viscosity was studied considering the Krieger-Dougherty model in order to predict the maximum solid loading to which the slips remain stable.

It has been found that the best dispersing conditions are achieved in ethanol when using GTO as deflocculant in a concentration of 0.6 wt.% with respect to dry powder, as can be observed in figure 1. After tape-casting the concentrated slips (37 vol.%), the properties of the green tapes were tested in terms of density, microstructural analysis and thermo-differential and thermo-gravimetric analysis (DSC - TGA) in air. $Cu_5Ba_3O_8$ tapes with a green density of 62 % were obtained from the optimized suspension. Microstructural morphology is displayed in figure 2, where a cross-sectional SEM micrograph of one of the obtained tapes is shown.

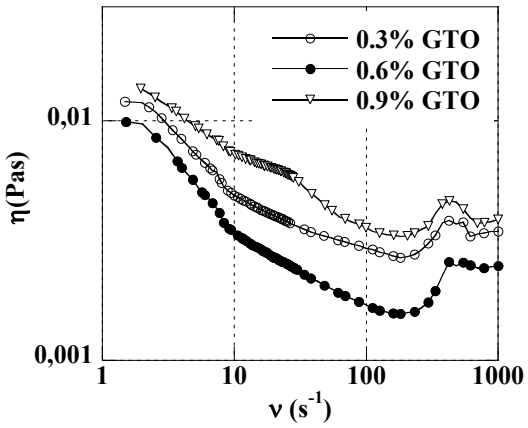

Fig.1. Viscosity curves of suspensions with 18 vol.% solids deflocculated with.GTO.

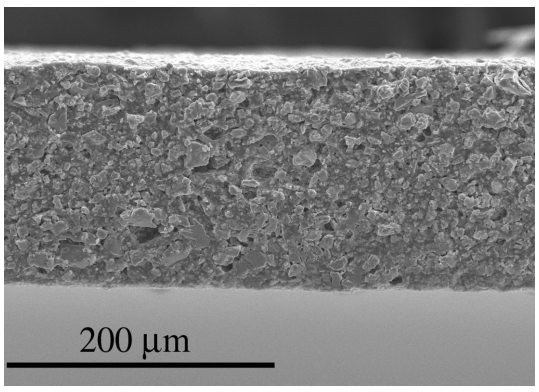

Fig. 2. Cross-sectional SEM micrograph of a green tape.

Acknowledgement:
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TAPE CASTING OF CLAY BASED COMPOSITIONS FOR TILES

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Tape casting is a process that was originally used with traditional ceramics but it is currently used in the manufacturing of advanced ceramics. In particular, tape-casting methods are used to make substrates for integrated circuits and the multilayer structures used in both integrated-circuit packages and multilayer capacitors. In this process ceramic powder slurry, containing an organic solvent such as ethanol and various other additives as binder and plasticizers, is continuously cast onto a moving carrier surface made of “no-stick” material such as Teflon® or Mylar®. A smooth double knife edge spreads the slurry to a specified thickness, the solvent is evaporated, and the tape is rolled onto a take-up reel for additional processing. In subsequent firing operations the organic component of the tape are burned away, leaving the ceramic structure.

Traditional ceramics as tiles are uniaxially pressed and the surface was covered by a glaze composition by the waterfall technique where a conveyor belt carries the green through a continuous, recirculated waterfall of slurry. The flexibility of the process is low but the large scale facilities of tile factories operated continuously at efficiency cost. The preparation tape casting layer of tile based composition could benefit the production of new tiles in the sense of short series as prototypes or could served to integrated functional materials as sensor.

Tape casting of clay based material shows the inconvenience of selective absorption by the clay particles. Thus, the tape casting of clays based materials must be studied in order to determine both the materials and the additives.

In this work we studied the effect of the different raw materials in the production of tile products as: clays, feldspar, and quartz, glaze… as well the effect of different water additives. The formulations for tape casting were optimized for water based systems. The rheology of the slips was studied and the parameters as thickness, density, flexibility of the resulting layers were evaluated. Laminated layers of stoneware composition showed better mechanical performance than uniaxial pressed materials and allowed a drastic thickness reduction of the tile. Color performance was attained with the reduction of 70% of the pigment by using tape casting glaze layers.

TWO ALTERNATIVE ROUTES FOR STARCH CONSOLIDATION OF MULLITE GREEN BODIES

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Starch consolidation technique can be used in the manufacture of porous ceramics for thermal insulations, gas burners, filters, catalyst supports, or bioceramic applications. This method of direct consolidation is based on swelling and gelatinization properties of starch granules in aqueous suspensions at temperatures between 55-80°C. The starch granules perform as both consolidator/binder of the green body and pore former at high temperature in the final body.
In the present work, two alternative routes (labeled as CR and PGR) for starch consolidation of mullite green disks were studied with a view to develop green bodies with homogeneous microstructures. In CR route, disks were formed by pouring the mullite/starch aqueous suspension at room temperature directly into metallic molds and heating at different temperatures and times. In PGR route, disks were shaped by pouring pre-gelling mullite/starch suspensions into the molds and heating at the same experimental conditions. The pre-gelling process of the mullite/starch suspensions was carried out at 61-62°C, temperature range slightly lower then the onset gelatinization temperature of cassava starch (64.1°C).

Commercial available powders of mullite (Baikowski, France) and cassava starch (Avebe S.A., Argentine) were employed as raw materials. Cassava starch was used as consolidator agent of the mullite aqueous suspension and binder of the ceramic particles. Both cassava starch and mullite powder were characterized by several techniques: i.e. measurements of real density by He-pycnometry (Multipycnometer, Quantachrome, USA), particle size distribution by laser diffraction (Matersizer S, Malvern Instruments, UK), scanning electron microscopy, SEM (JEOL JSM-6460, Japan). The mullite powder presented a high purity level and fine particle size (D_{50} = 1.46 μm).
Mullite/starch aqueous suspensions (0.25 starch volume fraction of 40 vol.% total solid loading) were prepared by intensive mechanical mixing and homogenization in a ball mill. The optimum stability conditions of both mullite and mullite/starch aqueous suspensions were determined by measuring zeta potential (Zetasizer Nano ZS, Malvern Instruments, UK) and shear flow properties in controlled stress and controlled rate operating modes (Haake RS50, Thermo Electron Corp., Germany): 0.45 wt.% of dispersant (Dolapix CE-64, Zschimmer & Schwarz, Germany), pH 8.7 and ball milling for 6h. Furthermore, the viscoelastic behaviour (RDA-II, Rheometric Scientific, USA) of starch and mullite/starch aqueous suspensions as a function of temperature (30-95°C) and strain (0.1-625%) was studied in order to determine the consolidation experimental conditions used in each route. In both routes, CR and PGR, the thermal consolidation process of the mullite/starch suspension was carried out in an electric oven (Memmert UFP 400, Germany) at 70 and 80°C during different times (1 and 2 hours). Once consolidation process finished, samples were unmolded and dried in a ventilated oven at 40°C for 24h.

The green bodies obtained were characterized by bulk density measurements using the Archimedes' method (immersion in mercury), apparent porosity and microstructural analysis by SEM (Jeol JSM-6460)/EDX (Genesis XM2-Sys) on external surface and fracture surface. The results were analyzed taken into account the behavior of the cassava starch in aqueous suspension at temperature (swelling and gelatinization process) and the characteristics of the gelatinized granules.
The analysis of the green microstructures (mapping of aluminum, silicon and carbon) developed by both consolidation routes in function of the temperature and time of consolidation, allowed to evaluate mainly the homogeneity of the distribution of raw materials in the green bodies, outstanding aspect to be considered in the porous ceramic processing.

UNIFORMED POWDER COMPACTS FABRICATED FROM AIR DRY FORMING METHOD USING CONDENSED SLURRIES WITH ADDITION OF GLYCEROL

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The wet forming processing which produces the compact from slurry has a big advantage in producing the homogeneous ceramics with higher density. High dispersion state in slurry directly affects the particle packing structure in green compact. Homogeneous packing structure contributes to near-net-shaping. We have studied a drying-induced forming method, in which dispersed slurry with high concentration 60vol% is poured into a mold and simply dried. The obtained powder compacts have high density and no preferred orientation. Similarly, it is easy to induce slurry flocculation during drying with high density slurry. In this study, we improved the drying-induced forming method by using highly concentrated alumina slurry with the visco-elastic property. Explicitly, the elastic storage modulus of slurry is reduced by giving the shearing strain to the slurry. Destruction of the network structure between the particles results in a well-packed green compact. Characteristics of green compacts and the sintered body produced with this method are examined.

PRODUCTION OF POROUS CERAMICS AND HOLLOW CAPSULES FROM PARTICLE-STABILIZED EMULSIONS

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We propose a versatile and simple approach to produce solid macroporous ceramics from emulsions stabilized with particles of various chemical compositions. Stabilization with particles hinders extensive droplet coalescence during solvent extraction, allowing for drying and sintering of the emulsions directly into macroporous materials in the absence of any chemical reaction.

Emulsions were stabilized without the use of hazardous and toxic surfactants. Surface modification of initially hydrophilic inorganic particles was achieved through the in situ adsorption of short amphiphilic molecules.

The properties of the emulsions can be tuned according to their specific applications. Diluted emulsions can be used for dip coating, spin coating and spray deposition on substrates. In addition, the high dilution of the emulsions allows the production of semi-permeable hollow capsules which are attractive materials for the encapsulation and delivery of active agents in food processing, pharmaceutical and agricultural industries

Here, we demonstrate examples of macro-porous ceramics with various microstructures, as well as hollow capsules from different metal oxides, including alumina, silica, and iron oxide.

STUDY OF Al_2O_3-TiO_2 GRANULES OBTAINED BY FREEZE-DRYING PROCESS

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Nanostructured materials allow enhancing the properties of their conventional counterparts while improving the performance. The production of nanostructured coatings using thermal spraying methods should provide coatings with interesting properties. Unfortunately, nanoparticles cannot be directly sprayed because of their low mass and poor flowability. However, several studies have shown that this problem can be solved by agglomerating them in micrometer sized granules. One of the most commonly used material for the deposition of wear resistant coatings is Al_2O_3-TiO_2, which has been studied in this investigation.

The use of granules obtained by freeze-drying of a stable suspension ensures the homogeneity of the starting powders and avoids some problems of other drying techniques, such as the possible formation of a surface-segregated layer of binder after the granule is dried. This work deals with the granulation of Al_2O_3-TiO_2 nanosized powders through a freeze-drying process in order to obtain a sprayable nanostructured feedstock for atmospheric plasma spraying. Commercial colloidal suspensions of Al_2O_3 and TiO_2 with 10 vol% solids were first characterized. Nanosized powders were used to increase the solids loading of the mixture from 10 to 15 vol%. The stability of the suspensions was studied in terms of zeta potential and rheological behaviour, considering the effect of deffloculant content, the sonication time and the addition of PEG to improve adhesion. The different parameters involved into the process (solid loading of slips, binder additives, freezing conditions, etc.) have been studied and optimised. The characteristics of the shaped granules (size, porosity, etc.) were studied and compared to those of granules obtained by spray-drying.

Acknowledgement:
This work has been supported by Spanish Ministry of Science and Innovation (project CIT-420000-2008-2).

USE OF EXTRUSION TECHNOLOGY FOR OBTAINING OF DENSE TITANIUM OXIDE CERAMICS

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Extrusion is a perspective forming technology for obtaining of objects with certain profile. Nowadays it is widely used ceramic materials forming technology, because it is relatively cheap and productive. Titanium oxide can be used as biomaterial, photocatalyst, electrode in the electrolysis process etc. The aim of this work was to obtain dense titanium oxide ceramics using extrusion technology and to compare obtained products with isostatic pressing technology obtained ones.
Sample preparation of pressed samples includes uniaxial pressing of TiO_2 powder with different additives, followed by isostatic pressing. The green density of products obtained was in range from 55 to 59 % of theoretical density.

During the investigation extrusion mass preparation technology for obtaining of dense and qualitative samples was developed. Overall mass preparation and sample processing technology can bee seen in Fig.1. Additives used for mass preparation includes water, plasticizer and binding agent. Green density of cylindrical samples after extrusion was in range from 54 to 59 % of theoretical density.

Pressed and extruded samples were calcinated and sintered in equal conditions.

Microstructure of samples obtained was investigated using scanning electron microscopy.

During the investigation it was concluded that sample density is influenced not only by raw materials composition but also by mass preparation and forming technology.

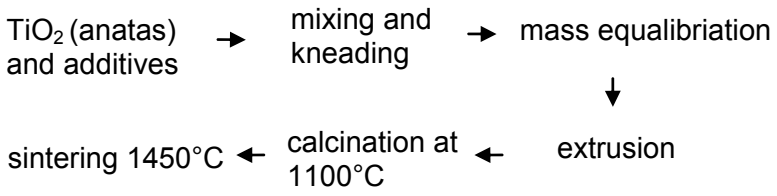

Fig.1. Schematic chart of extrusion sample processing

Acknowledgement:
This work has been partly supported by the European Social Fund within the National Program "Support for carrying out doctoral study programs and post-doctoral researches" project "Support for the development of doctoral studies at the Riga Technical University".

CONTINUOUS EXTRUSION OF SUSPENSIONS OF NATURAL ZEOLITES

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Natural zeolites have exceptional physical-chemical properties that arise from their crystalline structure containing large voids and channels, which permit the transfer of ions and the selective sorption of molecules. Natural zeolites can be used as “molecular sieves” to separate ions from aqueous solutions by ionic exchange and to separate mixtures of gases in hydrocarbon reactions amongst many other applications.

The extrusion of zeolite monoliths and profiles is typically done by discrete processes as piston or ram extrusion, with almost not attention to their suspension rheology. In this work, the rheological characterization and continuous extrusion of suspensions of natural zeolites (clinoptilolite-heulandite) was analyzed in order to obtain long profiles. For this purpose, the technique of colloidal processing of ceramics was used to prepare concentrated suspensions at 61 v/v% of solid content with and without binder.

Mined stones were obtained from the region of Catano-Etla, Oaxaca, in the south of México, and further dry ball-milled up to reach particle sizes smaller than 105 μm. A rheological characterization of the suspensions was carried out under steady and oscillatory shear flow using a Paar Physica UDS 200 rotational rheometer. Then, continuous extrusion of tubes was performed at different speeds by using a Brabender conical twin screw extruder. For the suspension without binder the extrusion took place smoothly without defects on the extrudate surface. However, due to the relatively low yield stress of the suspension, the tubes did not retain their shape after extrusion (Fig. 1). In contrast, the extrudates obtained by adding the binder appeared with excellent surface quality and retained their shape after extrusion, in agreement with a significantly higher yield stress value and slip flow during extrusion (Fig. 2). These preliminary results suggest the continuous screw extrusion as a promising technique for shaping ceramic compounds (Fig. 3).


Figure 1

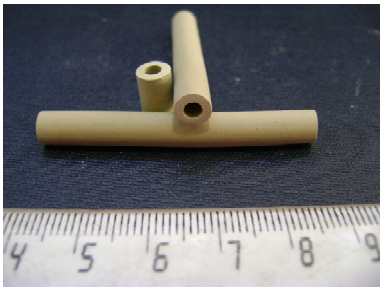
Figure 2

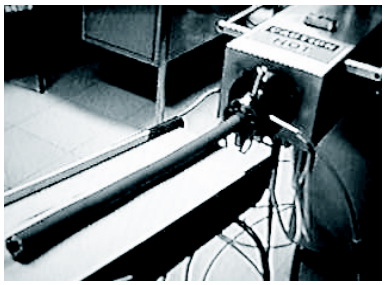
Figure 3

SYNTHESIS OF CERAMIC NANOPARTICLES BY LASER ABLATION IN LIQUIDS

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Ceramic nanoparticles have attracted much interest due to the huge number of applications. Laser ablation of a solid target immersed in a liquid is receiving increased attention as a new technique to form colloidal nanoparticle suspensions. A remarkable advantage of this method is the relative simplicity of the experimental setup. Moreover, it has been shown that the laser ablation in liquids is suitable for obtaining nanoparticles of metals and compounds.

In this work, we present a novel approach to directly produce highly-dispersed Al_2O_3 colloidal nanoparticles using laser ablation in water of four different ceramic and metallic targets: a) a dense, sintered (1550 ºC) alumina ceramic sample; b) a green pellet prepared by uni-axial pressing (200 MPa) of alumina micron-sized powder; c) a green sheet of alumina obtained by tape-casting; and d) an aluminium plate. Laser ablation of samples was performed using the fourth harmonic (266 nm) of a Q-switched Nd:YAG laser with a pulse width of 7 ns. The ablation was carried out during 60 min at a pulse repetition rate of 10 Hz. Laser radiation was focused by a 100 mm focal length lens at normal incidence to the surface of sample placed inside a glass vessel filled with de-ionized water. The height of the liquid layer above the target was kept to 10 mm. The laser entered the solution from the top at normal incident angle and was then focused on the target to a spot of about 500 μm in diameter. The glass cell was displaced under the laser beam using a computer-driven X–Y stage. The experiments were carried out, for each sample, at fluence values of 5, 10 and 20 J cm^{-2}.

Crystalline phases, elemental composition, particle size and morphology, and optical properties were investigated by dynamic light scattering, SEM, TEM, XRD, UV-VIS absorption spectroscopy and Raman spectroscopy. Depending on both the target type and the fluence value, nanoparticles with different shape, size and composition were obtained. The use of alumina targets (mainly the sintered sample) and low fluence values led to the formation of nearly spherical Al_2O_3 nanoparticles with 5-10 nm in diameter and a narrow size distribution, as can be seen in a representative picture shown in Figure 1.

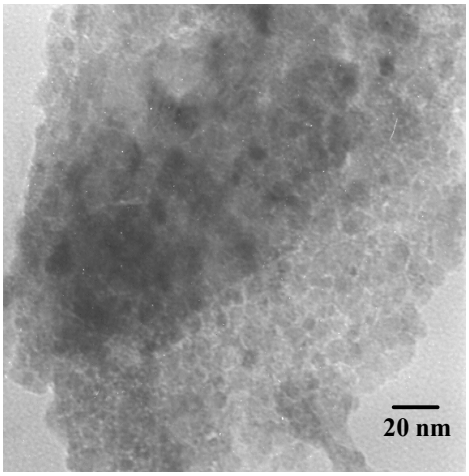

Figure 1. TEM image of Al_2O_3 nanoparticles prepared by ablating the sintered alumina target at a fluence of 5 J cm^{-2}.

Student Contest 1

Chair persons: A. Segadães &. C. Baudin

CHEMICAL STABILITY OF $CoAl_2O_4$ BLUE PIGMENT IN AQUEOUS SUSPENSIONS

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Ceramic pigments are inorganic structures containing chromophore ions, which impart colour to ceramic wares by forming heterogeneous mixture with body or glaze. In other words, inorganic pigments are solids which do not react physically/chemically with medium and practically insoluble in the medium which they are dispersed. The ceramic tile market is showing increased interest in customized products, including the reproduction of high-quality images on tile surfaces. Ink-jet technology is a very promising decorating technique for this application which is a non-contact method based on projecting ink droplets onto a surface and permits better control of the image quality. The ceramic pigments with particle size in the nanoscale used in ink-jet printing have an enormous potential market, because of their high surface area, which ensures higher surface coverage, higher number of reflectance points and hence improved scattering. In addition, pigments are desired not to dissolve in media due to deterioration of color intensity and quality. However, smaller particle size of pigment results in easy dissolution of some ions from pigment surfaces in water because of increasing surface area and energy. Therefore, colloidal properties of pigments in aqueous environment and physical-chemical interactions between pigments and solvent should be well known. Accordingly, in this study, one of the widely utilized pigment, blue $CoAl_2O_4$ (Co-aluminate) pigments' chemical stability in aqueous suspensions was investigated. First of all, industrially available blue Co-aluminate pigment was characterized by x-ray diffraction (XRD) and x-ray fluorescence (XRF) methods. 5 wt % pigment-water suspensions were prepared for pH/conductivity and zeta potential measurements. After grinding the pigments to nano size, pigments were dispersed in distilled water and again pH/conductivity measurement was performed. The interaction between both treated and untreated pigment particles and water was investigated by using Inductively Coupled Plazma Optic Emission Spectrometer (ICP-OES). The ICP-OES results show that cations of Co-aluminate pigments dissolve in aqueous system. In this presentation, the kinetics of the dissolution and effects of surface area on dissolution behaviour will be discussed. In addition, some approaches will be presented to eliminate the dissolution of cations and subsequently to maintain the colour quality.

Acknowledgement:
This work was supported by TUBITAK under grad no: MAG-107M366.

MONOSACCHARIDES DERIVAIVES AS MONOMERS IN GELCASTING PROCESS

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Recently colloidal processes are willingly applied in fabrication of high-quality ceramic elements of complicated shape. Among these processes we can distinguish gelcasting which combines conventional moulding from slips with polymer chemistry. Gelcasting process allows obtaining high-quality, complex-shaped ceramic elements by means of an *in situ* polymerization, through which a macromolecular network is created to hold ceramic particles together.

The key role in the process plays selection of a suitable monomer, which is able to provide high mechanical strength of a gelled element. The applied monomers must perform a series of conditions: they must be water soluble, non-toxic and high mechanical properties must be achieved by as low as possible concentration of monomer in a ceramic slurry. There are a few commercially available monomers which can be applied in gelcasting process, but they still have some disadvantages. Above all their toxicity is not low enough. Secondly, they must be used in pair with a cross-linking monomer, otherwise the received green bodies would not be rigid enough. For that reasons the synthesis of new low-toxic monomers is essential.

The authors therefore performed research on synthesis new monomers on the basis of monosaccharides. Monosaccharides are non-toxic, soluble in water and due to the presence of many hydroxyl groups in a molecule can be applied in gelcasting without adding any external cross-linking agent. The cross-linked polymer network is created by forming hydrogen bonds between polymer chains.

The aim of the research was obtaining ceramic elements from different alumina powders by gelcasting method with application of the new monosaccharide monomer 3-acrylic-D-glucopyranose. The properties of received elements have been compared with those obtained with application of commercially available monomer 2-hydroxyethyl acrylate.

Acknowledgement:
This work was financially supported by Ministry of Science and Higher Education of Poland, (Grant No. N N209 150236)

WATER BASED SYNTHESIS AND DISPERSION OF $Ni(OH)_2$ NANOPARTICLES

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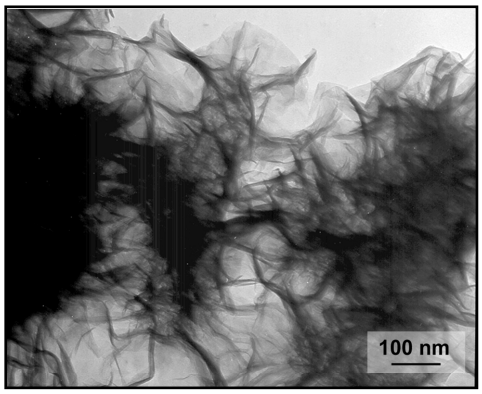
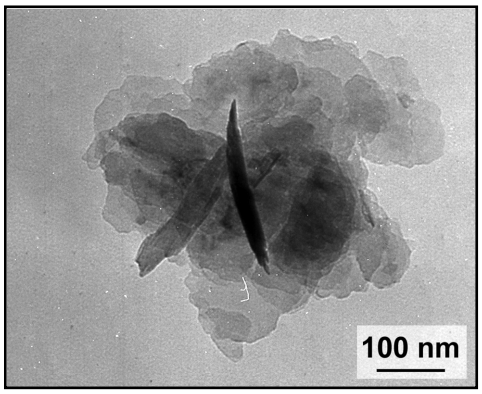
It is well known that nickel hydroxide, $Ni(OH)_2$, can be used as the active material of the positive electrode in alkaline rechargeable batteries as well as the precursor of NiO for catalytic and electrochromic devices. As the performance of alkaline rechargeable batteries highly depends on the size, morphology and phase of the active material, considerable work have been focused on controlling these parameters. $Ni(OH)_2$ presents two polymorphs, α-$Ni(OH)_2$ and β-$Ni(OH)_2$. The α-$Ni(OH)_2$ phase displays a more disorderly and larger interlayer spacing as the interlamellar space contains anions or water molecules, whereas, the β-$Ni(OH)_2$ phase has well-oriented $Ni(OH)_2$ layers perfectly stacked along the *c*-axis with an smaller interlamellar distance.

In this work it is presented the synthesis of both polymorphs of the nickel hydroxide through aqueous ammonia complexes by a sonochemical method what leads to particles with D_{BET} of 30 and 50 nm for α and β phase respectively. Synthesized powders are morphologically characterized by SEM and TEM and phases are determined by XRD. Polyacrylic acid has been employed as dispersant in order to decrease and control the particle size of the obtained powders. Results are discussed in terms of both zeta potential and mean particle size.



TEM image of α-$Ni(OH)_2$



TEM image of β-$Ni(OH)_2$

DEFLOCCULATION OF NANOZIRCONIA POWDERS BY MEANS OF MONOSACCHARIDES ADDITION

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Zirconia is nowadays one of the most important structural ceramic materials. Its mechanical and physicochemical properties enable it to be applied in various aggressive media and under intense mechanical stress.

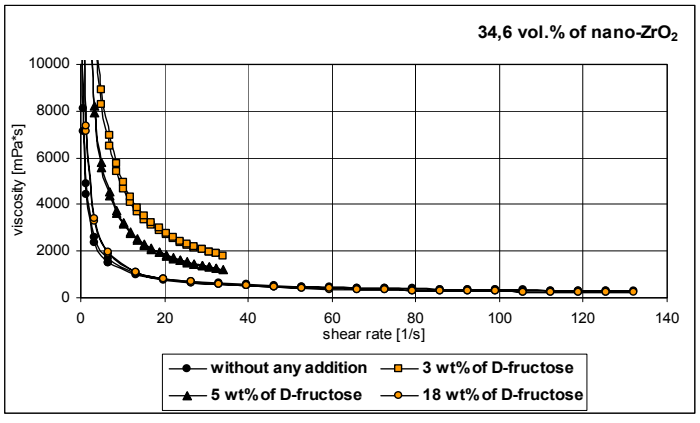
It is commonly known, that products made of nanopowders can be characterized by higher mechanical strength than those made of micropowders. Therefore, there is an urgent necessity to develop suitable preparation method of nanopowders on each step – from powders synthesis to sintering route. Because slip casting is one of the most significant methods for nanopowders forming, many researchers look for some efficient deflocculation agent, to obtain unviscous slurries with high solid phase content.

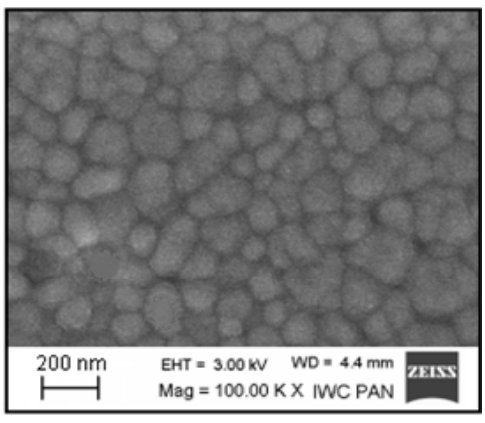
The purpose of these investigations was to examine, how some monosaccharides influence on rheological properties of ceramic slurries with content of nanozirconia powder. Monosaccharides as processing agents have a number of advantages – they are non-toxic, water soluble, inexpensive and readily available. Moreover, they effect high mechanical strength of green bodies and can be easily removed during sintering process. Besides, it has been proved recently, that addition of monosaccharides, particularly D-fructose, effectively decreases the viscosity of nano-Al_2O_3 slurries.

In comparison to alumina nanopowder, rheological investigations demonstrated that monosaccharides increase the viscosity of ceramic slurries made of nano-ZrO_2 with an average particle size of 44 nm (Inframat Advances Materials) – Figure on the right side. Explanation of such unexpected phenomenon requires some additional investigations.

There were also some attempts of nanozirconia sintering performed. The samples with addition of selected monosaccharides, made by means of slip casting method, were sintered at 1300°C. For densification of samples two different sintering techniques were applied: one- and two-step sintering route. Relative density as-sintered bodies exceeded 97% of TD. Their microstructure was characterized by nanometric size grains less than 130 nm. Figure on the left side shows microstructure of sintered body made of nanozirconia sintered at 1300°C by means of one-step sintering (with a dwell time 1 h).

This work was supported by Warsaw University of Technology





SPRAY DRYING OF TiO_2 NANOPARTICLES INTO REDISPERSIBLE GRANULES

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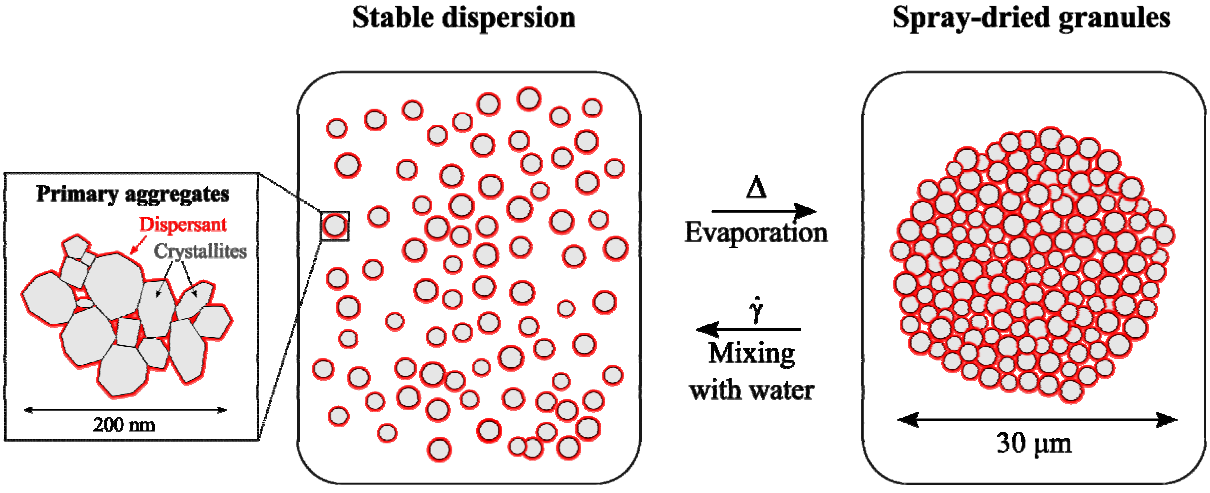
The reduction of the grain size is a straightforward method to improve or introduce new functionality in ceramics. However the handling of nanosized powders requires specific facilities and expertise, due to their potential toxicity, agglomeration issues, and poor flowability.

We have demonstrated a simple and industrially viable method for the upgrading of a commercial TiO_2 powder into safe redispersible free-flowing granules by spray-drying. Titania nanoparticles have been deagglomerated in aqueous media and the effect of various aqueous dispersants has been evaluated using rheology, particle size and electrokinetic measurements. The spray drying performance was assessed in several different configurations with respect to granule size distribution, morphology, mechanical strength and flowability.

The possibility to prepare nanoparticle granules by spray-drying that have a good flowability and a size that minimize respiratory intake without inducing additional hard agglomerates could provide a route to safe handling of nanoparticles. This method can be easily implemented and adapted to other materials.



Acknowledgement:

This collaborative work was performed within the framework of the European Project "SAPHIR—Safe, integrated & controlled production of high-tech multifunctional materials and their recycling".

ELECTROCONDUCTED ASSEMBLY OF NANO-YSZ PREPARED BY MILD – HYDROTHERMAL SYNTHESIS.

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Research on nanostructured materials is motivated by the fact that control of the materials nanostructure results in enhanced properties at macroscale. Major efforts have been made to synthesize, establish and shape weakly agglomerated nanoparticles using *environmental-friendly routes*. In this sense, Hydrothermal Synthesis can be considered one of the best alternative methods to reach such purposes. It is an ecological soft chemical route, using low work temperatures and obtaining nanometer sized crystalline powders with weakly bonded agglomerates. On the other hand, Electrophoretic Deposition (EPD) stands out as a powerful and versatile colloidal process to face inexpensive and mass production of films using low concentrated suspensions (< 10g/l).
This work focusses on the performance of nanostructured films in a single step process, joining nanoparticles synthesis and their further assembly by EPD. The c-ZrO_2 was attained doping the ZrO_2 structure with different rare earth cations such as Y^{3+}. In the present work, nanoparticles of YSZ synthesised under mild hydrothermal conditions were considered. Their dispersion in the synthesis mother liquor was optimised in terms of particle size and zeta potential considering different sonication times and dispersant agents. The physical parameters associated to EPD (current density, potential, electric field and deposition time) were optimised to produce homogeneous thin films of nano-YSZ by EPD from low concentrated suspensions (< 1g/l).

Acknowledgement:
This work has been supported by CICYT (Spain, contract Nº MAT2006-01038) and CAM (CCG08-CSIC/MAT-3811) projects. I. Gonzalo-Juan acknowledges the Spanish Ministry of Science and Innovation for the concession of a FPU-AP2006 grant.

POWDER CONDITIONING FOR THERMAL SPRAYING PROCESSES

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Mullite and mullite/YSZ have interest as protective layers for Si-based components such as SiC/SiC or Si_3N_4 materials to reduce the silica recession, which occurs in atmospheres containing high water vapour concentrations as it happens in engines and turbines. These coatings are called environmental/ barrier coatings (EBCs) and are normally produced using plasma spraying methods. In the plasma stream, particles reach very high velocities and temperatures with in-flight values of the order of 400 m/s and 3000ºC, which produces their melting. The powders have certain restrictions regarding size and flowability to avoid excessive vaporisation in the plasma torch or powder feeder clogging, then reducing the whole process efficiency. Usually, commercial powders for plasma spraying are produced either by spray dry methods or by fusing and crushing, although, in the case of mix mullite/YSZ compositions there are not commercially available powders. Present work deals with the production of mullite and mullite/YSZ powders feedstock suitable for plasma spraying at laboratory scale, using three different routes: spray drying, flame spheroidation and freeze granulation. These routes allow obtaining batches in the order of Kg in reasonable times. The yields of each method are compared and the characteristics of the powders, such as particle size distribution, grain shape, crystallinity and flowability are contrasted for the different conditions. Scaling-up of the processes will be also comparatively addressed. Characteristics of the mullite powders and the mix compositions obtained by the three methods will be discussed in relation to their suitability for the plasma spray process. Example of the freeze granulated powders and corresponding particle size distribution are shown in Fig 1 for pure mullite conditioned from commercial fine powders.

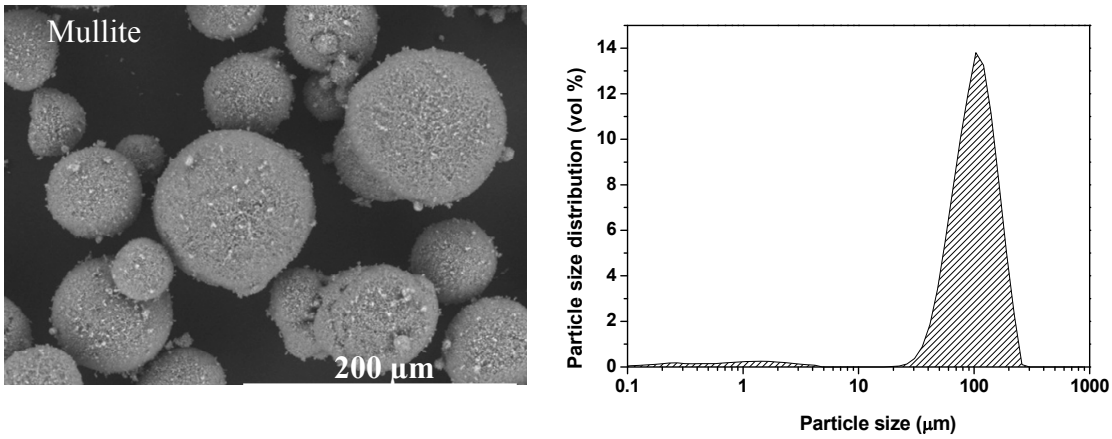

Fig. 1 Freeze granulated mullite powders (left) and corresponding particle size distribution (right) after thermal treatment at 1500ºC

HOLLOW SPHERICAL TiO_2-BASED PHOTOCATALYSTS PREPARED BY SPRAY PYROLYSIS

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Titanium oxide (TiO_2), especially in the anatase phase, is known for its photocatalytic properties which are utilized in various applications ranging from self-cleaning windows to antibacterial coatings. Incorporation of metal nanoparticles like Au and Ag in the TiO_2 particles is found to improve the photocatalytic properties. Spray pyrolysis is a unique preparation technique for oxide materials as it easily provides nanosized particles with desired composition at high production rate. The technique can also synthesize hollow particles with porous shell that gives higher surface area per volume of the catalyst. The feasibility of spray pyrolysis for production of photoactive nanoparticles of TiO_2, pure and doped with metals, for removal of natural organic matters and pesticides in potable water was investigated in this study.

The materials were prepared by spray pyrolysis of aqueous precursors based on titanium oxalate or titanium isopropoxide, with and without addition of Au or Ag compounds. The as-synthesized powders (raw powders) were mostly amorphous consisting of hollow spheres 3-10 µm in average diameter and with average shell thickness of 200-400 nm (Figure 1). A correlation between the precursor concentration and the resulting size of the hollow spheres was established. TEM investigations confirmed a homogeneous distribution of Au and Ag nanoparticles with diameters down to 2 nm (Figure 2). By a subsequent calcination step, the phase composition and crystallite sizes of the raw powder were modified. The hollow spheres were found to be stable upon calcination. The material characteristics and the phase composition can be tailored by varying the metal doping and precursor concentration. The produced particles oxidized methylene blue and humic acid solutions under UV-irradiation; some materials with photoactivity similar to the commercial photocatalyst Degussa P25.

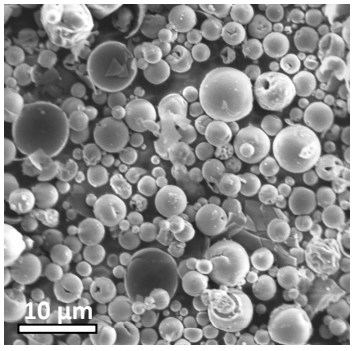

Figure 1. Spray pyrolysis-made raw powder of TiO_2.

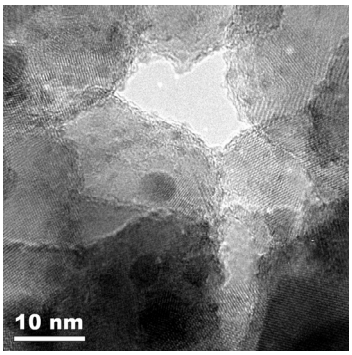

Figure 2. TiO_2 with 5 % Ag (dark spots) calcined at 625 ^{o}C.

Acknowledgement: The study is partially financed by EEA in the form of the grant PL0084

RHEOLOGICAL BEHAVIOUR OF ZIRCONIA AND TITANIA SUSPENSIONS TO SYNTHESIZE ZIRCONIUM TITANATE-BASED COMPOSITES

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Zirconium titanate presents high potential as constituent for low thermal expansion materials with structural applications due to its crystallographic anisotropy in thermal expansion. One of the requirements to produce structural materials is to find suitable conditions to produce bulk pieces. Colloidal processing has demonstrated to be a powerful route for the synthesis and shaping of reaction sintered materials with complex shape and microstructure.

This work deals with the synthesis of zirconium titanate-based composites by reaction sintering of green compacts obtained by colloidal filtration of aqueous concentrated suspensions of titania and zirconia with and without Y_2O_3 as stabilising oxide. Suspensions of TiO_2, m-ZrO_2 and Y-TZP were prepared to 45 vol.% solids by dispersing with a polyacrylic-based deflocculant and ball milling for 24h. Rheological behaviour of monophase suspensions were studied as well as the stoichiometric mixtures of TiO_2/m-ZrO_2 and TiO_2/Y-TZP. Green compacts were obtained by slip casting in plaster molds and their green densities were determined using mercury by the Archimedes' method. Reaction sintered materials were obtained after heating at 1500ºC/2h.

When using m-ZrO_2 as zirconia source, the high temperature phase $ZrTiO_4$ was obtained as the only phase present in the material. When Y-TZP is used, a multiphasic material is obtained consisting of a matrix of the low temperature phase of zirconium titanate ($Zr_5Ti_7O_{24}$), c-ZrO_2 and a pyrochlore phase as secondary phases.

The rheological behaviour of the single and the mixed suspensions is related to the shaping performance and the final characteristics of the sintered materials, including density, phase evolution and microstructure development.

WATER BASED PROCESSING OF NANO Y_2O_3 DISPERSED HYDROXYAPATITE COMPOSITES.

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Hydroxyapatite – Y_2O_3 composites for orthopedic applications have been prepared by slip casting starting from water based slurries of commercially available powders. The final goal was to obtain a well-dispersed suspension of micron size HAp with a content of nanosize Y_2O_3 particles in the range of 5-10%. The colloidal processing technique was chosen with the aim to avoid agglomeration of Y_2O_3 that occurs using other consolidation techniques. An ammonium poliacrylate with a molecular weight of 2000 has been used as dispersant and the optimal amount for both two powders has been determined by mean of the zeta potential determination. Rheological measurements have been performed to optimize the solid content of the HAp suspensions as well as the influence of Y_2O_3 addition. The effect of processing parameter on the properties of sintered compacts was investigated in order to optimize processing conditions.

SLIP PREPARATION FOR BIOCERAMICS CONTAINING MACROPORES

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The use of macroporous bioceramics, with interconnected pores and channel diameters greater than 100 µm, represents a promising substitute for natural bone, specifically for oral, orthopaedic and maxillofacial applications. Porous bioceramics have been extensively studied as scaffold materials that mimic natural bone macrostructure. The ability to fully control the interconnectivity and size of the open pores is crucial for the repair and reconstruction of diseased or damaged parts of the body.

At present, there are numerous traditional as well as novel processing routes available in order to fabricate porous ceramic scaffolds. High definition stererolithography rapid prototyping (SLA-RP) technology using ceramic slips is one approach being investigated to produce bioceramics with the desired 3D interconnectivity and pores with diameters of 200 microns and narrow size distribution. The ceramic scaffold itself must have maximum density in order to achieve maximum strength.

The present research endeavours to obtain alumina slips of highest solids loading and optimum viscosity with the aid of dispersants. The alumina powders were characterised for particle size, surface area, morphology and porosity. Alumina slips were prepared and evaluated for agglomerate size, zeta-potential and viscosity. The dispersants used included octanol and latex. The change in the properties of the slip with the addition of dispersants are compared and discussed.

Acknowledgement:
The authors are grateful for the support of the funding from the Dublin Institute of Technology/University of Limerick Postgraduate Research Initiative Common Interest Group.

RHEOLOGY OF UV CURABLE COLLOIDAL SILICA DISPERSIONS FOR RAPID PROTOTYPING APPLICATIONS

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Silica is a very promising material for micro-devices produced by rapid prototyping techniques involving UV curable dispersions. Dispersions for rapid prototyping methods such as stereolithography have to posses specific rheological properties which are crucial for such applications. We developed highly filled, low viscous silica dispersions (up to 60 vol. %) in UV curable acrylates. The influence of silica particle size, solid loading, temperature and shear rate on dispersion viscosity was investigated. The dispersions exhibited different types of shear thickening depending mostly on particle size. The critical shear rate defined as the shear in which shear thickening occurs was found to be dependent on temperature, particle size, solid loading and type of monomer mixture. The understanding of these rheological properties enables the design of new dispersions which meet rapid prototyping requirements.

Reference

Wozniak, M., Graule, T., de Hazan, Y., Kata, D., Lis, J.,Highly loaded UV curable nanosilica dispersions for rapid prototyping applications. *Journal of the European Ceramic Society (2009),* Available online 27 February 2009

Invited Lectures and Session 3

Chair persons: J.P. Eraw &. I. Santacruz

POROUS CERAMICS FOR GAS AND BIOMOLECULE SEPARATION

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We will introduce novel routes to the synthesis and processing of porous ceramics for gas and biomolecule separation applications. A novel and facile powder processing approach for the rapid production of mechanically stable hierarchically porous materials from porous particles, e.g. mesoporous silica particles, diatomite powders and zeolites is demonstrated. Rapid heating of a powder body subjected to a compressive pressure results in a local surface deformation and activation of the particles at the contact points yielding a strongly bonded powder body without the use of secondary binders. Examples on how the pore size distribution can be engineered will be shown and efforts on green machining will be demonstrated together with preliminary data on catalytic activity and the selectivity and total uptake of carbon dioxide.

We will also briefly describe how surfactant-templated mesoporous spheres can be synthesised and how these carriers can be used for controlled release and delivery. Confocal laser scanning microscopy (CLSM) has been used to follow the time-dependent transport of charged fluorescent dyes and fluorescently tagged macromolecules within mesoporous silica spheres with a well-defined pore size. Relating bulk release to the local molecular transport within the mesopores provides an important step toward the design of new concepts in controlled drug delivery and chromatography. The release kinetics and the time-evolution of the concentration profiles within the mesoporous spheres have been analyzed and fitted to diffusion models.

Recent work on how the mesoporous silica colloidal particles can be coated with supported lipid bilayers will also be shown. The cell membrane coating of the mesoporous silica is achieved by rupture and fusion of small unilamellar vesicles onto the particle surfaces. A multisubunit redox-driven proton pump, cytochrome c oxidase, was incorporated into the membrane and we show that the enzyme is fully functional, both with respect to catalysis of O_2 reduction to water, and charge separation across the membrane. Furthermore, cytochrome c oxidase could maintain a proton electrochemical gradient across the supported proteomembrane, i.e. the membrane system was proton tight, defining an interior particle compartment that is separated from the surrounding aqueous media. The use of this type of “artificial cells”, biofunctional cellular interface supported onto a colloid that has a connected interior cytoskeleton-like pore structure, for applications within drug delivery will be discussed.

Acknowledgement:
Research performed with support from EXSELENT, the Berzelii Center for Porous Materials, the Swedish Science Council (VR), the Centre for Controlled Delivery and Release (CODIRECT), supported by VINNOVA, the Knowledge Foundation and the Foundation for Strategic Research (SSF) and Industry, the Center for Biomembrane Research at Stockholm University, and the Knut and Alice Wallenberg Foundation.

NOVEL PROCESSING AND FORMING OF BIOMATERIALS

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There are several methods to freeform fine biomedical architectures Jetting methods such as three dimensional printing (3DP) and ink-jet printing are amongst these techniques and, in general, have been exploited to their limits. In contrast, electrohydrodynamic jetting is a powerful materials forming technology, which can create monolithic, composite and porous structures useful for a variety of advanced biomedical engineering applications. The electrohydrodynamic route offers better resolution than ink-jet printing and can generate both nanostructured architectures at the ambient temperature using a simple procedure. This process is the focus of this paper.

A fluid or a suspension or a liquid precursor is made to flow through needles at a controlled rate under the influence of an electric field. In the right flow rate-voltage regime a jet evolves from the tip of the needle and depending on the properties of the medium breaks into regular droplets. These droplets can be used for spraying, coating, threading or printing with or without a template, or can be made to spin into a fibre. We have extended single needle processing and forming to co-axial multi-needle forming, where media are subjected to electrohydrodynamic flow simultaneously to generate porous or encapsulated microstructures or bubbles, notably electrohydrodynamic microbubbling was pioneered by us.

This paper will describe and discuss recent developments, for example the use of co-axial multi-needle systems to uncover novel architectures, which can cause a step change in biomedical engineering advances, for example, in orthopaedic engineering, tissue engineering, imaging and therapeutics. A whole range of special devices are being prepared and explored in our laboratories, these devices are used to achieve a raft of new materials forming capabilities. The architectures generated in this way can be utilised to revolutionise, for example, controlled-release of drugs to encounter progression of disease.

AN X RAY TOMOGRAPHY STUDY OF AGGLOMERATE BREAKDOWN DURING PASTE FLOW

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The mechanical properties of ceramics frequently depend on the quality of the mixing process used to disperse the powder prior to shaping. This is true when extruding conventional particulate paste systems. In this study the influence of die shape and location are investigated for simple ram extrusion. In the past visualising the breakdown of individual agglomerates has relied on laborious sectioning but with the advent of higher resolution tomographic techniques it is possible to view the internal structure of ceramics non-destructively.

Pastes were formed from Kaolin, these pastes were stained to allow referencing of position and individual agglomerates formed from zirconia were placed in the paste system. They were placed at the centre, at the wall and at the midpoint, on a plain perpendicular to the direction of extrusion. Due to the difference in atomic number there is strong X-ray contrast and the agglomerate can be imaged in the paste. After extrusion the extudate was first found with simple macro X-ray imaging. Sections of interest were then tomographically examined and the dimensions of the agglomerate and its break up determined. To calibrate the images small dense cylindrical grains were also analysed. The relationship between deformation and the rate of that deformation on the breakup of agglomerates will be examined. The configuration of the die in terms of entry angle will be considered again to determine the influence on agglomerate removal. The position of the agglomerate in the barrel will also be factored into the discussion.

SYNTHESIS, COLLOIDAL STABILITY, PHOTOCATALYTIC AND ANTIMICROBIAL PROPERTY OF AG-DEPOSITED TIO_2 COMPOSITE NANOPARTICLES

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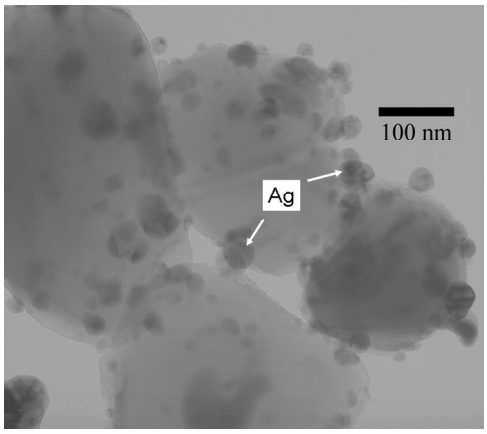
Silver nanoparticles prepared by a reverse micelle process were sequentially deposited on anatase-structured TiO_2 particles via an electrostatic layer-by-layer (LbL) adsorption alongside with a hydrophilic/hydrophobic interaction. The TiO_2 surface was first mediated by a preferential adsorption of poly(allylamine hydrochloride) (PAH) molecules, before being mixed with the Ag nanoparticles encapsulated in reverse micelle consisting of anionic surfactant of sodium bis(2-ethylhexyl) sulfosuccinate (AOT) in a hydrophobic isooctane solvent. The micellar nanoparticle coating renders a controllable coverage of the Ag deposition on the TiO_2 particles, and reduces particle aggregation which leads to an improved dispersion and colloidal stability. Rheology of the concentrated Ag-TiO_2 aqueous suspensions was examined by addition of an amphiphilic surfactant consisting of a 1,2,3-trihydroxybenzene group (pyrogallol) as a head group, and a water-soluable poly(ethylene glycol) (PEG) chain as a tail group. A tailored tail length was determined for an optimum dispersion. Finally, the photocatalytic disinfection of *E. coli* using the composite Ag-TiO_2 particles will be reported. The Ag nanoparticles are expected to facilitate channeling of charged carriers by reduction of electron-hole recombination, and to release Ag ions as well for the antibacterial effect.



ELECTROPHORETIC DEPOSITION OF ADVANCED FUNCTIONAL CERAMICS INTO COMPLEX SHAPES AND CONFIGURATIONS

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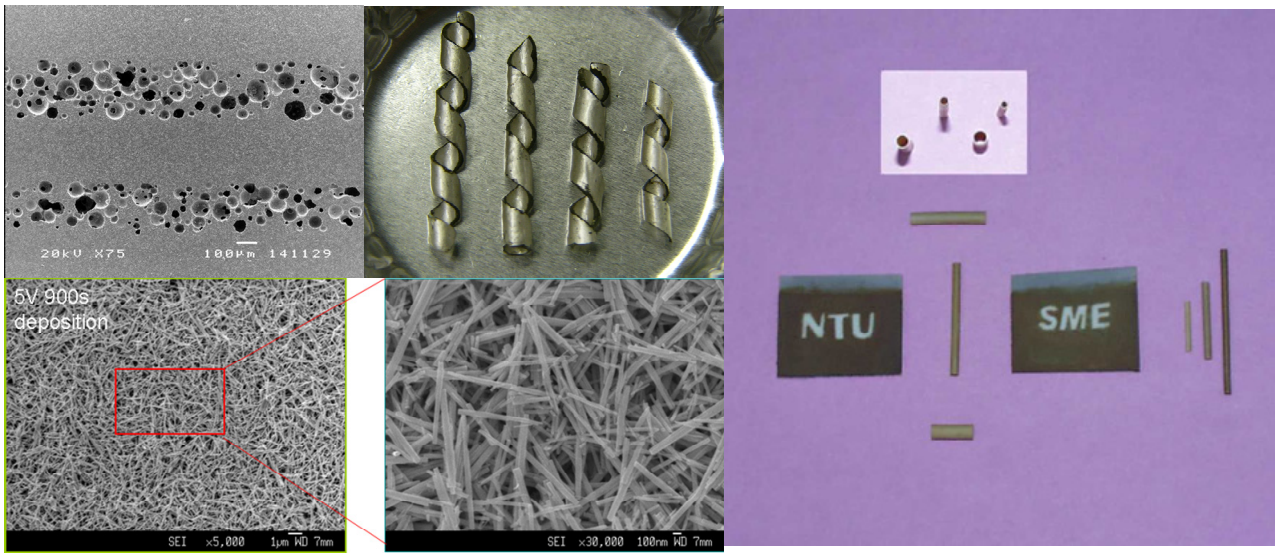
The application of Electrophoretic Deposition (EPD) to form various shapes such as tubes and helix, and various configurations such as multi-layered and porous structures, are presented. EPD is probably one of the most effective and efficient processing methods to assemble nano-particles into controlled shapes and configurations. In recent years, EPD of non-equiaxed materials, such as nanowires and nanorods, has attracted huge attention as the process could incorporate both functionality and structural enhancements/variations in the components. In the present work, the application of EPD on the above will be discussed, and their applications in advanced applications such as biomedical applications will be presented.



CO-EXTRUSION OF MULTILAYERED CERAMIC MICRO-TUBES

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Current manufacturing methods for tubular solid oxide fuel cells (SOFCs) involve multiple steps of extrusion and multiple steps of layer deposition and sintering, leading to high manufacturing costs. The aim of the work presented in this paper is to reduce the cost of manufacturing SOFCs. This is done by developing a method for manufacturing a five layered micro-tubular structure by a multi-billet co-extrusion process. With the implementation of continuous screw extrusion equipment, this co-extrusion process could easily be adapted into a fully continuous manufacturing process.

The co-extrusion process presented initially involves rheologically unifying five pastes made up of individual powder compositions. These pastes are then extruded as billets from separate extrusion barrels into a single nozzle, using a novel die design which does not require the use of a mandrel to form the tubular structure.

The sintered structure comprises four Ni/YSZ anode layers and a YSZ electrolyte layer, each layer being approximately 60 μm thick, forming a tube with an outer diameter of 3 mm and an inner diameter of 2.4 mm.

POROUS ALUMINA CERAMICS PREPARED WITH WHEAT FLOUR

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For more than one decade starch has held its position as one of the most popular pore-forming agents for the preparation of porous ceramics. Its possibilities and limits are relative well known. With the ever-increasing use of starch, however, economic aspects come into play as well. Wheat flour, whose main constituent is wheat starch, is considerably cheaper than pure native wheat starch (which is a refined product with non-negligible added value). Therefore, wheat flour (which is commercially available in several types or grades from fine to coarse and, finally, semolina) might be an interesting competitor of starch for some applications. This work deals with the possibilities of preparing porous alumina ceramics by using wheat flour as a pore-forming and body-forming agent. Due to the fact that wheat flour consists predominantly of wheat starch, the well-known starch consolidation casting (SCC) principle can be used, i.e. a flour-containing suspension can be cast into a non-porous (usually metal) mold and heated up to allow the starch fraction contained in the flour to swell. The first part of this contribution concerns the characterization of wheat flour by X-ray fluorescence analysis, differential thermal analysis and thermogravimetric analysis, including the mass-spectrometric analysis of gases evolving during the burnout of flour. The second part focuses on particle size analyses during the homogenization and swelling steps (suspension preparation and body formation). Different suspensions have been prepared containing varying amounts of submicron alumina powder (Almatis CT-3000 SG) and fine wheat flour. Green samples were prepared by casting into metal molds, heating up to 80 °C (SCC principle), demolding and drying, followed by firing according to a standard schedule (2 °C/min, 2 h hold at 1570 °C). The following parameters were systematically changed: concentration of alumina, concentration of flour and homogenization time. The influence of each of these parameters on bulk density and porosity is discussed, and selected sampled are characterized by image analysis and mercury porosimetry. The results of this work show that, in contrast to SCC using pure native starch, the microstructure of the resulting ceramics is strongly dependent on the homogenization time (time of shaking in a laboratory shaker). In particular, with increasing homogenization times the spatial distribution of bubble pores in the microstructure of the porous ceramic becomes more homogeneous, the size distribution more uniform, and the overall porosity increases (apart from assisting in deagglomerating the alumina powder, longer homogenization times are necessary to attain higher flour contents and to achieve better deagglomeration of flour particles, i.e. grains fragments, into starch granules). The reason for this behavior (in contrast to SCC using pure native starch) is that, apart from the well-known pores resulting from wheat starch burnout (median approx. 20 μm), another pore type occurs with flour, viz. pores resulting from the bubbles evolving during mechanical agitation. These pores are one order of magnitude larger (median approx. 200 μm) and result from the action of enzymes and proteins, which are usually present in flour (but not contained in sensible amounts in pure native starch). In this sense SCC with flour is in fact a combination of two shaping principles: SCC and foaming. Therefore it allows generally higher porosities to be achieved than with pure native starch. The porous ceramics thus prepared exhibit a hierarchical microstructure, consisting of at least three pore generations: large spherical pores from foam bubbles (pore body diameter approx. 200 μm), medium-sized, slightly oblate (from the large so-called “A-fraction” of wheat starch), pores from burnt-out starch granules (pore body diameter approx. 20 μm), and the small interconnecting openings or channels (pore throat diameters of approx. 2 μm). Additionally, very small interstitial pores between the submicron ceramic powder (with pore body diameters of approx. 200 nm and interconnections of approx. 20 nm) may be achieved by partial sintering.

Acknowledgement:
This work was part of the frame research programme MSM 6046137302 “Preparation and research of functional materials and material technologies using micro- and nanoscopic methods”, supported by the Ministry of Education, Youth and Sports of the Czech Republic.

OSMOTIC DRYING OF GELCASTED BODIES PREPARED FROM FINE ALUMINA POWDER

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Gel casting is an attractive ceramic forming process for making high-quality, complex-shaped ceramic parts. It utilizes the advantage of colloidal processing which is of exceptional importance for reliable and defect-free processing of fine and nanometer-sized powders with their tendency to agglomeration. However, the inherent lower loading of suspensions prepared with fine powders results in gelcasted bodies which are subjected to large shrinkage during drying and are susceptible to warpage and cracking especially in bodies with large wall cross-sections. Osmotic drying of gelcasted bodies in a liquid desiccant offers the possibility to remove high local stresses responsible for such defects formation. The osmotic drying totally prevent from capillary stresses to come in action. During osmotic drying the body is immersed in an appropriate polymer solution and the gel acts as a semipermeable membrane. Due to different chemical potential of water in the gelcasted body and in the surrounding solution, the water is removed from the body. Osmotic pressure governs the water removal from gelcasted bodies and acts homogenously over the whole body. The osmotic pressure can be predicted and easily controlled.

In this investigation gelcasted bodies prepared from alumina powder with mean particle size of 100 nm were immersed in water solutions of various polyethylene glycols (PEG) with molecular weight ranging from 1000 to 35000 daltons. Up to 27 % of water content could be removed from the gelcasted bodies in 40 wt% solution of PEG 35000. It was also found that PEG 1000 was less effective desiccant than the PEGs with higher molecular weight even though the osmotic pressure was similar. The smaller PEG molecules penetrated into the gelled bodies and reduced the dewatering. Moreover, the penetrated PEG molecules affected the pore structure of green bodies and in this way influenced the sintering behaviour. The time dependence of dewatering and the effect of the size of gelcasted bodies were investigated and correlated with green body structure and sintering behaviour in order to optimize the osmotic drying process. Some successful examples of osmotic drying will be presented.

Invited Lecture and Session 4

Chair persons: P. Colombo &. M. T. Colomer

CAPSULES AND FOAMS FROM NANOPARTICLES

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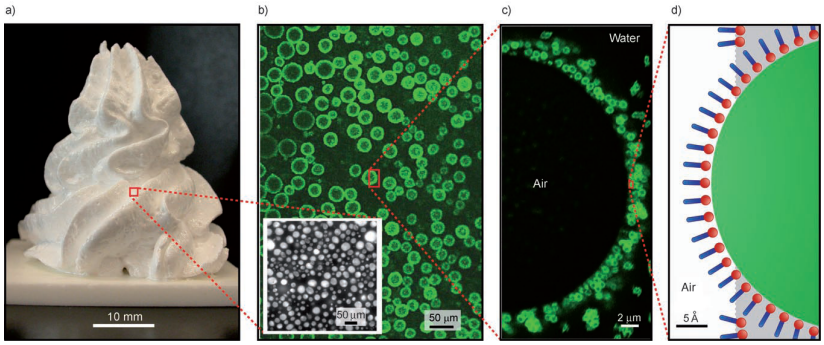
Novel materials can be derived via colloid chemistry routes from particles that are driven to a liquid/gas or liquid/liquid interface. To irreversiibly adsorbe particles (metals, ceramics, polymers or cements) at gas/liquid or liquid/liquid interface their surfaces are lyophobized through the adsorption of short-chain amphiphilic molecules and used to stabilize foams [1], emulsions [2,3]. This functionlization of the surfaces enables to create foams, emulsions and composites of different microstructural architectures. New highly porous ceramics [4], metals and polymers are possible with air contents up to 98%. These materials have many potential applications from insultation boards to bone replacement and electrets. Hollow and filled particle capsules can also be prepared by this method and applied in paints and as drug release agents [5]. The same principles again can be used to create free standing, transparent ceramic films with aspect ratios of 1:20 000.



Foams and capsules derived from functionalized particle suspensions.

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PREPARATION AND PROPERTIES OF ULTRAHIGHLY POROUS SILICON CARBIDE

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Porous silicon carbide has attracted much attention for the industrial use such as particulate filter, molten metal filter, catalyst support and membrane support because of its excellent thermal stability, thermal shock resistance as well as chemical stability at elevated temperatures. For these applications, high porosity is one of the most important factors, because that is expected to result in high permeability and effective filtration due to the low-pressure drop.

In the present study, silicon carbide with ultra high porosity and oriented micrometer-sized cylindrical pores was prepared using a novel gelation-freezing method. Gelation agent, water for pore forming agent, and raw powder were mixed, and gelation of the obtained slurry was performed. By casting the slurry into various shaped molds, disk, rod, plate and tube could be formed. After gelation, the obtained gel was frozen at -10 to -70°C, dried using a freeze drier under vacuum, degreased at 600°C and sintered at 1800°C for 2 hr under flowing Ar.

The obtained porous SiC showed high porosity around 88%, which was measured by Archimedes method or calculation of weight/dimension. The effect of sintering temperature on porosity was observed. Porosities were observed to decrease with increasing sintering temperatures. Further, porosities were also observed to be directly affected by the slurry concentration. Thus, porosity could be varied by sintering temperature and slurry concentration. On the other hand, closed porosities for all samples were less than 1.0%. This suggests that the water was almost completely converted into pores that were interconnected.

Seen from SEM micrographs of fracture surfaces of porous SiC in the perpendicular direction to the freezing direction, the observed pores were honeycomb-shaped with micrometer-sized cells, in which the shape was obviously different from that of foam, pore-forming agents and those observed in other freeze-casting routes. This cylindrical structure was observed throughout the porous body, with the exception of the side and bottom of the porous body. The average diameters of the cells were 34-147μm, which could be varied by freezing temperature. The number of cells in the cross section was about 47-900 cells/mm^2.

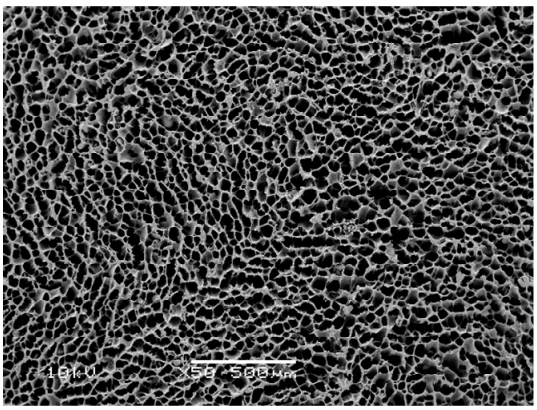

Fig. SEM observation of porous SiC in the perpendicular direction to the freezing direction

Compressive strength of a porous SiC with porosity of 87% was 6MPa, which was higher than that of porous SiC prepared by other method. And, air permeability of the porous SiC showed 2.3^{-11} - $1.0^{-10}m^2$, which was dependent on pore size of porous SiC. Air permeability result is almost consistent with that of calculated ideal value, which means excellent pore connectivity and pore orientation. Furthermore, the permeability was much higher than those required value for DPF ($10^{-11}$to $10^{-12}m^2$).

This novel method can provide porous SiC with various shapes and is thought to be a potential process for a number of applications such as filters and catalyst supports.

ICE TEMPLATING: A VERSATILE PROCESS TO PRODUCE FUNCTIONALLY GRADED CERAMIC FILTER MEDIA

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Owing to their unique structure and properties, ice templated ceramics have in less than a decade drawn attention as high performance cellular ceramic for various applications as e.g. bone substitutes in hydroxyapatite. One major attractiveness of this templating technique (namely freeze casting of a suspension) stems from its versatility. Depending on the solvent used, different pore morphologies (namely dendritic or lamellar) can be achieved. The pore structure can be further tailored by controlling the freezing conditions, the suspension solid loading and the ceramic particle size.

In the present work, the use of this innovative process for the production of functionally graded materials was evaluated. In particular the role of the binder as well as that of the mold dimensions was investigated. It was shown that the binder is not only crucial to guarantee the cohesion of the highly porous green parts after sublimation of the water, but that its nature and concentration directly affects the morphology of the ice crystals developed during the solidification step. Under optimized experimental conditions, homogeneous lamellar pore structures were developed over relatively large zones, irrespective of the solid loading of the suspensions used. It was also shown that the mold dimensions affect the pore structure as well. The main features of the various structures developed (pore channel size, lamellae thickness …) as determined using scanning electron microscopy, were correlated with the set of experimental conditions.

Finally, a filter medium with graded pore-size distribution while keeping the directionality of the porosity was prepared. The resulting morphology of the porous network was observed using X-ray tomography and will be presented.

Acknowledgement:
One of the authors, Moeko Tabata, wishes to acknowledge the support of the "Vulcanus in Europe" training programme for Japanese students.

POROUS ECOMATERIALS BASED ON INDUSTRIAL WASTE AND WOOD TO BUILDING MATERIALS

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Currently, there is a political as well as a societal demand for products, which require less energy for the manufacturing, process and are easy to recycle. The new materials have to display properties analogous or even improved with respect to those of existing materials. Geopolymers are amorphous three-dimensional alumino-silicate binder materials, which were first introduced to the inorganic cementitious world by Davidovits in 1978. Geopolymers may be synthesized at room or slightly elevated temperature by alkaline activation of alumino-silicates obtained from industrial wastes, calcined clays, natural minerals or mixtures of two or more of these materials. They need less energy than cement to be synthesized and resist under high temperature.

Two kinds of composite samples were prepared. The first consisted on an in situ material composite based on raw mineral. They were synthesized from a solution containing dehydroxylated kaolin and KOH pellets dissolved in potassium silicate. Fibers, wood chips or sawdust were then added and the mixture was transferred to a polyethylene mould sealed by a top and placed in an oven at 65°C during 24 hours. After drying, some samples were calcined at 600°C for 2 hours in order to create porosity and to eliminate the organic particles in the composite. The second composite was prepared from a solution containing fume silica as industrial waste, dehydroxylated kaolin and KOH pellets dissolved in potassium silicate. The mixture will be then deposited on wood and place for 24 hours in an oven at 65°C. These samples were characterized without thermal treatment.

The presence of the organic materials allowed to obtain mechanical reinforced composites or materials with a control of porosity. The introduction of silica fume leads to an inorganic foam, which adheres naturally and fastly to the wood. The characterization of this compound by SEM and EDX analysis reveals a migration of some species such as the element potassium. This phenomenon proves the chemical nature of interaction between foam and cellulose (Figure 1 (b), (c)). Double-shear tests show efficient adhesion properties, with shear strength of 1.3 MPa.

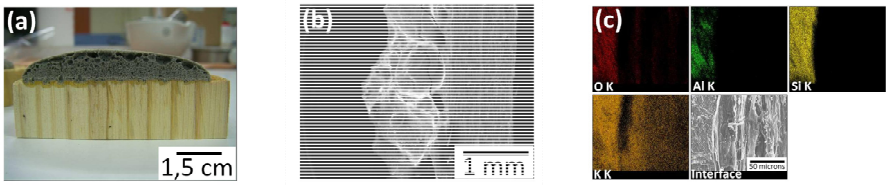

Figure 1: (a) Foam-wood composite, (b) foam-wood composite Interface, (c) X-ray mapping of wood/geomaterial interface.

This study put in evidence the use of industrial waste to create porosity and protect the wood by geoplymerization reaction. The thermal properties by the "Flash Laser" method or by a heat flux sensor and the mechanical properties by double-shear or by compression of all composites in progress seem promising.

POROUS POLYMER DERIVED CERAMIC COMPOSITES DECORATED WITH IN-SITU GROWN NANO-STRUCTURES

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Commercially available preceramic polymers such as polysiloxane and polysilazane, were used to produce open cell ceramic composites by varying the parameters such as the type and the ratio of the precursor and blowing agent, pyrolysis temperature and atmosphere. Nano-structures (nanorods, nanowires and nanobelts) were obtained directly on the surface of the macro-porous components upon heating by catalyst-aided reactions of gases deriving from the pyrolysis of the preceramic polymers. Various characterizations were performed on the resulting components, including the measurements of the specific surface area, open/closed porosity, phase constitutions and the microstructural evolutions by SEM and TEM.

POLYMER DERIVED CERAMICS FOR BEARING APPLICATIONS

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High precision shaping of polymer derived ceramics for bearing applications was investigated. Polymethylsiloxane was granulated with the active filler ferrochromium (FeSiCr) and the passive filler SiC. Rectangular green bodies were warm pressed by remelting at 240 °C and a pressure of 9 MPa in a crosslinked thermoset preform. High precision green machining was conducted on a Computerized Numerical Control (CNC) high speed cutting machine providing an accuracy of 10 µm. After machining the components were debinded and pyrolyzed at 950 °C and subsequently heat treated at 1275 °C under N_2 pressures up to 5 MPa and exposure times of 90 min and 180 min. Total linear shrinkage remained below 7 %, while the residual porosity decreased to 3 % for 25 vol.-% FeSiCr. The mechanical properties of the bearing material were measured: bending strength 300 MPa, fracture toughness 3.4 $MPam^{-0.5}$, hardness 9.5 GPa and elastic modulus 190 GPa. The optimized material system was used for the fabrication of bearing components such as cylindrical rolls and linear bearing systems, Figure 1.

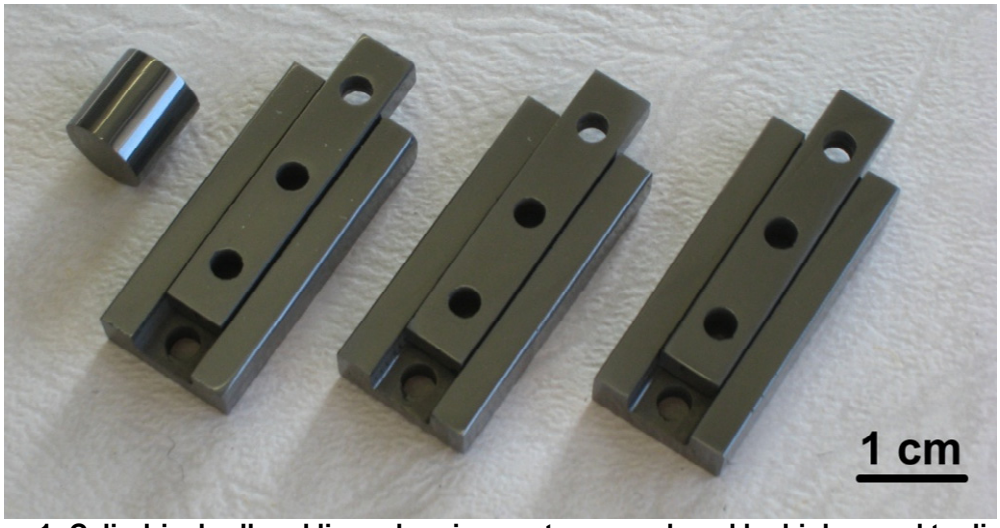

Figure 1: Cylindrical roll and linear bearing systems produced by high speed tooling of thermoset preforms.

[1] M. Steinau et al.. **Polymer-Derived Ceramics for Advanced Bearing Applications**. Adv. Eng. Mat., 10, 12, 1141-1146, 2008.

JOINING OF PRECERAMIC PAPERS FOR THE PRODUCTION OF FILTER SYSTEMS

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Macro-porous paper-derived corrugated ceramic structures show promising properties for use in fluid filter systems for water cleaning [1]. Alumina-loaded preceramic papers were produced and shaped to form corrugated filter structures. Shrinkage during conversion to alumina ceramics, mass loss, microstructure and porosity of the corrugated structures were measured. Emphasis was put on defect free joining of layer segments to achieve superior mechanical loading capacity of the corrugated filter structures.

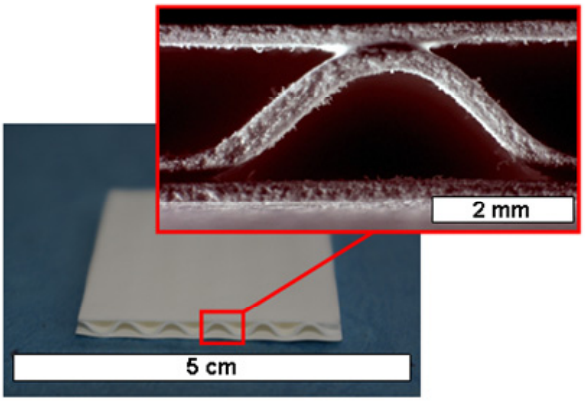

Figure 1: Corrugated alumina filter produced from preceramic paper.

[1] Travitzky, N. et al. 2008. Preceramic Paper-Derived Ceramics, Journal of the American Ceramic Society 91 (11): 3477-3492

Tuesday 17th November.

Invited Lecture and Session 5

Chair persons: G.L. Messing &. A.J. Sánchez-Herencia

POROUS CERAMIC STRUCTURES AS A TOOL FOR MANY APPLICATIONS

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Due to an inventive use of their pore architecture, porous ceramics find their way in many applications. Indeed, they can be used as membranes, filters, catalyst carriers, heat exchangers, scaffolds for bone regeneration, sensors, thermal insulation, aerospace applications, gas combustion burners, lightweight materials and art design.

Nevertheless there is no such one general technique for the fabrication of porous ceramics because each application has its own requirements for pore size distribution, pore structure, total porosity, and mechanical or other application related properties.

For this reason, we present an overview of the manufacturing routes which are followed for the preparation of different types of porous ceramics with pore sizes in the vast range from 1 nm to a few mm.

Ceramics with very small nanopores <2nm, are used in the selective separation toplayers of ceramic membranes. Mostly they are being produced by the sol gel technique on a multilayer substrate with a gradient in pore size. A second kind type of porous ceramic materials with very large pores, are the foams. There are many methods to produce such structures, mostly based on sacrificial templates or on direct foaming techniques, e.g. PU- replica technique, gel casting or hollow bead method.

Free forming techniques are used to produce structures with well controlled pore sizes, the periodical porous ceramics, e.g. print techniques, robocasting, fused deposition and selective laser sintering. Different types of ceramic foams are shown in the figure below.

Application of extra functional coating, further enhances the possibilities for use of these porous structures. Well known examples are a catalytic wash coating on a foam or honeycomb structures, a calcium phosphate coating on a titanium scaffold for bone regeneration, TiO_2 coatings for photocatalytic applications,…

In this contribution we illustrate the performance of a variety of porous ceramic materials in combination with the results from several characterization methods like µCT, FESEM, IA, MIP and mechanical testing.

TAPE CAST POROSITY-GRADED PIEZOELECTRIC CERAMICS

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Porous graded piezoceramics are of interest for ultrasonic applications, as a consequence of the gradual decrease of the acoustical impedance, that improves the transfer of acoustical energy to water or biological tissues assuring at the same time an high piezoelectric response. Moreover, reducing the material thickness (< 1 mm) let to reach higher resonance frequency, and, as a consequence, high resolutions at a reduced depth of field for transducers applications.
In this work a functionally graded porous Nb-doped PZT was produced by tape casting, the most used technique for the production of multilayer ceramic packages of thickness less than 1 mm. Each step of the production process (slurry formulation, lamination and thermal treatments) were thoroughly investigated. The porosity gradient was formed by in situ sintering layer-stacked green tapes of stepwise-varied contents of carbon black (CB).

Slurry formulations with CB concentration between 3 and 43 vol% were optimized to produce each of the green sheets. Linear relationships between CB and solvent, binder and plasticizers amounts were found. Moreover the additional polymer, needed to well-disperse CB, was found to increase proportionally with CB amount. As a consequence of this work, slurry formulations that can lead to defect-free green tapes, can be easily planned for whatever CB concentration.

The conditions necessary to laminate 6 layers of different CB content were optimized by tailoring the binder to plasticizer volume ratio of each single green layer. Finally, by gradually increasing CB amount, adjusting the binder burnout procedure, and tailoring the multilayer thickness, cracks and delaminations were eliminated leading to a well controlled microstructure (Figure 1).

The optimization process led to a well developed, crack-free porosity-graded multilayer less than 400 μm thick and with porosity along the thickness ranging from 10 to 30 vol %. The application of a load during the heating treatments was absolutely required to obtain warpage-free planar multilayer specimens. The piezoelectric properties of these materials were found to be suitable for ultrasonic applications.

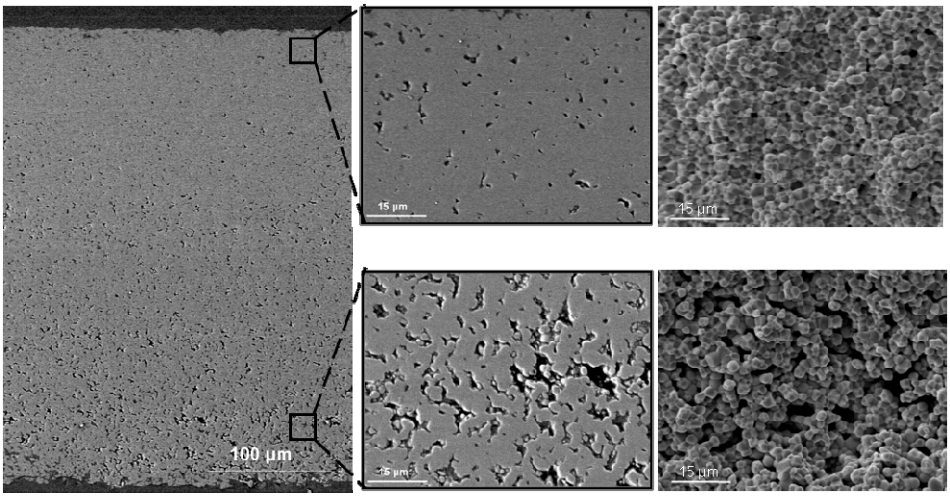

Figure 1 SEM micrographs of the microstructure of the porous graded PZTN material.

A NOVEL TEMPLATED GRAIN GROWTH APPROACH FOR THE PROCESSING OF ⟨001⟩-TEXTURED PMN-PT CERAMICS

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The processing of textured ceramics of $Pb(Mg_{1/3}Nb_{2/3})O_3$-$PbTiO_3$ (PMN-PT) with compositions around the morphotropic phase boundary (MPB) has become a key issue over the last years in the effort of obtaining low-cost materials with piezoelectric properties comparable to those of single crystals [1]. Improved properties have been obtained in textured PMN-PT ceramics fabricated by templated grain growth (TGG), using anisometric templates of isostructural phases, such as $BaTiO_3$ [2]. High aspect ratio PMN-PT particles are not easy to obtain, and textured PMN-PT ceramics have also been processed using cube-shaped microcrystals grown in PbO flux as templates [3].

In this work, the feasibility of a novel homogeneous-TGG approach for the processing of textured ceramics of PMN-PT is demonstrated. Novelty rests on the use of a nanocrystalline powder synthesized by mechanochemical activation for the matrix, and also for obtaining cubic templates by exaggerated grain growth and TGG processes. Templates were (100) faceted cube-shaped microcrystals with average sizes of 30 and 12 μm [4], which were successfully aligned by tape casting for the processing of ⟨001⟩-textured PMN-PT ceramics. The approach used involves only conventional ceramic technology from a single source powder to obtain textured PMN-PT piezoceramics.

The rheological behavior and tape casting performance of the ethanol based slurries are presented [5]. Suspensions were prepared up to solid contents of 32 vol%, significantly higher than those usually reported for non-aqueous systems. The replacement of toxic solvents commonly used for tape casting of functional ferroelectric materials, often toluene, with ethanol, which is a safe and environmentally friendly solvent, is an additional advantage. The effect of a number of processing parameters, such as those of the lamination of tape stacks, on texture is established. An advanced combined approach, using quantitative texture analysis and the Rietveld method, is used to analyze the X-ray diffraction data in order to obtain accurate results on the global texture of the ceramics. The electrical and electromechanical properties of the materials are also presented.

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[2] S. Kwon et al., J. Am. Ceram. Soc. **88**, 312 (2005).
[3] M. P. Thi et al., J. Eur. Ceram. Soc. **25**, 3335 (2005).
[4] H. Amorín et al., J. Eur. Ceram. Soc., **28**, 2755 (2008).
[5] H. Amorín et al., J. Am. Ceram. Soc., **92**, 996 (2009).

PROCESSING AND CHARACTERIZATION OF TEXTURED MULLITE CERAMICS FROM PHYLLOSILICATES

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The implementation of ceramics processes that promotes the organization of grains and grains interfaces within microstructure is favorable to mechanical properties of materials. In this work, new ceramics are obtained from a compact of oriented layers of phyllosilicates, kaolinite or/ and muscovite. Preferred orientations of particles in powder compacts are favored by centrifugation and aqueous tape casting processes, and pressed samples served as reference. During sintering, muscovite particles act as template for the achievement of an organized microstructure with mullite. A small amount of muscovite templates is necessary (10wt%), but the organization degree of microstructure depends strongly in the parameters of shaping processes and in the densification stage during the thermal treatment.

After sintering at 1350°C-1410°C, the microstructure of samples was characterized by X-ray diffraction, Quantitative Texture Analysis (QTA) and analyze of SEM images. The fracture strength was obtained by biaxial flexure test, the Young's modulus by nano-indentation and by ultrasonic testing, toughness was measured by the Vickers' method.

From the comparison of results of the different shaping processes, a microstructure-relation with mechanical properties is shown. Beside the effect of initial muscovite crystals and their orientation within powder compacts, mechanical properties are closely related with porosity.

For centrifugation, QTA and SEM show that mullite crystals in microstructure are oriented quasi-parallel to centrifugation direction and the important role of process parameters in the organization degree is evidenced. Mechanical properties are closely related to the organization degree of mullite crystals that is consistent with the development of an interconnected mullite network within the microstructure. At the microscopic scale, Young's modulus by nano-indentation in directions parallel and perpendicular to layers evidences an anisotropic behavior of mechanical properties.

Similar results of mechanical properties and microstructure are obtained with samples from tape casting and pressing processes since highly organized microstructure and low porosity are obtained. The specific and important role of residual porosity is also shown with the comparison of mechanical behavior of different samples from centrifugation, tape casting and pressing.

This work is funded by the European Community (European Social Fund and FEDER) and the Limousin Region.

ELABORATION OF $LA_{1-X}SR_XFE_{1-Y}GA_YO_{3-\Delta}$ MULTILAYER MEMBRANE WITH SR SUBSTITUTION GRADIENT BY TAPE-CASTING AND CO-FIRING

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The catalytic membrane reactor (CMR) is a promising solution for the production of fuel from natural gas, via gas to liquid process (GTL). This reactor allows to produce syngas from natural gas in a single step. The main element of this technology is the ceramic membrane, which on the one hand, separates oxygen from air and, on the other hand, allows the partial oxidation of methane from natural gas in hydrogen and carbon monoxide.

The CMR performances, for partial oxidation of methane, mainly depend on the oxygen semi-permeation properties of membrane materials and on the membrane architecture. This work presents the elaboration by tape casting lamination and co-firing process of a membrane consisting of a multilayer architecture of $La_{1-x}Sr_xFe_{1-y}Ga_yO_{3-\delta}$ dense membrane with Sr substitution gradient. The aim of the multilayer membrane is to establish the best compromise between oxygen semi-permeation performance and thermo-mechanical properties in using conditions.

In order to avoid membrane cracking during the sintering and under working conditions, the material of the multilayer was identified and adapted to have the same shrinkage during the co-firing and a thermal expansion coefficient similar to the membrane material. This study gives the main key of the development of multilayer membrane.

Another way, this work presents the elaboration of a crack free $La_{0.8}Sr_{0.2}Fe_{0.7}Ga_{0.3}O_{3-\delta}$ (LSFG8273) dense thin film with a porous layer on the both membrane faces. In the porous layer, the development of surface exchanges on the both membrane faces increases the performances of membrane in terms of oxygen permeation. This suggests that the limiting mechanism is oxygen surface exchanges and not the oxygen diffusion in volume.

These membrane architectures lead to an enhancement of the oxygen semi-permeation flux compared to traditional membranes with excellent thermo-mechanical properties in working conditions. Indeed, the multilayer membrane with Sr substitution gradient has a beneficial influence on thermo-mechanical properties of the membrane.

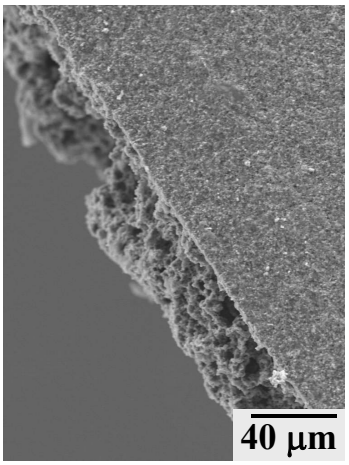

Figure : Microstructures of the LSFG8273 membrane with porous layer for CMR application.

*This work was supported by Air Liquide compagny, french a*gency for environment and energy management *(ADEME) and french national centre of scientific research (CNRS).*

PROCESSING ROUTE TO GENERATE AND DIRECTLY TAPE CAST NANO-SIZED α-Al_2O_3 POWDERS

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Tape casting is an established low cost process for the manufacture of large areas of thin ceramic sheets of controlled thickness. These sheets are the basic product for substrates or ceramic multilayer devices like capacitors, inductors, high integrated circuits, actuators and gas sensors. For the fabrication of tape cast products powders with average grain sizes between 1 and 3 µm are typically used. The enhanced availability of nano-sized powders increased the interest in the evaluation of these powders concerning their advantages in processing and properties. In case of alumina, nano-sized alpha-alumina powders are not available, but transition aluminas.

This work describes the generation of nano-sized α-Al_2O_3 powders by using stirred media mills and well-dispersed suspensions. The chosen solvent and dispersing agent were suitable for the subsequent tape casting process. By increasing the solid loading of the milling suspensions, these suspensions were directly processed to tape casting slurries to avoid any drying of the stable suspensions. The obtained slurries of nano-scaled α-alumina particles (< 60 nm) were tape cast by using the doctor-blade technique.

In this study, the overall process chain beginning from the production of the highly filled suspensions of nano-scaled α-alumina particles by mechanical breakage, and its direct processing via tape casting will be discussed. The effect of nano-sized particles on the properties of the slurries, the casting process, the sintering behaviour as well as on the final properties of the sintered tapes like surface roughness and mechanical strength will be presented.

Acknowledgement:
The authors want to thank the Arbeitsgemeinschaft industrieller Forschungsvereinigungen "Otto von Guericke" e.V. (AiF), Germany for their financial support of this work (AiF-ZUTECH project 194 ZN).

A MULTI-SCALE SIMULATION MODEL FOR TAPE CASTING

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Tape casting is the preferred shaping technique to produce large, thin ceramic tapes. In this process a slurry, containing a solvent, polymeric additives and the ceramic powder, is cast on a moving substrate by utilizing one or two blades. The resulting tapes are subsequently dried and sintered. Recently, they have gained traction in the use for complex multi-layer circuits. Here, the circuits are directly printed on the tapes and sintered together with them at low temperatures (so called low temperature cofired ceramic technology, LTCC). The microstructure of the tapes, which is strongly influenced by tape casting, has a profound effect on the sintering behavior. Optimization of the process thus requires a quantitative understanding of microstructural evolution.

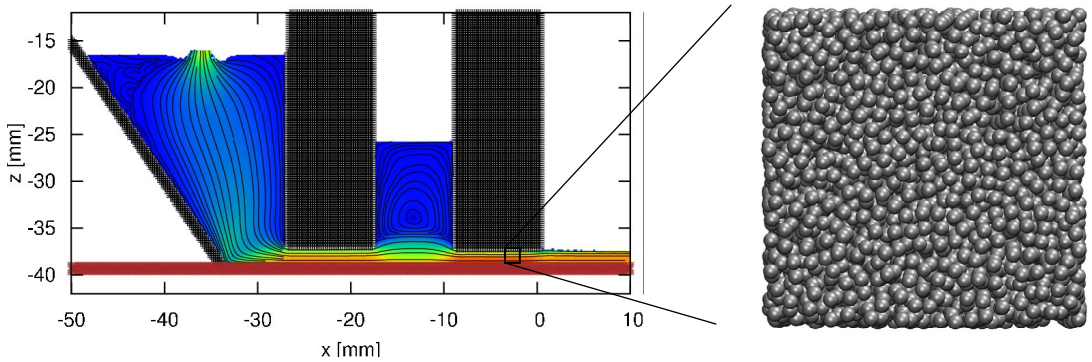

Figure 1: Simulating tape casting at the macroscopic scale (left image) allows extracting flow information which can be used to simulate representative volumes of individual powder particles (right image).

For this purpose a multi-scale simulation model for tape casting is proposed. The model allows considering both the macroscopic scale of the tape casting process, including the geometry of the casting chamber, and the microscopic scale at the level of individual grains. The concept of the multi-scale simulation method is depicted in figure 1: The flow field of the ceramic slurry (velocities, shear rates, viscosities etc.) can be extracted from macroscopic simulations of real geometries and is used as input for microscopic simulations of small, representative volumes. For the macroscopic simulations the particle-based Smoothed Particle Hydrodynamics method (SPH) is employed, with a newly developed viscosity model taking the non-newtonian slurry rheology into account. For microscopic simulations powder grains are modeled as individual particles by using the Discrete Element Method (DEM). Appropriate force laws describe the interaction between grains, polymers and the solvent.

First results of this new simulation technique are presented. Special focus is laid on investigations concerning the development of an anisotropic microstructure.

This work was supported by the Deutsche Forschungsgemeinschaft (DFG) under contracts No. Kr 1729/5-1 and Kr 1729/9-1.

Student Contest 2

Chair persons: C. Galassi &. C. Baudin

SHAPING AND DENSIFICATION OF β-TRICALCIUM PHOSPHATE BIOCERAMICS

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Since many years, calcium phosphate macroporous ceramics (tricalcium phosphate β-TCP, hydroxyapatite HA) were used as biomaterials for human skeletal repair and reconstruction, because of their chemical composition close to the bone one and their excellent biocompatibility. However, weak mechanical properties limit the use of these materials and require, frequently, osteosynthesis materials during surgical operation.

The objective of this study is to improve mechanical properties by sinterability optimization of the β-TCP powders. The influence of various parameters on the sinterability was studied such as:

- phosphocalcic powder stoichiometry,
- sintering time and temperature and post-HIP treatment impact.

β-TCP powders are obtained by precipitation technique in aqueous phase. A perfect control of powder synthesis parameters (Ca/P ratio, temperature, pH and ripening time) allows a close-control of powder chemical composition. The non-stoichiometry of β-tricalcium phosphate leads to the presence of secondary phases. This work shows beneficial effect of low level of HA phase in β-TCP matrix on the sample densification.

β-TCP sinterability is optimized according to powder characteristics (calcination), pressureless sintering parameters (time, temperature) and hot isostatic pressing treatment (post-HIP). Post-HIP, carried out under Ar/O_2 atmosphere (1050°C-150MPa), leads to obtain relative density higher than 99.9% resulting in transparent samples.

Hipped samples Young modulus and flexural strength were respectively 108 GPa and 133 MPa.

These various results were used for the shaping of macroporous materials which present a controlled porous architecture in terms of interconnection and macropores.

PROCESSING OF CARBON NANOTUBES CONTAINING SILICON NITRIDE NANOCOMPOSITES

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Composites containing carbon nanotubes (CNTs) have raised enormous expectation due to their potential for enhancing composite properties, particularly electrical, thermal and wear performances. Most papers deal with polymeric matrices and relatively scarce works have been done in ceramic matrices, especially for those ceramics like silicon nitride (Si_3N_4) that require very high temperatures for densification. However, Si_3N_4 materials present a wide range of technological applications, such as cutting tools and structural components, which demand good mechanical and wear properties at high temperatures.

In this work, some important aspects for manufacturing multi-walled carbon nanotubes (MWNTs)/Si_3N_4 composites were addressed. The first one was the functionalisation of the carbon nanotubes by surface oxidation treatments and by coating of nanotubes with a silica or a boron nitride layer. The second was to achieve stable suspensions of unbundled MWNTs, which was especially critical in the case of BN coated nanotubes, using aqueous or alcoholic media, ultrasonic bath, and the addition of dispersants. Other point we focused was the homogeneous dispersion of the CNTs within the matrix, which was achieved by independently dispersing MWNTs and Si_3N_4 powders in alcoholic media and, then, mixing them using sonication and mechanical stirring methods. Next step tackled was the complete densification avoiding nanotubes degradation, which was attained by spark plasma sintering at temperatures in the range of 1575-1600 ºC. The possible degradation of the treated MWNTs and the composites was analyzed using micro-Raman spectroscopy. Fully dense and homogenous MWNTs/Si_3N_4 composites with up to 9 vol. % of nanotubes were achieved using the original and the treated nanotubes. Composites were characterised in terms of crystalline phases and microstructure. In Figure 1, scanning electron (SEM) and transmission (TEM) micrographs of the composite containing 5.3 vol% of nanotubes are shown.

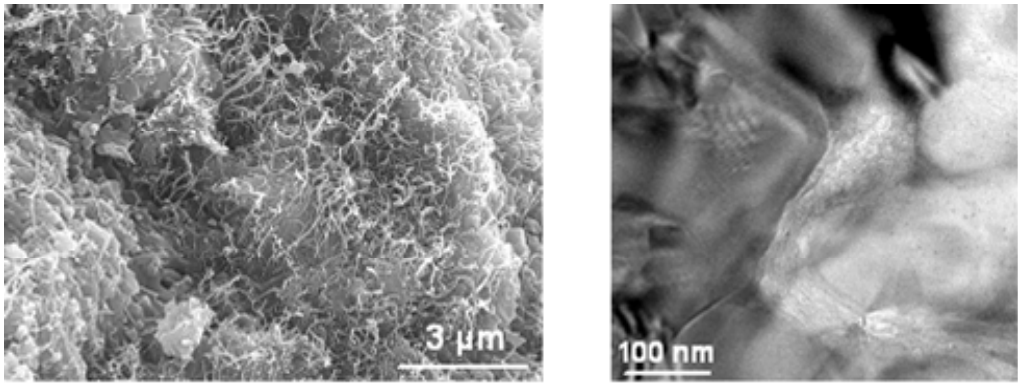

Figure 1. SEM and TEM micrographs of the MWNTs/Si_3N_4 composite containing 5.3 vol% of nanotubes

BINDER DISTRIBUTION DURING WICK-DEBINDING OF CERAMIC PARTS PREPEARED BY LPIM

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Binder removal from parts, formed by low pressure injection molding (LPIM) is delicate and time consuming operation, in which defects can be introduced that remain in the material to finished product. With the use of high porous wicking powder embedment, a partial thermal debinding can be utilized, which can considerably speed up the process and reduce many debinding flaws.

In this work we report on the kinetics of the partial thermal debinding in the wicking embedmend of ceramic parts prepeared by the LPIM. High solid content suspension for injection molding was prepared from Al_2O_3 powder and paraffin-based binder. The binder was thermally removed by the different time-temperature controlled procedures. After the process the distribution of the binder inside the samples was investigated.

We found that debinding takes place in two separate stages. In the first stage, the rate of binder removal depends on capillary extraction of binder into the surrounding wicking embedment. A characteristic binder distribution forms, with clearly distinct binder-depleted and binder-rich regions. The first is located in the center of the molded part, while the later occupies place between the binder-depleted region and the surface. With the progress of the debinding, the binder-rich region is shrinking until it eventually dissapears. Our hypothesis is that the binder-rich region is preventing almost all gaseous transport between the interior of the green parts and the atmosphere. Only after it dissapears, air can enter the interior and bulk oxidation can occur. At this moment the second phase of debinding starts, in which gaseous transport and reactions of binder degradation into volatile components control the rate of the process. A model, which is a modification of German model, has been proposed to describe evolution of binder distribution in the first phase of the process.

OPEN-CELL CERAMIC FOAM STRUCTURES PRODUCED BY DIRECT FREEZE FOAMING

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Direct foaming of ceramic suspensions by freezing and following freeze-drying is a new environmental-friendly process to produce open-cell foams. In contrast to existing processes this new method generates products without impurities in a relative simple and economic technical procedure.

Instead of the addition of blowing agents vacuum is applied to the suspension for foaming. During decompression the ceramic suspension is inflated by the partial pressure of the vapour and by entrapped air forming a cellular structure. The cellular structure is stabilized by freezing after reaching the equilibrium temperature in the vacuum. By freeze-drying the solvent is extracted by sublimation before subsequent debinding and sintering.

Main challenge of this process is ensuring a stable foam growth without cell collapse. In the experimental work a suspension was developed containing alumina powder and binders. In this study the optimal suspension composition was determined and main influences on the later foaming results are highlighted. Additional processing parameters were analyzed.

The produced samples were characterized by X-ray computed tomography (Fig. 1) and scanning electron microscopy (Fig. 2). CT-images revealed an irregular pore size distribution and open cells. Thin cell membranes of only one grain thickness were found by FESEM.

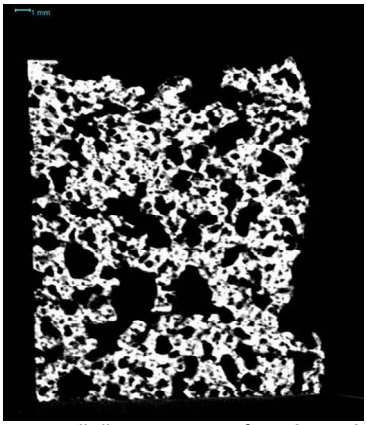

Fig.1: CT-image, cell diameter range from 0.5 to 3 mm

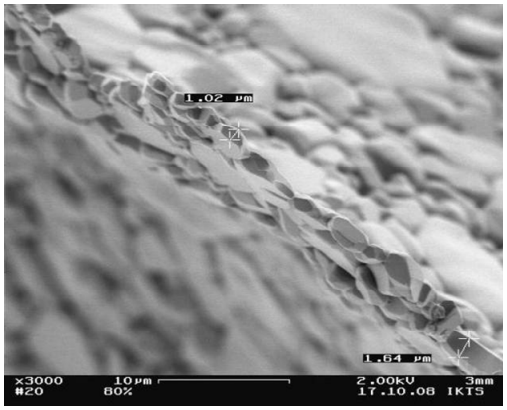

Fig.2 FESEM-image of a cell membrane with a thickness of 1 µm

ELECTROPHORETIC SHAPING WITH COAXIAL ELECTRODES

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The electrophoretic deposition (EPD) is an interesting and versatile process for shaping compacts especially for nanosized powders. Compacts of nanosized powders, either glasses or ceramics, can be completely sintered at much lower temperatures than conventional micron powders, if a sufficient green density can be achieved. Aqueous suspensions are favorable for industrial application due to the high polarity of water enabling high solid loadings and for environmental reasons. The problems with the decomposition of water can be easily solved with the membrane method. In this case the compact is deposited on the membrane, which is mounted between two electrodes. For many applications a local deposition might be of interest. Therefore the electric field has to be focused on a small point. This is a real problem in aqueous suspensions with a high electrical conductivity.

Previously, an electrode configuration was developed with two movable point electrodes facing each other (similar to Figure 1). Both electrodes are configured in such a way, that the cross sectional area is the only source of the electric field and the rest of the electrode is shielded (coaxial cable). This cable takes advantage of the Faraday cage effect because the internal metallic core is isolated with insulating material and a metallic grid. Due to the small dimensions of the electrodes, selective deposits can be attained. Another possibility is to replace one of the point electrodes by a steady plate electrode. In both arrangements structures with sizes in the millimeter range can be deposited. To improve the resolution, the electrodes must be positioned closer to each other. The limiting factor is, however, the formation of bubbles caused by water electrolysis at both electrodes, which disturb the electric field. Therefore the main problem that has to be solved is to avoid this bubble formation during the deposition process. This is not easy because the current density on a point electrode is high.

In this work, the formation of bubbles was suppressed by using a pulsed asymmetric alternating current. Additionally, modeling (finite element method) different tip shapes (Figure 2) of the point electrodes and calculating the electric field distribution was applied to improve the focusing of the electric field. In this way, deposits with smaller dimensions were obtained. Furthermore, different EPD parameters, electrode materials, profiles and motion rates were evaluated with aqueous suspensions and nanosized silica powders as a model system. Future possible applications for this technique are prototyping or commercial manufacturing of individual structures like those required in the dental industry.

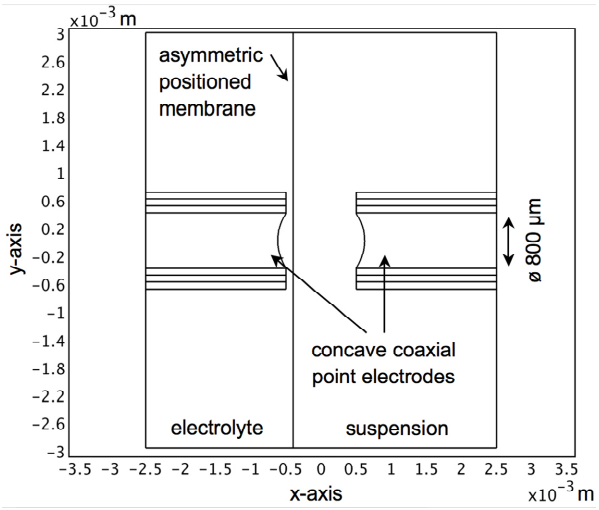

Figure 2: simulation results with 300 µm and 800 µm electrode from figure 1

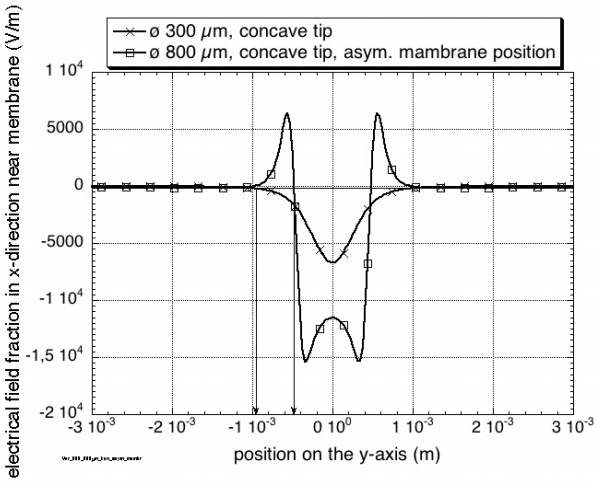

Figure 1: simulation setup, concave coaxial electrodes, membrane asym. positioned

INCORPORATION OF SEPIOLITE FIBER CONTAINING t-ZIRCONIA NANOPARTICLES TO A CERAMIC GLAZE

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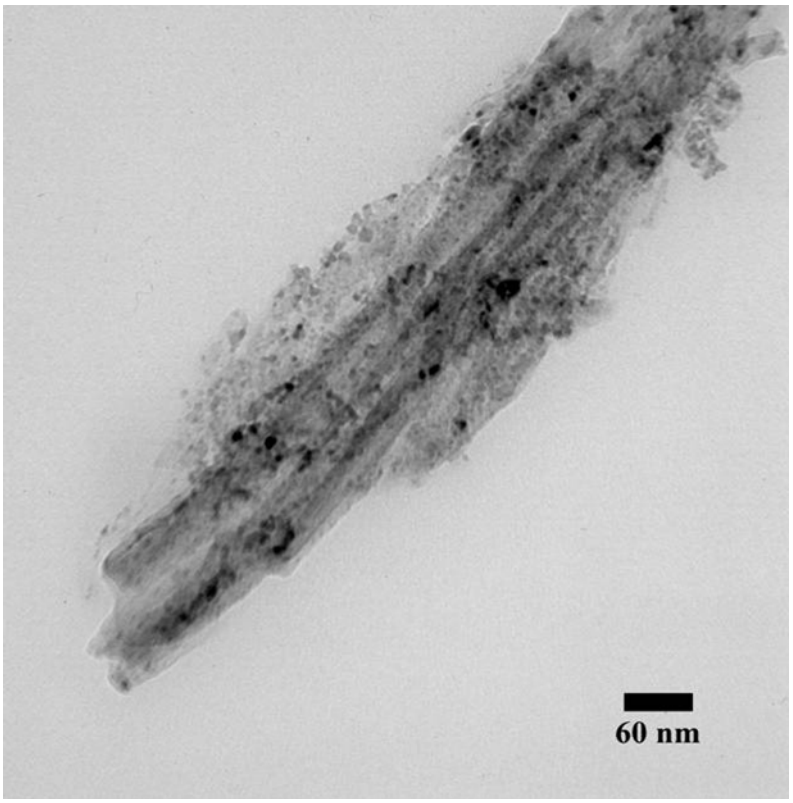
Sepiolite fibers were used as a carrier to obtain by a simple chemical route, a homogeneous distribution of t-zirconia monodispersed nanoparticles (< 10 nm).These sepiolite fibers containing t-ZrO_2 (15 wt.%) nanoparticles (Fig.1), were added (3-15 wt.%) to a commercial transparent ceramic glaze. The influence of the fraction of zirconia nanoparticles incorporated to the glaze at 1220ºC, on its optical properties and tribological behaviour has been studied.



Fibers containing t-ZrO_2 nanoparticles

PROGRESS IN THE ELECTROPHORETIC DEPOSITION (EPD) OF BIOACTIVE GLASS AND BIOACTIVE GLASS-BIOPOLYMER COMPOSITE COATINGS

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The electrophoretic deposition (EPD) technique was utilized for the fabrication of novel osteoconductive and bioactive coatings for biomedical applications. Firstly, bioceramic coatings were synthesized using 45S5 Bioglass® particles (mean size ~ 5 μm) suspended in an aqueous medium at different concentrations and pH values. The kinetics of the EPD process and its yield dependence on electric field, suspension concentration and pH were studied. A "design of experiments" approach based on Taguchi method was utilized to optimize the deposition rate. To increase the structural integrity of the bioactive glass coatings and to design novel functional coatings well adhered to the substrate which can be processed fully at room temperature, the electrophoretic co-deposition of bioactive glass-polymer composites was investigated. Natural biodegradable polysaccharides, i.e. chitosan and alginate, were considered. It was found that the processing window (electric field, deposition time, suspension concentration) for successful EPD of bioactive glass coatings at high rate is narrow. However when the macromolecules (natural polymers) were introduced to the suspensions, a significant increase in deposition rate of bioactive glass particles occurred due to the interaction of polymer macromolecules with the glass particles. Dependent on the pH and electric field, forced-deposition or massive-deposition of the glass particles can take place and homogeneous chitosan/Bioglass® and alginate/Bioglass® composites can be fabricated. The results also provided insight into the mechanisms and kinetics of electrophoretic co-deposition of bioactive glass and macromolecules, which will be discussed. The results of this study are relevant to design EPD experiments for successful deposition of bioactive glass and bioactive glass-polymer composite coatings useful in biomedical applications.

EFFECT OF SHAPING TECHNIQUE ON SINTERED DENSITIES OF SILICON CARBIDE

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In the presented work we have aimed to optimize the parameters for forming the silicon carbide ceramics. Colloidal processing of silicon carbide suspensions and electrophoretic deposition (EPD) was studied. EPD has been recognised as a very promising, popular and cost-efficient near-net-shaping technique for forming ceramic green parts. We compared the obtained results with the results of uniaxially pressed pellets which were sintered at the same conditions.

For the zeta-potential increase of SiC particles suspensions, pH value was adjusted by NaOH or a deflocculant (CTAB, cetyl trimethyl ammonium bromide or TMAH, tetramethyl ammonium hydroxide) was added. Different voltages were applied (up to 30 V/cm) and in order to avoid electrolysis during EPD various electrodes were tested.. The effect of zeta-potential, pH and conductivity of suspensions on the properties of the suspensions and of the deposits were observed. Green density, pore size distribution and homogeneity of deposits formed from suspensions with different compositions at various voltages were compared with those for green parts produced by conventional techniques.

The SiC samples without sintering additive were pressureless sintered at two different temperatures (2000 °C and 2100 °C) in vacuum. Microstructures were analysed using scanning electron microscope and mechanical properties were measured. The results indicate that the shaping technique significantly affects the density and functional properties of the sintered SiC ceramics. Electrophoretic deposition enables preparation of sintered parts with higher densities and hence better mechanical properties than dry pressing providing that well dispersed suspension with high zeta-potential and moderate conductivity is used. The highest densities of green and sintered ceramics were obtained by using TMAH as a deflocculant.

PROCESSING AND TAPE CASTING FROM COLLOIDAL SUSPENSIONS OF DOPED LANTHANUM CHROMITE SYNTHESIZED BY COMBUSTION SYNTHESIS

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Lanthanum chromite ($LaCrO_3$) is currently the most studied material for applications such as solid oxide fuel cell interconnector (SOFC). The complexity of microstructures and geometries of SOFC devices, which are usually built-up by lamination of the different constitutive layers, make it necessary a precise control of processing parameters to get the desired combination of properties. Much effort has been devoted to the processing of electrodes and electrolytes but the other layers, such as that of interconnecting material, have received scarce attention.

This work deals with the optimisation of surface properties and rheological behaviour of concentrated suspensions of Sr and Co-doped $LaCrO_3$ and the subsequent tape casting to produce homogeneous thin sheets.

The starting powder was produced by combustion synthesis from the corresponding nitrates and had a final composition of $La_{0.80}Sr_{0.20}Cr_{0.92}Co_{0.08}O_3$. These powders had a complex surface behaviour and readily dissolve at acidic pH. Then, concentrated tape casting suspensions were prepared to solids loading of up to 58 wt% in pure ethanol and dispersed with commercial copolymers (Hypermer, KD1). The binding system (BS) consisted of a mixture of a binder (polyvinyl butyral-co-vinyl alcohol-co-vinyl acetate (PVA-PVAc) and two plasticizers, polyethyleneglycol (PEG400) and benzilbutylphtalate (BBP). The content of binding system was 10 and 15 wt% and binder/plasticizer ratios (B/P) of 1/1, 2/1 and ½ were used.

The rheological behaviour of all prepared slurries was studied with a rheometer (Haake RS50, Germany) operating under control shear rate (CR) and control shear stress (CS). Tape casting experiments were performed varying the blade gap between 200 to 1000 µm at casting speeds of ~5 and ~10 cm s^{-1}. The cast tapes were sintered at 1600ºC/4h. Microstructural observations were performed on green tapes using scanning electronic microscopy (XL30-Philips) on as cast upper and bottom surfaces.

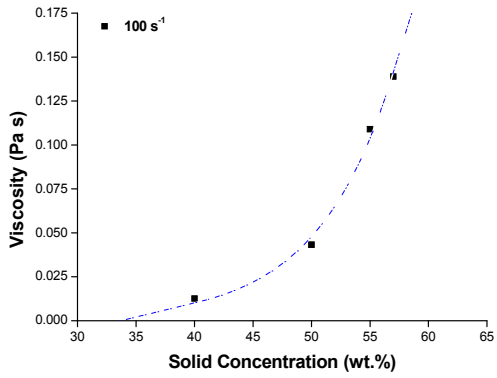

Fig. 1. Viscosity vs solids loading at 100 s^{-1}.

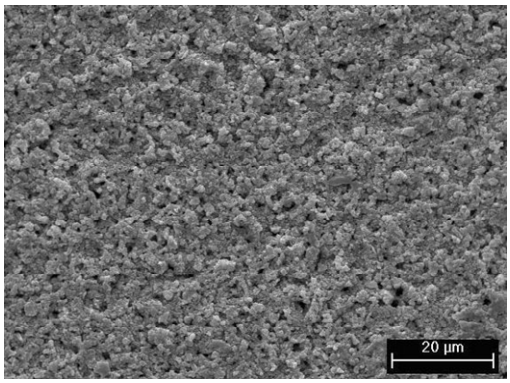

Fig. 2. SEM microstructure of the upper surface of a green tape.

Acknowledgment: *This work has been supported by MICINN (Spain) under contract MAT2006-13480, MCT/Finep/CNPq (process 142855/2005-7 Brazil) and CAPES (process 4710-06-1 Brazil).*

NOVEL STRATEGIES TO PRODUCE HIGH SPECIFIC SURFACE AREA CERAMIC FOAMS FROM PRECERAMIC POLYMERS

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Cellular SiOC ceramic foams containing 3D interconnecting open porosity were fabricated from preceramic polymers by applying two different methods: (i) Synthesized Periodic Mesoporous Organosilica (PMO) particles were embedded into a foamed polysiloxane preceramic polymer and then green samples pyrolyzed at 1000°C in inert atmosphere. (ii) Utilizing the commercially available preceramic polymer, a physical blowing agent and a metal halide, macro-porous ceramics with the cell wall surface decorated with 1D nanostructures (nanowires) were produced by catalyst-assisted-pyrolysis at temperatures higher than 1250°C. For both of these strategies, by varying the processing conditions, it was possible to obtain SiOC ceramics possessing specific surface area values ranging from ~10 to 120 m^2/g. Various characterizations were performed on the resulting components, including the measurements of the specific surface area, open/closed porosity, phase constitutions and the microstructural evolutions by SEM and HRTEM.

ZNO-BASED THIN FILMS BY EPD

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Electrophoretic deposition is a very useful method to obtain thin and thick films with controlled thickness and morphology. It has also been used to design complex structures in the micro and nanoscale, such as laminates or micropatterned arrays. The main parameters that determine the effectiveness of EPD are those related to the suspension's stability (concentration, conductivity and electrophoretic mobility): suspensions must be stable and well dispersed in order to be able to obtain uniform deposits. In case of nanoparticles, because of their strong trend to agglomerate and become micrometer range colonies, it is necessary a complete study of stability to obtain a monodisperse suspension. Once the stability is studied, EPD parameters must also be optimised to control and design the nanoparticle arrangement.

In this work we have studied the parameters that control the deposition of Zn-O based nanoparticles, current density, directionality and intensity of the electric field, time of deposition, etc. to achieve the desired objectives of organization in the assembly and compaction of nanoparticles, as well as the drying and sintering processes of the thin films. EPD was performed under previously optimized suspensions' conditions and over electro-polished substrates. Deposition times and intensities were optimised by analyzing the resulting thin films' characteristics. Finally the deposits were characterised by DRX, AFM, FE-SEM and different spectroscopic techniques.

Acknowledgement:
This work has been supported by MAT2006-01038, MAT2007-66845-C02-01 and CCG08-CSIC/MAT-3811 projects.

Invited Lectures and Session 6

Chair persons: K. Uematsu &. E. Sánchez-Vilches

COMPLEX SHAPE FORMING USING CROSSLINKABLE POLY VINYL ALCOHOL (PVA)

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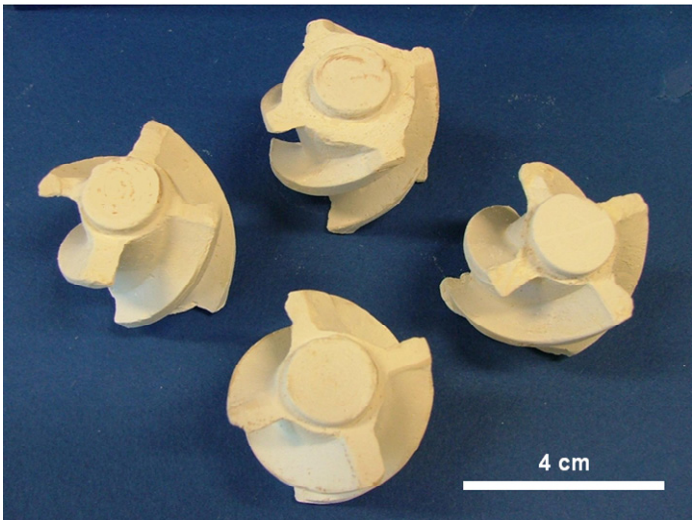
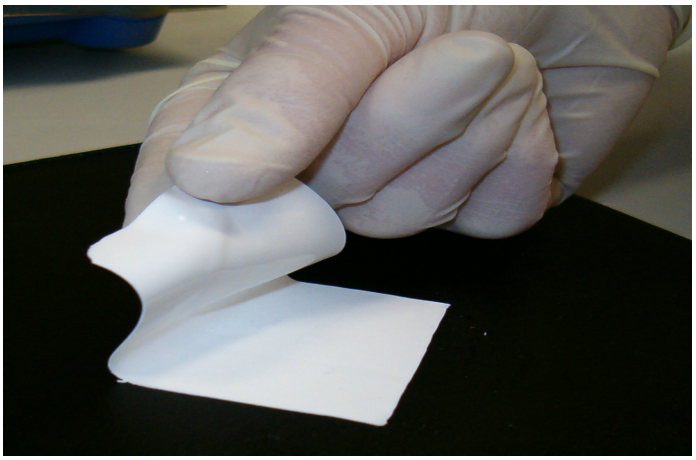
A new aqueous binder chemistry for producing near net shape ceramic components is presented. The formulations consist of dispersions of submicron ceramic particles in aqueous solutions containing polymers such as poly vinyl alcohol and a temperature activated crosslinking agent (DHF, 2,5-dimethoxy-2,5-dihydrofuran). These formulations can be produced to have low viscosity so that they can either be poured or injected into complex shape molds or cast into tapes. After casting, the suspension is heated to about 70 ^{o}C to activate the crosslinking agent. During crosslinking of the polymer, the rheological and mechanical behavior of the suspension is changed from liquid-like to solid-like. This allows the complex shaped bodies to be removed from the mold, dried and sintered. Thick section (at least 3 to 5 cm) alumina, 3D shapes can be produced and sintered to >99% density. Addition of a small amount of plasticiser produces formulations suitable for aqueous tape casting. The strengthening of the cast tape due to crosslinking the polymer allows it to be dried without cracking. Very thick tapes (more than 8mm) can be produced. Rheological and mechanical behaviour, green and fired densities as well as examples of formed components will be presented.



Photograph of molded complex shaped rotors after drying, produced from 42 vol % Al_2O_3 suspensions in 3.8 wt% (per solution) PVA solutions at pH=1.5, gelled at 60°C with 100mM of DHF.



Flexible and strong crosslinked alumina tape.

MICROSTRUCTURAL REQUIREMENTS AND IN SITU PROCESSING FOR ALUMINA MATRIX NANOCOMPOSITES

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Alumina matrix nanocomposites have better mechanical properties than pure alumina. The vast majority of previous work has been on alumina/SiC nanocomposites. The resistance to severe wear of these materials is typically a factor of 3 better than that of alumina. The quality of surface finish for a given grinding treatment shows similarly impressive improvements and the strength is also increased.

These improvements are in commercially important properties but, despite this, alumina matrix nanocomposites have not been applied commercially. The obvious reason is that they are difficult to shape and to sinter. The majority of reports of these materials use hot pressing for densification, which makes shaping difficult. Although the addition of Y_2O_3 helps pressureless sintering, very high temperatures are still required, the amount of SiC that can be added is limited and there is only a narrow processing window that avoids abnormal grain growth. The use of a nanosized oxide second phase instead of SiC leads to coarsening and the consequent destruction of the essential nanostructure of the composites.

This presentation reviews the mechanisms thought to be responsible for the property improvements in alumina matrix nanocomposites. A key component is a dispersion of strong, incoherent nanoparticles within the matrix grains. This is thought to inhibit twin formation and dislocation pileups. The possibilities for producing such a dispersion by precipitation are discussed and a method of producing alumina/$FeAl_2O_4$ nanocomposites by this route is described. The material is sintered to full density as a single phase solid solution, thus avoiding the problems of shaping associate with hot pressing, and the subsequent precipitation of the nanoparticles avoids the problems of coarsening during sintering encountered with mixed powder processing. Results relating to microstructural development and properties are presented that show this to be a promising route for the shaping of alumina matrix nanocomposites with improved properties.

TEMPLATED GRAIN GROWTH AND PROPERTIES OF BIOINSPIRED CERAMIC MICROSTRUCTURE COMPOSITES

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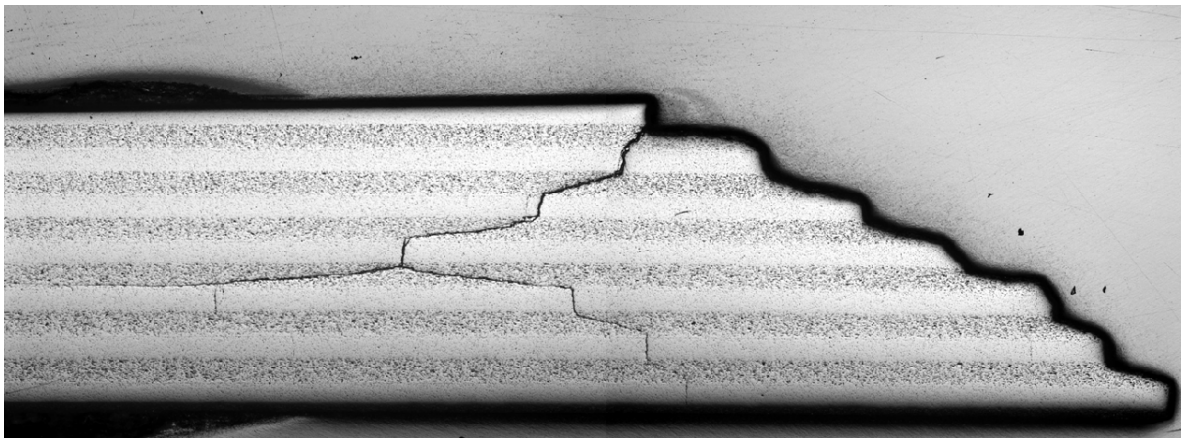
The properties of ceramics are governed by a combination of intrinsic crystallographic and extrinsic microstructural characteristics. Microstructure characteristics, such as grain size/morphology/orientation, the amount and distribution of porosity and grain boundary phases, have pronounced effects on mechanical and non-mechanical properties. The advent of the templated grain growth (TGG) process has enabled access to a variety of unique bulk textured ceramics. TGG allows us to mimic the microstructural characteristics of fracture resistant composites found in nature such as mollusk shells, bone and teeth. Nature uses combinations of inorganic and organic phases to achieve unique structural characteristics like high fracture toughness and large stain to failure. We demonstrate how different microstructural elements of differing connectivities (2:2, 1:3 and 0:3) can be processed *in situ* by TGG in a single phase material to yield flaw tolerant ceramic microstructure composites that are similar in fracture behavior to natural occurring composites. Alumina composites with high strength (~425 MPa) and high load retention (up to 0.5 mm deflection in 4-point bending in a 1.5 mm thick x 2 mm wide x 25 mm alumina composite) were designed in 2:2 composites. This non-catastrophic failure behavior is directly tied to crack deflection and bifurcation along basal surfaces of templated textured layers (see below). We discuss how properties of the textured layers can be modified to enhance the load to failure in alumina microstructure composites and about how this approach can be applied to other systems. Evidence of flaw tolerance will be discussed in terms of indentation-strength measurements and the occurrence of multiple crack initiation and crack arrest.



Crack deflection in a 2:2 alumina microstructure composite

PREPARATION OF HIGH SOLIDS CONTENT NANOTITANIA SUSPENSIONS FOR ATMOSPHERIC PLASMA SPRAYING

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The manufacture of nanostructured coatings by atmospheric plasma spraying (APS) requires the reconstitution of starting nanopowders into a sprayable size by spray drying. This makes it necessary to prepare and optimise the nanopowder suspensions with a high solids volume fraction to obtain homogeneous spray-dried granules.

This work deals with the dispersion of nanosized TiO_2 particles in an aqueous medium. TiO_2 suspensions were reconstituted into plasma sprayable powders. The reconstitution process was performed by spray drying, followed by thermal treatment in order to reduce porosity and enhance agglomerate sinterability for the subsequent APS deposition. Nanosized TiO_2 powders with an average primary size of ~20 nm and surface area of ~50 m^2/g were used (Aeroxide P25, Degussa-Evonik, Germany) as starting material. A colloidal titania suspension of the same supplier was also used (W7 40X).

The dispersing conditions were studied as a function of pH, dispersant type (ammonium citrate, AC, and polyacrylic-based polyelectrolyte, PAA) and content, solids loading and stability with time. Well-dispersed TiO_2 nanosuspensions with solids contents up to 30 vol% (62 wt%) were obtained by dispersing the powder with 4 wt% PAA. Solids contents as high as 35 vol% could be prepared by adding those TiO_2 nanoparticles to the TiO_2 colloidal suspension at optimised dispersing conditions.

TiO_2 powder reconstitution was performed by spray drying both types of nanosuspensions followed by thermal treatment of the resulting spray-dried powders. Spray-drying variables were optimised to obtain free-flowing powders with an adequate granule size distribution, which led to better performance during deposition and enhanced coating properties.

Finally, spray-dried nanostructured TiO_2 granules were deposited on austenitic stainless steel coupons using APS. Coating microstructure and phase composition were characterised using SEM and XRD techniques. The coating microstructure was formed by semi-molten feedstock agglomerates surrounded by fully molten areas that acted as a binder, thereby maintaining coating integrity. Optimum spraying conditions gave rise to limited fusion of the starting powder, retaining most of the initial nanostructure in the final coating.

This study has been supported by the Spanish Ministry of Science and Innovation (project CIT-420000-2008-2) and by the Institute of Small and Medium-sized Enterprise (IMPIVA) of the Autonomous Government of Valencia and the European Social Fund through the European Social Fund (ESF) Operational Programme for the Valencia Region 2007-2013.

MULLITE COATINGS ON CERAMIC SUBSTRATES: STABILISATION OF Al_2O_3-SiO_2 SUSPENSIONS FOR SPRAY DRYING OF COMPOSITE GRANULES SUITABLE FOR REACTIVE PLASMA SPRAY

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The present work deals with the preparation of stable mixed alumina+silica suspensions with high solid loading for the production of spray-dried composite powders. These composite powders are to be used for reactive plasma spraying whereby the formation of mullite and the coating on a ceramic substrate are achieved in a single step process. Electrostatic stabilisation of alumina and silica suspensions has been studied as a function of pH. Silica suspensions are most stable at basic pH whereas alumina suspensions are stable at acidic pH. We show that the addition of ammonium polymethacrylate (APMA) makes it possible to stabilize alumina and prepare a stable 50 wt% alumina+silica suspension at pH 10. The optimum amounts of dispersant and binder have been determined by zeta potential, viscosity and settling measurements. Spray drying of the suspension yields composite powders whose morphology, size distribution and flowability have been characterized before realizing reactive plasma spraying tests.

MEASUREMENT OF BULK DENSITY DISTRIBUTION IN LARGE CERAMIC TILES BY A NON-DESTRUCTIVE METHOD

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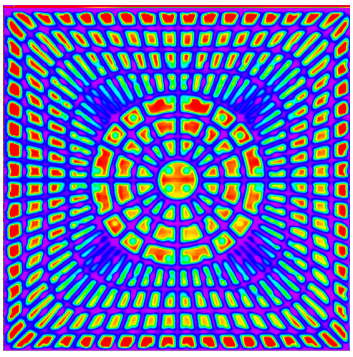
Numerous research studies have demonstrated the pronounced influence of unfired ceramic tile porosity on the physical properties of both the unfired tile (mechanical strength, permeability, etc.) and the fired tile (linear shrinkage, water absorption, etc.). The most common defects that occur in tile manufacturing (black core, low mechanical strength, etc.) stem from, and/or are highly influenced by, unfired tile porosity as a result of variations in porosity during the manufacturing process (differences in size, etc.) or of non-uniform porosity distribution in the tiles (departures from rectangularity, etc.). The foregoing evidences the importance of establishing accurate and permanent control of average unfired tile porosity, as well as of determining porosity distribution in the tile, through tile bulk density measurement.

The most widely used method of measuring ceramic tile bulk density in Spain is by immersion in mercury. The mercury displacement method has enabled large defect-free ceramic tiles to be fabricated, which have contributed to positioning Spain as one of the world's top tile manufacturers. In fact, Spain currently has about 250 tile manufacturing companies and is, after China, the world's second-largest manufacturer of ceramic tiles. The **mercury displacement method** is **very accurate** (ε_a<0.1%). However, the method is harmful, since it involves handling mercury, and is also **destructive and discrete** because the tile needs to be cut into pieces for the bulk density measurement. In recent years, other methods based on the same principle, without mercury, have been developed and used, but these methods are less accurate than the mercury displacement method, while remaining destructive and discrete.

ITC has built and patented an apparatus that measures bulk density distribution by X-ray absorption in large ceramic tiles (up to 120 cm x 120 cm). This technique, based on the Lambert-Beer law, has many advantages compared with other methods: it allows **tile bulk density distribution** to be mapped and is **neither destructive nor toxic**, provided the X-ray tube and detector area are shielded to prevent leakage.

In the present study, this technique, whose technical feasibility and accuracy had been verified in previous studies, has been used to scan ceramic tiles with different compositions (stoneware, earthenware, and porcelain tiles), formed under different industrial conditions, modifying press working parameters. The use of high-precision lasers allows tile thicknesses to be mapped, facilitating the interpretation of manufacturing defects produced in pressing, which cannot be interpreted by just measuring bulk density. The results of these tests are presented in this study.

The bulk density distributions obtained in the same unfired and fired tiles are also compared, a possibility afforded only by this measurement method, since it is non-destructive. The comparison of both unfired and fired tile bulk density distributions allows the influence of the pressing and firing stages on tile end porosity to be individually identified.



Bulk density distribution, obtained by X-ray absorption, of a porcelain tile designed for areas with heavy pedestrian traffic

Acknowledgement:
This work has been supported by the EU through the European Fund for Regional Development (EFRD). and by IMPIVA (Generalitat Valenciana)

DESIGNING PARTICLE SIZING AND PACKING FOR FLOWABILITY AND SINTERED MECHANICAL STRENGTH

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Processing of particulate systems (loose powders, slurries, pastes) is determined by particle packing, hence particle size distribution and particle morphology. These characteristics also greatly affect many properties and the performance of bodies consolidated from powders (dry and sintered powder compacts). However, the particle requirements for consolidated powders are frequently opposed to those for loose powder systems. Refractory concretes provide a unique example of this antagonism: fresh castables require easy flow for improved workability and casting; set and sintered castables require low porosity and high mechanical strength. More than a century's work has been dedicated to finding the best compromise solution, from Academia (thorough explanations and comprehensive models) and industry (competitive practical solutions) alike, from the spherical particle packing models of Furnas and Andreasen to the development of the latest generation ultra-low cement castables. Still, it is difficult to define the requisites for an adequate new formulation and the last resort is simple adjustment of older ones, based on rule of thumb or virtue of experience. In this work, alumina powders in various commercially available size fractions, together with the "Lisa" software (Elkem Materials) and the statistical design of mixture experiments and response surface methodologies (Statistica—StatSoft Inc.), were used to prepare various powder mixtures which were characterized for packing density, size distribution modulus, flowability and after sintering properties, in order to investigate the relationships between these variables. The optimized all-alumina castable was found to require 47.5 wt.% of a fine size matrix with high flowability, which provides the necessary flow bed for 52.5 wt.% of coarse aggregates (Table 1), and presented a fresh flowability index above 106% and a sintered modulus of rupture above 52MPa.

Table 1: Optimum size composition for self-flowability and high mechanical strength.

Alumina matrix [wt%]			**Alumina aggregate (T60) [wt%]**		
CT3000SG	<25µm (T60)	<63µm (T60)	1–3 mm	0.5–1 mm	0.2–0.6 mm
28.5	9.5	9.5	11.375	11.375	29.75

The optimization of matrix, aggregate (Figure 1) and matrix-aggregate proportion, subjected to different property requirements brought to light the relationships between Andreasen size distribution modulus (q), specific surface area (SSA) and maximum paste thickness (MPT). The prevailing mechanisms were investigated for three fundamental processing steps, namely, dry powders, fresh paste and consolidated dry body. A graphical interpretation is provided.

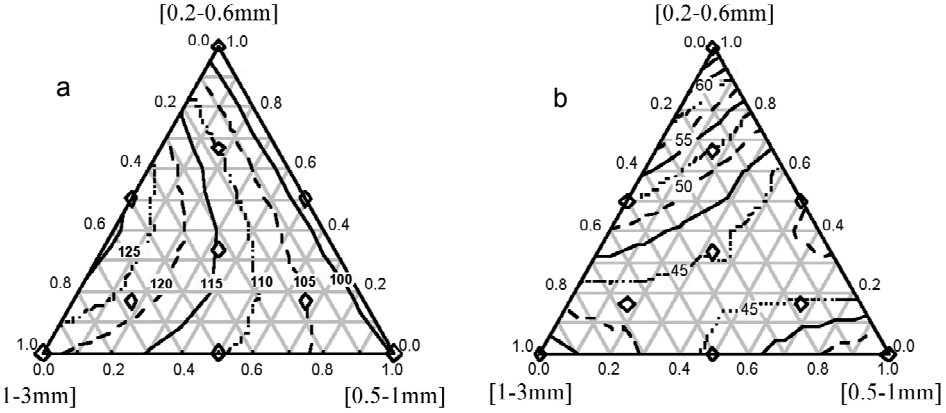

Fig. 1: Aggregate properties contour plots: a) Flowability Index [%]; b) Mechanical strength [MPa].

STUDY OF SHAPING CONDITIONS OF ALUMINOSILICATES BASED SLABS

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To reduce the raw materials and energy consumption in ceramics production, shaping techniques other than uniaxial pressing were evaluated. This work aimed at setting-up processing conditions for shaping of aluminosilicates based slabs by plastic processing of suitably developed mixes.

The mix used to carry out experimental tests was prepared, selecting commercially available raw materials: two clays, one feldspar and a quartz sand. After raw materials characterisation, different preparation conditions were evaluated: mix composition (to affect both plasticity of the mix and firing temperature), particle size distribution of raw materials (d_{50} from some tenth down to few microns), mix preparation conditions (wet or dry milling), water content of the mix to be processed (from 12 up to 18 wt. %), shaping technique (lamination or extrusion). Drying and firing cycles for differently prepared mixes were set-up. After firing, the obtained samples characteristics (microstructure, linear thermal shrinkage, density, water absorption, flatness of slabs, mechanical resistance) were evaluated.

Suitable shaping conditions were set-up for differently prepared mixes and relationships between characteristics of both raw materials and mixes and final products were discussed.

Invited Lecture and Session 7

Chair persons: J. Heinrich &. A.C. Caballero

FABRICATION OF HIGHLY STRUCTURE CONTROLLED CERAMICS THROUGH ADVANCED COLLOIDAL PROCESSING

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The controlled development of texture is one of the ways for effectively improving properties of ceramics. Recently, high magnetic fields with a field strength up to 14 T is readily available without the use of liquid helium due to the development of superconducting technology. These new magnets have been used in studies of many fields, such as crystal alignment, levitation, separation, etc. We have demonstrated the new processing of textured ceramics with a feeble magnetic susceptibility by colloidal processing in a high magnetic field and subsequent heating [1]. The principle of the process is that a crystal with an anisotropic magnetic susceptibility will rotate to an angle minimizing the system energy when placed in a magnetic field. To obtain the oriented materials with feeble magnetic susceptibilities, the following conditions are necessary: (1) the particle should be single crystal and well dispersed, (2) crystal structure should be non-cubic to yield an anisotropic magnetic susceptibility, (3) magnetic energy should be larger than thermal motion energy, (4) the viscosity of the suspension should be low enough to rotate the particles with a low energy, and (5) grain growth is necessary to obtain a highly oriented structure especially when spherical particles are used.

As colloidal processing, slip casting and electrophoretic deposition (EPD) have been conducted successfully. Slip casting is a powerful method to prepare dense and fine grained ceramics that show excellent superplasticity. The EPD is useful for preparing laminated ceramics. The slip casting in a high magnetic field confers several advantages and it is possible for this type of processing to be applied to non-cubic ceramics, such as alpha-alumina, titania, zinc oxide, tin oxide, hydroxyapatite, aluminium nitride, silicon carbide, silicon nitride, etc.[1] Also textured ceramics with complicated structure can be fabricated by reaction sintering, such as beta-alumina, SiC-mullite-alumina nanocomposite, beta-Si3N4, etc. However, when we use whisker or plate-like particles, special attention is necessary owing to the effect of gravity energy that is the highest energy in the colloidal dispersion system.

We have also demonstrated that EPD in a high magnetic field is an excellent method to fabricate crystalline textured ceramic thick bodies. Here, the direction of the electric field relative to the magnetic field can be altered to control the dominant crystal faces. Crystalline-textured controlled laminated composites can be fabricated using EPD by varying the angle between the vectors of electric field and magnetic field [2]. This method can be also applied to prepare crystalline-oriented or specific crystal face thin films for functional applications, such as dielectric ceramics, thermoelectric devises.

Acknowledgments
This study was supported in partly by the Grant-in Aid for Scientific Research of the JSPS and World Premier International Research Center Initiative (WPI) on Materials Nanoarchitronics (MANA), MEXT, Japan.

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PRESSURELESS SINTERING OF Ti_3SiC_2 POWDER

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Ti_3SiC_2 exhibits a unique combination of properties of both, metals and ceramics. Light weight, good stiffness and thermal shock resistance, easy machinability, excellent damage tolerance and good mechanical properties at high temperatures; have made this material a potential candidate for the applications in the automotive, aerospace and the industrial sectors. However, producing fully dense and high purity Ti_3SiC_2 through pressureless sintering has been a daunting task. The pressureless sintering process has many technological advantages for economical production of the parts. The present investigation attempts to produce Ti_3SiC_2 powder and to understand its sintering behaviour during pressureless condition. Efforts have been made to enhance the densification by using small amounts of elemental silicon and nickel powders as additives. Ti_3SiC_2 powder was produced by heating the mixture of Si and $TiC_{0.67}$ powders at 1150°C for 2 hours, under argon atmosphere. Cold-isostatically pressed (at a pressure of 275 MPa) compacts of Ti_3SiC_2 powder (-400 mesh size) were sintered at various temperatures (1200 °C to 1550 °C) for an isothermal time of 2 hours. The little amount of TiC impurity present in the as-synthesized powder was found to be converted into the Ti_3SiC_2 phase completely, during sintering at 1500 °C. The onset temperature of sintering was found to be at about 1060 °C using dilatometer. The solid state reaction of silicon with titanium carbide and interdiffusion of silicon in Si-deficient $Ti_3Si_xC_2$ (where $x<1$) phase were found as sintering mechanisms at lower temperature range; whereas at high temperatures, molten silicon caused reactive liquid phase sintering. The additions of 1 weight% of silicon and nickel powders enhanced the densification process significantly and the sintered densities were increased up nearly 99% (relative densities). The pure Ti_3SiC_2 showed uniformly distributed angular grains, whereas the addition of extra Si and Ni produced microstructures with bimodal grain size distributions. Additives improved the mechanical properties significantly.

SPARK PLASMA SINTERING OF Si-BASED CERAMICS

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The spark plasma sintering (SPS) technique opens new potential for materials development, especially when grain growth or phase transformation aims to be limited. That is the case of liquid phase sintered silicon nitride (Si_3N_4) and silicon carbide (SiC) ceramics, whose thermomechanical and tribological properties are drastically controlled by both the α/β phase ratio and microstructure. Despite the complexity of the sintering mechanisms in Si-based ceramics, they are relatively well-known when conventional techniques such as hot pressing (HP) are used. However, the development by SPS of dense materials with almost negligible grain growth requires re-examining those mechanisms, which is presently a matter of major debate. In this work, we studied the SPS process in Si_3N_4–based materials, both monolithic and composites. The role of the amount of sintering additives (from 2.5 to 7 wt% of Al_2O_3 plus Y_2O_3 mixtures) and the addition of an electrically conductive second phase (up to 9 vol% of carbon nanotubes, CNTs) was investigated. Shrinkage curves, microstructure evolution, α/β ratio and morphological characteristics were evaluated and compared with those obtained using the HP method (Fig. 1). Fully dense materials with hardly α→β phase transformation and little grain growth were attained using SPS, conversely to the HP method. Glassy phase distribution is pointed out as the main difference between the materials sintered by both techniques. On the other hand, CNTs play an important role in the SPS process, affecting the initial steps of the liquid phase sintering mechanism and shifting in around 100 ºC the first shrinkage temperature.

In addition, SPS technique can be managed to develop continuous in-situ functionally graded materials (FGMs) that are of interest for certain applications, such as cutting tools or gas turbine components. SPS allows getting temperature profiles in the specimen through an asymmetric design of the graphite mold. In this way, Si_3N_4 FGMs were obtained from a single additive composition at temperatures in the range of 1550 to 1650 ºC, with continuous gradations of α-phase content, from 4 to 61%, and grain sizes, from 200 and 500 nm (see Fig. 2). Hardness values form 15 to 20 GPa and fracture toughness in the range of 3.7 to 5.7 MPa•$m^{1/2}$ were measured by Vickers indentation as a function of the location within the specimen.

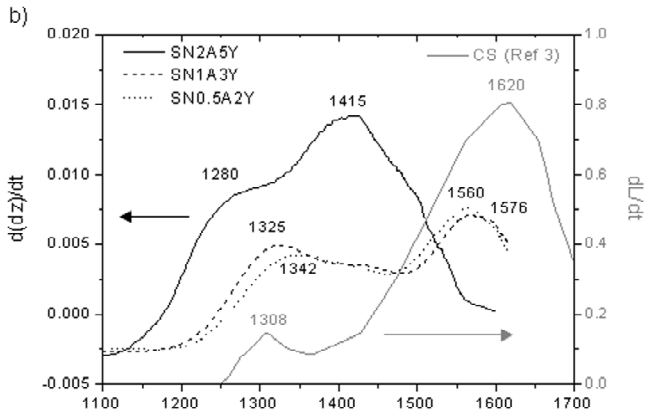

Figure 1. SPS displacement rate (d(dz)/dt) curves as a function of the sintering temperature for different additive compositions. Petzow's shrinkage rate data for conventional sintered Si_3N_4 (CS curve) containing similar Y_2O_3/Al_2O_3 ratio to the SN2A5Y specimen is also plotted.

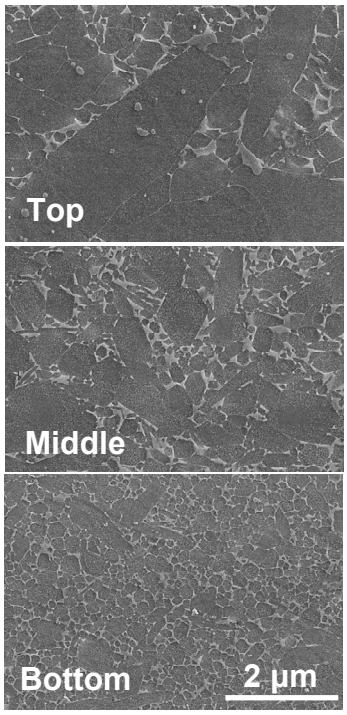

Figure 2. Examples of microstructures corresponding to different zones in the axial direction of the Si_3N_4 FGM.

POROUS MULLITE MATERIALS WITH VERY LOW THERMAL CONDUCTIVITY

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Macroporous ceramics are increasingly being considered for many applications, such as molten-metal filters, high temperature insulation, catalyst supports, exhaust gas particle filter for diesel engines or gas burners. In all these applications, porous ceramics are advantageously used because their characteristics, such as low thermal mass, low thermal conductivity, high permeability and low density. In addition, mullite ceramics combine other interesting properties like high temperature strength, creep and corrosion resistance. Several research papers can be found studying the thermal conductivity of porous mullite materials as function of microstructure, mostly of morphology and size distribution of pores and grains. However, all of them deal with fully crystalline materials, despite the big effect that the presence of amorphous phases can have on the thermal properties. In the present work, porous mullite materials were prepared by spark plasma sintering (SPS) at different temperatures, before and after crystallization, amorphous mullite compact beads of about 30 μm diameter. These beads were obtained by flame spraying spray dried mullite powders in water. The effect of a crystallization pre-treatment of the beads prior to sintering on the material properties was also analyzed.

The thermal diffusivity of the SPS compacts was measured up to 800 ºC and thermal conductivity was calculated from them using the specific heat of mullite and the density of the specimens. Data were comparatively discussed with those of highly porous mullite materials fabricated by the direct consolidation method, based on the swelling properties of starch granules in concentrated aqueous suspensions. Materials fabricated by SPS showed continuous porosity while those obtained by direct consolidation presented large isolated pores, which led to thermal conductivity values for SPS materials of ~1 $W \cdot m^{-1}K^{-1}$, 50 % lower than those prepared by direct consolidation (~ 2.5 $W \cdot m^{-1}K^{-1}$), for the same porosity level.

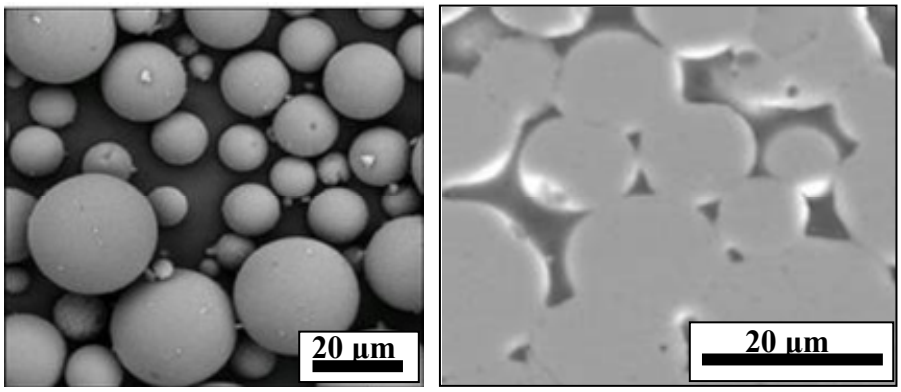

Figure 1. SEM micrographs of mullite beads used as starting powders and of the polished cross section of the SPS mullite material having 18 vol% of porosity.

PRODUCTION OF β-SiAlON CERAMICS WITH LOW AMOUNT OF ADDITIVE AT LOW SINTERING TEMPERATURE

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Silicon nitride (Si_3N_4) based ceramics such as SiAlON's are made of powders and the properties of final product such as corrosion/oxidation, thermal shock resistance and mechanical properties are depend on the quality of the starting powders. There are some studies about the effect of nano-sized SiAlON starting powders on the sintering behavior and quality of the final product. As mentioned in the literature nano-sized powders have positive effect on both sintering temperature and mechanical propeties. There are some routes to prepare nano-sized SiAlON powders such as plasma-chemical and laser synthesis, sol-gel and high-energy mechanical milling in dry medium. The most of these routes are expensive or have other disadvantages such as there could be contaminants in the milled powder because of wearing of vial and/or balls in high-energy mechanical milling route. In the present work wet milling process is used to produce nano-sized powders. Different milling times and mediums (Methyl ethyl keton, ethanol and toluene as solvents, polyethyleneglicol, oleic acid sodium tripolyphosphate and polyvinyl pyrolidon as dispersants) were performed for the determination of the most efficient milling system. The milled powders were characterized by dynamic beam scattering method, BET and XRD measurements and obtained nano-sized β-SiAlON starting powders which were sintered at lower temperatures with less amount of additives than the conventional powders. Results were discussed by using relationship between density, phase composition, microstructure and mechanical properties.

Acknowledgement:
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SINTERING OF POWDERS IN THE $CrSi_2$–Ti (Ta)Si_2 SYSTEMS DEPENDING ON THE METHODS OF SYNTHESIS

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Among various silicides used for heat resistance articles and coatings that operate up to 1200°C, chromium disilicides are most popular. Using silicon in the form of chromium silicides and its solid solutions with titanium, tantalum, rhenium, molybdenum, and boron is more efficient. Application of these materials is, however, limited to difficulties in formation of parts and mechanisms because they are difficult–to–sinter and so fabrication of compacts from them requires high temperatures and pressures. To reduce the temperature of sintering and obtain high-quality articles, it is expedient to use nanosized powders, which activate a sintering process.

The basic trend of the current technology for production of nanostructured materials with improved physicochemical and physicomechanical characteristics is synthesis of materials in a non-equilibrium state based on the so-called processes of activation/suppression. Mechanosynthesis and low temperature synthesis with prior mechanical activation have been chosen in this work for production of powder materials in a non-equilibrium state.

The microstructure of sintered samples shows that under the optimal sintering conditions, the grain size of nanosized powders does not exceed 1 μm while for microsized powders it reaches several tens microns. Herein the samples fabricated from powders produced by low temperature synthesis with prior mechanical activation are better: their structure is composed of practically equal grains and uniformly distributed micropores sizing under 1 μm. The samples from mechanosynthesized powder are characterized by the presence of great quantity of microcracks appeared, perhaps, under pressing and caused by the strained state of the powder.

The complex of carried out investigations of coating deposition using methods for electrolysis and galvanophoresis made it possible to clarify the regularities of the influence of powder dispersity on the parameters of deposition and the structure and content of refractory component in the deposit.

The use of nanosized powders resulted in increasing markedly (practically by twice) the volume content of refractory compound in the Ni matrix under electrolytic deposition and in obtaining denser phoretic deposits than in the case of using microsized powders.

The Ni–B–$CrSi_2$ microsized galvanophoretic coatings after heat treatment has longitudinal cracks on the bending surface, which corresponds to the coupling strength 3÷4 kg/mm^2. The Ni–B–$CrSi_2$ nanosized galvanophoretic coatings after heat treatment has longitudinal cracks on the bending surface, which corresponds to the coupling strength ≈10 kg/mm^2.

Microstructure of the compact samples prepared from mechanically activated powders shows grains of practically closer sizes with uniform distribution of micropores. The samples from mechanosynthesed powder have numerous microcracks, which may appear under pressing as a result of stresses this powder.

High energy mechanical treatment of initial components influences in the mechanism and kinetics of solid phase interaction and the $Cr_{1-x}TixSi_2$ solid solution formation decreasing the temperature of the start and finish of the solid solution formation by 200-300 °C.

The $Cr_{1-x}TixSi_2$ solid solution can be formed directly under mechanical treatment without formation of intermediate lower silicides.

Sintering of nanosized powders in vacuum occurs at temperatures lower by 100-150 °C as compared to that for microsized powders. Preference should be given to powders prepared by low temperature synthesis after mechanical activation. In this case an ultrafine structure remains after sintering. Sintering of mechanosynthesed powders is more difficult than the above due to complexity of pressing them.

The study was support by the Science and Technology Center in Ukraine (grant 3522)

THE FABRICATION OF POROUS CERAMIC ELECTRODES FOR APPLICATIONS IN ELECTROCHEMISTRY

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A series of new porous ceramic electrodes have been manufactured for application in electrochemical cells. Traditionally, electrodes used in these types of applications have been made from metals, such as dimensionally stable anodes (DSA). However, these electrodes present the inconvenience of being overly expensive.
The new electrodes have been prepared from ceramic powders that have a lower cost than the metal oxides used in DSA anodes. Typical electrode formulations were from mixtures such as Sb_2O_3-CoO-MnO and similar powder precursors, with a variable concentration of each component.

A series of electrochemical tests were undertaken with the new electrodes to test for the efficiency as compared with traditional metal electrodes. The results were found to be dependent upon the composition of the ceramic coating and the physical properties of the coating such as the porosity, which is determined by the sintering parameters.

Systematic studies were undertaken on the processing parameters of the electrodes, and electrochemical efficiency for the oxidation of cyanide was correlated with the physical properties of each class of ceramic electrode.

The new class of electrode is shown to be highly efficient in converting the cyanide into environmentally harmless oxidation products.

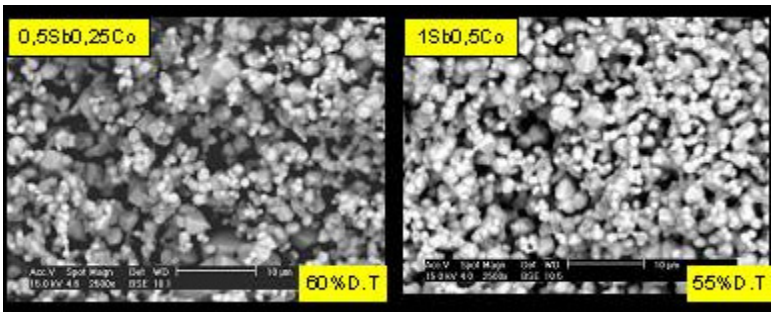

Figure 1. Surface morphology or porous ceramic electrodes for cyanide elimination

Invited Lecture and Session 8

Chair persons: A. Boccaccini &. B. Ferrari

POWDER PROCESSING WITH LASERS AS ENERGY SOURCE

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In ceramics processing lasers are attracting more and more attention because of their ability to provide a high energy density in a well defined volume. Focusing on particular aspects of the laser matter interaction, an overview of laser sources available on the market and suitable for ceramics processing will be given. The advantageous application of lasers as an energy source in the different steps of ceramics processing will be introduced, and results will be discussed. In this context, laser powder processing, different strategies for rapid prototyping of pure ceramics, and potential based self organized shaping are major topics.

PROGRESS IN THE ELECTROPHORETIC DEPOSITION OF CARBON NANOTUBES (CNT) AND CNT/ NANOPARTICLES COMPOSITES

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One very promising technique being developed for manipulating, ordering and shaping carbon nanotube (CNT) structures is electrophoretic deposition (EPD). In this presentation we will show that EPD is a robust and reproducible method to fabricate uniform coatings of CNTs on metallic and ITO-glass substrates. These coatings exhibit controllable thickness, tailorable (nano)porosity and excellent structural homogeneity. The deposition kinetics of CNTs can be controlled by varying EPD parameters such as applied electric field and deposition time. CNT films produced by EPD are suitable for a wide range of applications; including field emission devices, biomedical scaffolds and coatings, catalyst supports, as well as large surface area electrodes for fuel cells, capacitors and gas sensors. Nanoporous CNT films can serve as hosts for functional ceramic nanoparticles (SiO_2, Fe_3O_4,TiO_2) to develop functional nanocomposites. The processing of such nanocomposites can be carried out by sequential EPD or electrophoretic co-deposition techniques. Experimental results show that CNTs can be efficiently mixed with ceramic nanoparticles to form well defined composite network nanostructures. The results have confirmed that EPD is a very powerful tool for the ordered deposition of CNTs and ceramic nanoparticles for a variety of applications. Given the great potential of EPD for manipulation of CNTs and their assembly into ordered deposits, films and coatings, it is likely that novel applications of EPD based CNT structures will emerge. Further developments of the EPD process will allow the reliable fabrication (shaping) of three dimensionally controlled nanostructures and nanocomposites either in the form of dense materials or with a required porosity; graded, aligned, and patterned features may also be incorporated as desired. Remaining challenges of the development of the EPD technique in this area will be discussed.

ELECTROPHORETIC DEPOSITION OF STRUCTURED COATINGS

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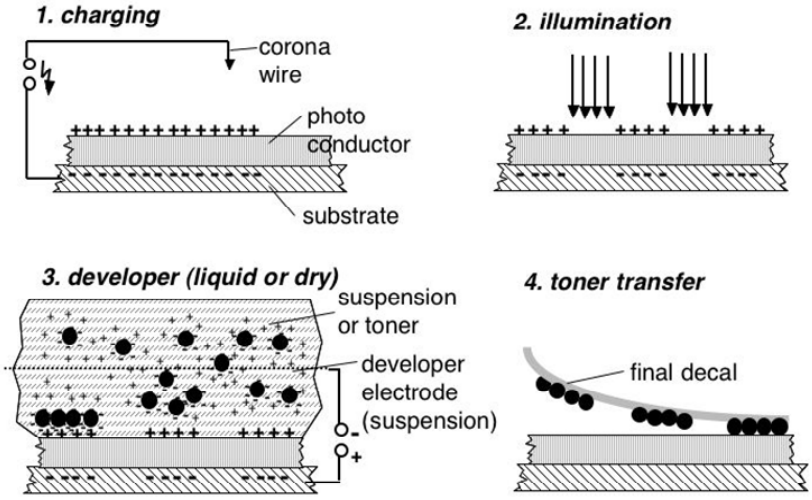
The electrophoretic deposition (EPD) has been proven to be a very attractive process for shaping compacts of nanopowders and bimodal mixtures of coarser particles and nanopowders. One of the reasons for that is that a high green density can be achieved with EPD. In aqueous suspensions high deposition rates are possible, which makes EPD attractive for shaping of large compacts.
In contrast to this most of the industrial applications of EPD are applied for coatings like the ETE-process for enameling of steel sheets or the cathodic lacquering of car bodies. Furthermore, EPD is widely applied in electrophotography (EP), which is the basic process of all laser printers. This digital printing replaced at least for small quantities established processes like screen-printing, but the biggest advantage is obtained for single individually shaped prints. The alternative to laser printing is ink-jet printing. Both methods are also attractive for decoration of glass and ceramics. To improve the long-term stability of inks, which generally contain solved organic colors inorganic pigments were added. These pigments might cause problems with sticking in the fine nozzle of the printing head. Therefore laser printing using either dry toner powders or liquid developers seems to be more favorable. But problems will arise with the more complicated transfer technique ("digital decals"). The principle of laser printing in shown in the following figure:



In the last years a lot of effort was done in developing toners for decoration of ceramics via a transfer technique. Now systems are available which work with commercial laser printers. Alternatively to the dry toners a suspension with charged pigment can be used. The effort for processing is higher, but the resolution is better due to the effect, that smaller particles can be used. Furthermore, the surface charge and, consequently, the electrophoretic motion can be better controlled. One disadvantage of these laser printers with a rotating photoconductor drum is the limited charge that can be stored on the photoconductor. This leads to a limitation of the amount of powders, which is deposited on the electric charge pattern.

Therefore new concepts were developed for the deposition of structured coatings based on the EPD. For non-conductive ceramic or glass substrates a transfer process is needed. First results of the deposition of structured coatings with increased thickness are presented which have a potential for application in ceramic or glass decoration via a transfer technique.

ELECTROPHORETIC DEPOSITION OF SIALON PHOSPHOR PARTICLES FOR PACKAGING OF FLAT PSEUDO-WHITE LIGHT EMITTING DEVICES

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Recently, the luminescent materials based on rare-earth-doped nitrides/oxynitrides have attracted attention because of their nontoxicity, thermal stability and interesting luminescence properties. Among the nitride/oxynitride luminescent materials, yellow light emission phosphor of Eu^{2+} doped Ca-α-SiAlON has advantages for the application of pseudo-white light-emitting diodes (LEDs). We have fabricated the warm-white LED lamps using the Eu^{2+} doped Ca-α-SiAlON and blue LED.[1,2)] In the near future, the SiAlON phosphor may be applied to general illuminations and liquid crystal displays.

The SiAlON phosphor is generally synthesized by solid phase reaction in the form of powders; therefore, it is essential to establish the packaging technologies for the diversity of applications. Electrophoretic deposition (EPD) is a colloidal process wherein ceramic bodies with high homogeneity and green density are directly shaped from a stable colloidal suspension by a dc electric field. The EPD process also has the advantage of the controllability of deposit thickness by altering the applied voltage and the deposition time. Therefore, EPD is a possible technique for the packaging of phosphor powder. In this study, the preparation of flat, uniform films of the Eu^{2+} doped Ca-α-SiAlON phosphor powder on ITO glass substrates was performed by EPD. The PL properties of the SiAlON phosphor films were characterized by using blue light of the wavelength of 450 nm as a pump source. The blue-light irradiation was conducted through the ITO glass substrates (Fig. 1). The light emission from the deposit surfaces was characterized by comparing the intensities of the excited yellow light and the transmitted blue light through the deposit using a multi channel photo detector.

Homogeneous Eu^{2+} doped Ca-α-SiAlON phosphors films were prepared on ITO glasses by EPD (Fig. 2).The color coordination of the deposit films was controllable from blue-white to yellow-white by optimizing the relative intensities of the transmitted blue light and the excited yellow light. Adhesions of prepared films were high and they were not peeled off from the ITO glass substrates even by centrifugalization at 5000 rpm for 5 min. The color on the chromaticity diagram calculated from the PL spectra agreed well with the actual color perceived by human eyes.

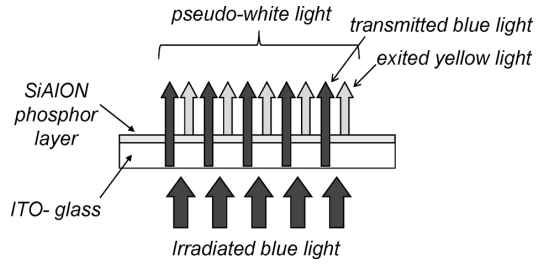

Fig.1 Schematic illustration of a flat pseudo-white light emitting device.

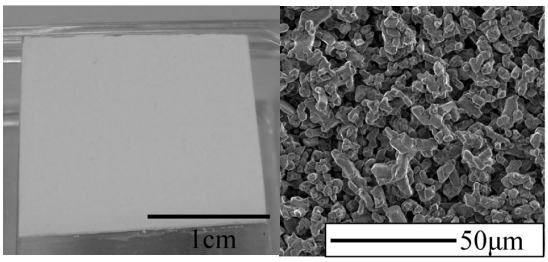

Fig. 2. Microstructure of the SiAlON phosphor film prepared on an ITO glass.

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ELECTRIC FIELD ASSISTED FORMING OF CNT-SiC$_f$/SiC COMPOSITE

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SiC-based ceramic-matrix composites have several advantages in comparison with other candidates for the structural parts in future fusion reactor, however, there are a few serious problems mainly connected to their fabrication. Although chemical vapour infiltration (CVI) and polymer infiltration and pyrolysis (PIP) result in pure low-activation silicon carbide, the techniques are unable to produce high density, which is required for assuring gas impermeability of the first wall as well as to achieve sufficient thermal conductivity of the material. For this reason ceramic routes, where the SiC fabric is infiltrated with a SiC powder slurry, are becoming more relevant. Next to the pressure-or vacuum assisted powder infiltration that did not yield in efficient filling the voids in 3D-fabric, electrophoretic deposition offers a versatile technique for infiltration ceramic fabric. Moreove, it also offers a possibility for fabrication of interphase layer on SiC fibres.

The paper will present the techniques for coating the SiC fibres with a thin layer of multi walled carbon nanotubes and for the infiltration of the fibre fabric with SiC powder in aqueous suspension. To achieve the best possible particle packing in the matrix and hence to minimize the shrinkage during drying, a comprehensive analysis of the effect of the suspension's composition and the processing procedure on the particles' packing density was performed using different types of surfactants. In a subsequent step the green parts were infiltrated with a sintering-aid precursor and sintered, which made it possible for us to obtain a reliable estimation of the effectiveness of the electrophoretic infiltration process.

ELECTROSTATIC AND KINETIC ASPECTS OF ELECTROPHORETIC DEPOSITION OF CERAMIC MATERIALS

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Electrophoretic deposition (EPD) is increasingly being used as a versatile technique for producing a wide array of ceramic products. Compared to other techniques, EPD has a number of advantages, from the possibility of obtaining thick, complex-shaped ceramic items to the production of very thin ceramic films on several types of substrates. Many of the artefacts produced by EDP, such as thin multi-layered ceramic structures having tight dimensional tolerances, could hardly be produced by using more conventional techniques like tape-casting..

EPD has been demonstrated to be feasible both from stabilized colloidal suspensions in aqueous medium or from dispersions of surface-charged particles in organic solvents. As the implementation of the technique is also quite simple, requiring only basic mechanical and electronic gear, and since suitable operative conditions permit the deposition of virtually every ceramic material, the growing interest of the scientific and technical community in this technique appears to be justified.

Currently, some debate is there on the mechanisms that lead to the accretion of solid particles to form a solid deposit from colloidal ceramic suspensions during EDP. A number of different deposition mechanisms have been devised and suggested in the literature, and it's not clear whether or not a single mechanism can be invoked for explaining the EDP-formation of the solid deposit, since colloidal systems for EDP can be quite different as to their composition and means of stabilization and particle charging. Furthermore, as ancillary electrochemical processes are observed during EDP, their role in the process must also be elucidated

In this presentation, we report our observations on some electrical and kinetic aspects of EDP from different colloidal suspension for the deposition of Titania, Lead-Zirconate Titanate (PZT) and Niobium-doped Lead-Zirconate Titanate (PZTN) particles. Current transients observed during EDP are investigated with regard to their meaning and role in the overall deposition process. An interpretation is proposed of the current transients observed during EPD from stabilized colloidal suspensions, in terms of resistive models of the EDP cell that take into account gradients of dielectric constants and ionic mobility in the suspension and deposit.

Financial support by CNR under the Free Theme Research "Study of rheological and colloidal properties of disperse ceramic systems for the optimization of products and processes" Grant n. 405, is gratefully acknowledged.

LEAD MAGNESIUM NIOBATE-LEAD TITANATE THICK FILMS PREPARED BY ELECTROPHORETIC DEPOSITION

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Lead-based perovskite ceramics have generated significant interest because of their outstanding piezoelectric properties, which are utilized in various electronic devices, such as multilayer capacitors, sensors, actuators and electro-optical devices.

We have investigated the processing of $0.65Pb(Mg_{1/3}Nb_{2/3})O_3–0.35PbTiO_3$ (PMN-PT) thick films using an electrophoretic deposition process (EPD), with the PMN-PT particles suspended in an ethanol-based suspension. Homogeneous, nano-sized PMN-PT powder prepared by mechanochemical activation has been used for our experiments. An excess of PbO with the melting point of 880 ^oC has been used to induce sintering in the presence of the liquid phase. The homogeneously distributed PbO particles in green deposit together with optimized sintering conditions (atmosphere, temperature, time) enable sintering in the presence of the liquid phase that is reflecting in a uniform, crack-free microstructure of the sintered thick-film with good functional response.

In our approach we have introduced 2 mol % of excess PbO into the system by mixing together two suspensions, namely PMN-PT and PbO in a corresponding stoichiometry. We have studied the properties of PbO and PMN-PT suspensions. The particles were stabilized in an ethanol using electrosteric stabilization. The zeta-potential and the viscosity of suspensions were measured as a function of amount of additives to identify the conditions for the preparation of suspensions suitable for the EPD. The properties of PMN-PT and PbO suspensions have been tailored in a way that both, PMN-PT and PbO particles have been deposited on the substrate forming uniform deposit with high green density.

EPD has been performed at constant voltage conditions. The applied voltage, the deposition time and the concentration of the powder in the PMN-PT suspension and in the PMN-PT suspension with excess PbO were investigated in order to obtain a high-quality deposit. The PMN-PT thick films prepared from PMN-PT and from PMN-PT with PbO-excess suspensions were sintered at 950 and 1100^oC. The sintered structures were examined by x-ray powder-diffraction analysis and scanning electron microscopy.

Our results show that the nano-sized powder and the densification in the presence of the liquid phase are required for processing PMN-PT thick films at temperatures compatible with the thick-film technology. By tailoring the atmosphere during the sintering, the PbO-free PMN-PT thick film has been obtained. The highest functional response was obtained for a homogeneous, dense, crack- and PbO-free PMN-PT thick film prepared from a PMN-PT suspension with excess PbO. The film was deposited at a constant voltage of 10 V and for a time of 120 s, followed by sintering at 1100 ^oC in a PbO-rich atmosphere. The film's properties were as follows: a dielectric permittivity ε of 20250 at a T_m of 172^oC, a remanent polarization of 17 $\mu C/cm^2$ and a coercive field of 5 kV/cm.

Acknowledgement:
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ELECTRICALLY DRIVEN ARRANGEMENT OF SILICA MESO-POROUS COATINGS

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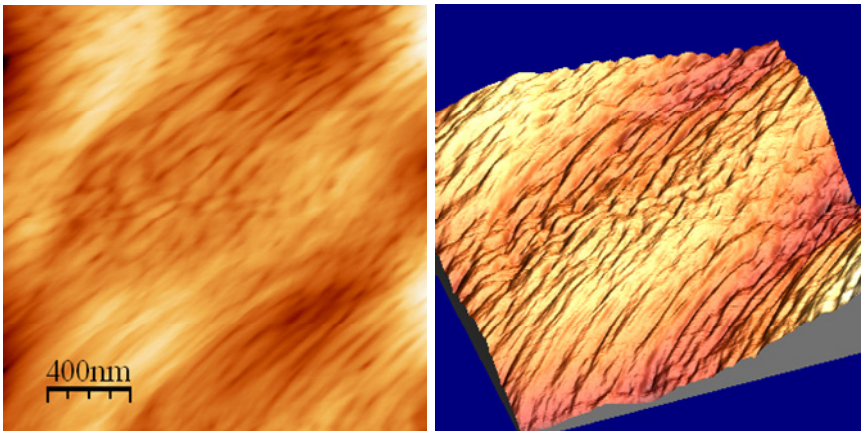
In the last years, preparation of mesoporous coatings has gained attention in different fields, since the presence of porous or high specific surface areas are needed in applications such as sensors, solar cells or photocatalysis. In order to produce homogeneous mesoporous coatings, different processes have been considered, the sol -gel method being one of the most important alternatives, due to great advantages, such as low sintering temperature and homogeneity at molecular level. The preparation of mesoporous thin films has become an dynamic field of study since the discovery of the first meso-organised silicates by the Mobil`s group. An important aspect of this process is the preparation of mesoporous materials that show a periodic distribution of pores of uniform size, combining the sol-gel method with the use of surfactants as structural agents at the EISA method (Evaporation Induced by Self- Assembly). However, this process is limited to the attained thickness of a crack-free and meso-structured film during solvent evaporation. In this work, electrophoretic deposition (EPD) has been considered as an alternative to increase the critical thickness.

This study describes the contribution of the CTAB and Silica in the electrically driven growth of mesoporous films. Different CTAB solutions in ethanol have been prepared to determine its depositing behavior. Silica sols with the optimized CTAB concentration, fixing the ratio H_2O/Silica to 5, were then used to study the arrangement of the generated structures within the sol in the coating process. Coatings were thermally treated and washed to determine by ellipsometry and AFM (figure) the effect of the key sol and deposition parameters in the shaped structure.



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Posters Session 2

MINERALOGICAL AND IONIC CONDUCTIVITY STUDY OF BENTONITE

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The clay mineral diversity and the complexity of their structure make them useful in many industrial estates [1]. The present study aims to evaluate the capacity of a clay sample coming from Keff Eddour from El Metlaoui in south-Western Tunisia.

The characterization of the clay is carried out using the X-ray diffraction (XRD), the infrared spectroscopy (IR), the thermal analysis (DTA and TGA), the chemical analysis, the determination of the cation exchange capacity (C.E.C.) and total surface area (S_T). The ionic conductivity of the clay sample is determined as function of the temperature (316 to 903K) and the nature of the exchanged cations (Na^+, $K^+$and Li^+), following the impedance spectroscopy method [2].

The dominant phase in the clay fraction is smectite 73% it also contains illite 23% and kaolinite. The cation exchange capacity (C.E.C.) and the total specific surface area (S_T) of purified clay are respectively equal 62 meq/100 g and 564 m^2/g of calcined clay. The electrical conductivity, σ, of the clay sample $KENa^+$ is derived from bulk resistance values. It increases from $\sigma = 7.50 \times 10^{-8}\ \Omega^{-1}.cm^{-1}$ at 629 K to $\sigma = 68.92 \times 10^{-7}\ \Omega^{-1}.cm^{-1}$ at 890 K.

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SELECTIVE LASER HEAT TREATMENTS FOR REALIZING COATINGS AND THIN ELECTRIC COMPONENTS – PART 1: SILVER CONDUCTOR FABRICATION ON GLASS AND ALUMINA SUBSTRATES

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The main technique used in microelectronics to realize passive components of hybrid circuits, combines screen-printing and furnace firing. The main disadvantage of this approach is to require numerous successive heat treatments to fire all the components of the circuits in suitable conditions. Using laser for local heat treatments offers numerous advantages: the treatments can be optimized for each material, the energy consumption is minimized and the whole processing time can be significantly reduced.

The topic of our research is the understanding of microstructural and dimensional modifications generated by selective laser treatments (melting, sintering, and crystallization) on thick coatings of material usually encountered in passive components of hybrid circuits. Our final goal is to develop an innovating process to realize bi-dimensional or three-dimensional components by coupling ink-jet printing and selective laser treatments in substitution respectively to screen-printing and to heat treatment furnace. Problems to be solved are: i)- the control of the consolidation, densification, microstructure and crystallization of the coatings and ii)- the bond between the coated material and the substrate or between the different layers in the case of multilayer coatings.

The present paper reports preliminary results concerning an original technique for manufacturing electrical conductive circuits through powder spraying and laser densification. It is linked to two other articles: i). Part II – Sintering of $BaTiO_3$ by Selective Laser Sintering ii). Part III – Melting and crystallization of a piezoelectric glass ceramic.

High quality silver conductors have been prepared on sodalime glass and alumina substrates. Our processing route involves the mixing of a micron size silver powder and a low melting point glass frit in an aqueous solvent. Almost stable suspensions have been obtained with addition of suitable amounts of wetting agent and other organic agents. The substrates have been coated by the suspensions through spraying with a pistol and dried at room temperature. Suitable lasing conditions have been identified through a parametric laser study (CW Nd:YAG, 10 W). We succeeded in obtaining full electrical functionality after laser densification in air without significant oxidation. The whole process is fast, inexpensive and might be a good alternative to screen-printing and low-speed ink-jet printing.

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SELECTIVE LASER HEAT TREATMENTS FOR REALIZING COATINGS AND THIN ELECTRIC COMPONENTS – PART 2: SINTERING OF $BATIO_3$ BY SLS (SELECTIVE LASER SINTERING)

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The topic of our research is the understanding of microstructural and dimensional modifications generated by selective laser treatments (melting, sintering, and crystallization) on thick coatings of material usually encountered in passive components of hybrid circuits. Our final goal is to develop an innovating process to realize bi-dimensional or three-dimensional components by coupling ink-jet printing and selective laser treatments in substitution respectively to serigraphy and heat treatment furnace. Problems to be solved are: i)- the control of the consolidation, densification, microstructure and crystallization of the coatings and ii)- the bond between the coated material and the substrate or between the different layers in the case of multilayer coatings.
This article is about the Selective Laser Sintering (SLS) of $BaTiO_3$ coatings and is linked to two other articles: i). Part I – Silver conductor fabrication on glass and alumina substrates and ii). Part III – Melting and crystallization of a piezoelectric glass ceramic.

$BaTiO_3$ is the most common material used to realize capacitor because of its high dielectric constant. Capacitors made of this material offer excellent frequency characteristics, high breakdown voltage, high reliability and excellent volumetric efficiency [1]. In the present work, thick coatings were realized by spray deposition on inorganic substrates (glass and alumina). The coatings were then treated by SLS using a YAG-laser with a maximal powder of 10W and a spot size of 40 µm.
Scanning parameters (Power %, scan speed and vectorization step) were investigated. The first results show that suitable scanning conditions make possible to obtain uniform and homogeneous $BaTiO_3$ coating exhibiting good bonding with both substrates. The control of the densification of $BaTiO_3$ spray deposited on an alumina substrate coated with a platinum layer is under investigation. A capacitor could be then realized in order to characterize the dielectric properties.

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Acknowledgement:
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SELECTIVE LASER HEAT TREATMENTS FOR REALIZING COATINGS AND THIN ELECTRIC COMPONENTS – PART 3: MELTING AND CRYSTALLIZATION OF A PIEZOELECTRIC GLASS CERAMIC

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The topic of our research is the understanding of microstructural and dimensional modifications generated by selective laser treatments (melting, sintering, and crystallization) on thick coatings of material usually encountered in passive components of hybrid circuits. Our final goal is to develop an innovating process to realize bi-dimensional or three-dimensional components by coupling ink-jet printing and selective laser treatments in substitution respectively to screen-printing and to heat treatment furnace. Problems to be solved are: i)- the control of the consolidation, densification, microstructure and crystallization of the coatings and ii)- the bond between the coated material and the substrate or between the different layers in the case of multilayer coatings.

This article is about the melting and the crystallization of a piezoelectric glass ceramic and is linked to two others talking about: i). Part I - Silver conductor fabrication on glass and alumina substrates and ii). Part II - The sintering of $BaTiO_3$ by SLS.

The piezoelectric glass ceramic investigated contains fresnoite crystals in a glass matrix and is obtained by crystallization of a parent glass of the $SrO\text{-}SiO_2\text{-}B_2O_3\text{-}K_2O\text{-}TiO_2$ system [1]. Fresnoite is a pyroelectric non-ferroelectric material that requires a suitable crystallographic texture to exhibit macroscopically piezoelectric properties [2]. In the present work, thick coatings were realized by serigraphy of a parent glass ink on alumina substrates. The coating were then heat treated by a YAG-laser with a maximal powder of 10W and a spot size of 40 µm. Scanning parameters (Power %, scan speed and vectorization step) were then investigated. The objective was in a first stage to melt a uniform and homogeneous glass surface and then, by mean of a second treatment, to crystallize the glass.

The next steps will be to control the crystallization of fresnoite in order to obtain the suitable crystallographic preferential orientation and then to characterize the piezoelectric property of the coatings.

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STABILIZATION OF AQUEOUS $BaCO_3$ SLURRIES AND COATING ON DENSE Y-TPZ SUBSTRATES FOR MAKING $BaZrO_3$ LAYERS BY THERMAL TREATMENT

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In this communication, the colloidal behaviour of concentrated aqueous suspensions of barium carbonate is investigated. Optimum dispersing conditions are investigated for aqueous suspensions. Stabilisation of the suspensions is achieved by using PMAA dispersant agent. Rheological studies have been made in order to determine the optimum of dispersant concentration and maximum solid loading. The best stabilisation of 30 vol. % suspensions is obtained by using 0.5 wt % PMAA. Zeta potential study shows that the use of dispersant increases the negative surface potential on the particles. The zeta potential values increasing from + 5 mV in a pH 2.5 to - 27 mV in a pH range between 7 and 11 for 0.5 wt % of polyelectrolyte with 0.5 wt. % PMAA. All suspensions show a Newtonian behaviour and maximum packing fraction (ϕ_m) determined by using Krieger Dougherty model is about 0.61.

$BaZrO_3$ phase is obtained by the solid state reaction between barium oxide, which comes from the thermal decomposition of barium carbonate, and zirconium oxide. After a thermal treatment at 1400°C for two hours, pure $BaZrO_3$ phase is formed on the dense stabilized zirconia substrate. During the reaction, the determining rate step follows a typical 3D-surface reaction. The thickness of $BaZrO_3$ layer increases when firing temperature is increased. However, up to 1500°C, the thickness of the $BaZrO_3$ layer becomes less depend on the firing temperature. This behaviour could be explained by the modification of the determining step of rate. The maximum thickness obtained is about 18 µm for a firing time of two hours at 1600°C.

UTILIZATION OF NIGERIAN SANDS FOR GLASS CERAMIC COATINGS

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In this study, Nigerian sand and feldspar were explored, sand inspection and analysis were carried out. Physical, Chemical and Mineralogical characteristics were determined through various analytical tools; Classical Chemical analysis, ICP (Inductively Coupled Plasma), DTA (Differential Thermal Analysis), XRD (X ray Diffractometry) and Infra Red.

Using mixtures of the sands samples, and other chemical substances as additives, several multi component ground - glass compositions with different properties were obtained by conventional melting procedures.

Glass frit properties were examined with the above mentioned analytical tools. Density of the frits was determined; granulometric parameters and High Temperature Microscopy were also evaluated.

The glass coatings were processed with some mill additions for dry and wet application to obtain special effects on application on enameling steel samples. The coated steel samples were heat treated in the electric furnace at various temperature ranges 790^{o}C – 900^{o}C.

The topography of the enameled specimens was studied with AFM (atomic force electron microscopy) at different magnifications to identify surface flaws. Adherence bend test at 90^{o} and thermal shock resistance were investigated.

Fresh transverse cuts of the coated samples were viewed with electron microscope (Scanning electron microscopy) to explore micro-structural changes / transformations that occurred during the coating process.

To understand the composition and microstructure aspects of the coating /substrate interface layer, EDAX analysis of the enameled surfaces were investigated.

PREPARATION OF $YBa_2Cu_3O_{7-x}$ SUPERCONDUCTING THICK FILMS ON METALLIC SUBSTRATES BY THE ELECTROPHORETIC DEPOSITION (EPD) TECHNIQUE

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The complex oxide $YBa_2Cu_3O_{7-x}$ (YBCO) is of major interest for applications because it is superconducting at the temperature of liquid nitrogen (77K). Most potential applications require a critical current density J_c as high as possible, e.g. for resistanceless cables above 77 K. However there are some low-J_c applications, in particular magnetic shielding at very low frequencies. For this application, substrates with large area and complex shape have to be coated with the superconducting ceramic. Electrophoretic deposition (EPD) is one of the few techniques able to meet these practical conditions. When a voltage is applied, migration of particles in the suspension occurs and finally the film grows on one electrode. After the deposition, a thermal treatment is required to densify the coating.

In the present work, YBCO powder is deposited as thick films on silver substrates by the EPD technique. The objective is to maximize the density of the as-deposited coating in order to reduce the duration and/or temperature of the heat treatment required to obtain a dense coating with appropriate superconducting properties (esp. current density and critical temperature).

YBCO suspensions are prepared from commercial powders with average grain size ~ 0.5µm in an organic solvent. Suspensions with very low solid content (typically 1g YBCO in 100 ml acetone) can be prepared without surfactant, using I_2 as an additive. However the density of the as-deposited coating is low and sintering is rather poor. Therefore suspensions with higher concentrations (up to 30 wt%) are prepared using suitable dispersants, resulting in much better densification behaviour. The microstructure of the thick films is investigated as a function of the deposition parameters : powder and additive concentrations in suspension, applied voltage, number and thickness of the layers.

MULTILAYER COATINGS OBTAINED BY COMBINATION OF PVD, CVD AND DIP-COATING TECHNIQUES

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The use of ceramic materials as thin films is a topic of current interest both, from the scientific and technological point of view. The study and development of coatings and thin films is aimed at improving the behavior of materials in different applications where high performance is requested under aggressive conditions or high working temperatures. The reduction of productions costs as well as a continuous improvement of the industrial processes is also required [1]. There are a number of procedures for the preparation of ceramic coatings. Due to their excellent implantation at industrial level different methods and techniques such as Physical Vapor Deposition (PVD), Chemical Vapor Deposition (CVD) plasma-spray and sol-gel are to be cited [2, 3]. The suitability of each of the referred methods depends on the nature and morphology of the substrate to be covered as well as on the characteristics and properties of the requested coating (figure 1).

Figure 1.- PVD and plasma spray coating processes.

Despite the fact that the basic knowledge on the different preparation methods of coatings is relatively broad nowadays, the study of different scientific and technological aspects of these processes should be widened and improved. In this connection, the present work is aimed at the development of multilayer TiN-, TiAlN- and ZrO_2-based multilayer coatings by combination of PVD, CVD and dip-coating techniques. Different coatings have been deposited onto AISI-D2 tool steel and AISI-310 stainless steel substrates with the purpose of characterizing such coatings and studying their behavior. The final goal of this work was to improve the working capacity of the substrates as well as to extend their useful life. With such an aim, the surface and morphology of the coatings have been studied by Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM). The compositions of the coatings as well as the interfaces existing between the layers were analyzed by Glow Discharge Optical Emission Spectrometry (GDOES). Finally, nanoindentation was used to test the mechanical behavior of the coatings.

The experimental results indicate a good compatibility between some of the coating techniques here used. In fact, they may complement each others by properly alternating layers of different nature as well as by varying the thickness and densification of such layers, thus obtaining a noticeable improvement of some properties of the multilayer coating here studied.

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ZIRCONIA COATINGS PREPARED BY EPD FROM SOL-GEL SOLUTIONS

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Sol-gel processes constitute one of the most interesting choices for the preparation of ceramic thin films. Such processes exhibit important advantages for the development of novel materials, the preparation of materials with tailored porosity or the deposition of coatings on diverse substrates of different nature. An additional advantage of the sol-gel method is its relatively low cost [1, 2].

A number of techniques have been developed in connection with the sol-gel method aimed at coatings deposition. The suitability of each of such techniques depends on the morphology and dimensions of the substrate as well as on the particular properties of the required coating. Electrophoretic deposition (EPD) constitutes a versatile and low-cost technique that is suitable to be applied on a wide variety of substrates allowing a fine control on the parameters that influence the deposition process [3, 4].

In the present study the EPD technique has been applied for depositing ZrO_2-3mol%Y_2O_3 coatings from sol-gel solutions. The aim of the research work was to combine the adequate control on the process parameters provided when applying the EPD method with the versatility and fine adjust offered by the sol-gel method with respect to the composition of the solutions and materials obtained. With such a purpose, AISI-310 stainless steel substrates have been used for the deposition of coatings from sol-gel solutions prepared with different pH and oxide concentration. Solutions were characterized in terms of density, pH, particle size in solution and rheological behaviour. Coatings were deposited by using a power supply that makes it possible to set the process control parameters (voltage or current intensity). The depositing process conditions were optimized by determining the thickness and densification of the layers. Such layer were subsequently heat-treated at 300, 500 and 700ºC and characterized by Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM) and nanoindentation tests.

The results obtained reveal a noticeable influence of oxide concentration and solution pH on the quality of the deposited layers. Furthermore, the densification and mechanical properties of the coatings here obtained may be tailored through carefully controlling the different parameters of the process, the treatment time and the temperature of the sintering step.

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MANUFACTURING OF (THICK) Ni-YSZ ANODE AND (THIN) YSZ ELECTROLYTE FOR TUBULAR SOFC BY POWDER THERMOPLASTIC EXTRUSION MOULDING

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In this communication, a powder extrusion moulding (PEM) process, based on thermoplastic binder system was described for the manufacturing of microtubes of YSZ and Ni-YSZ. In both cases the binders are constituted by a multicomponent thermoplastic binder based on polypropylene and paraffin wax. Different feedstocks were prepared from a commercial YSZ and NiO-YSZ powder mixtures plus the binder. The rheological study of the ceramic mixtures prepared, allow us to select those with a suitable viscosity value and flow behaviour. The influence of powder surface coating by stearic acid was studied. The feedstocks are mixed at a suitable temperature in a twin extruder which rapidly homogenizes the powder-polymer mixture.

The extrusion of the tubes was performed with an in-house die-tool designed to obtain different wall thicknesses. Thin-walled tubes with a diameter near to 5 mm and a length of 10 cm were obtained with good dimensional stability and without defects. The wall thicknesses of the green extruded tubes were 500 and 1000 μm. The binders are removed by thermal debinding. The thermal debinding mechanism has been investigated by thermogravimetry (TG) and differential scanning calorimetry (DSC). Finally the polymeric parts were removed by a combined solvent and thermal debinding process. The sintering of both kinds of materials was performed in air. In the case of NiO-YSZ, a subsequent reduction process was carried out in order to obtain the Ni-YSZ anode. XRD experiments confirmed the full reduction process. A systematic microstructural characterization was carried out. Finally, for some of the samples electrical characterization was performed, and the results were compared with those of the samples obtained by uniaxial compaction. Preliminary results conclude that this technology is suitable for the manufacturing of thin (<250 μm) YSZ and thick (500μm) Ni-YSZ microtubes to be used in self-supported and anode-supported solid oxide fuel cells.

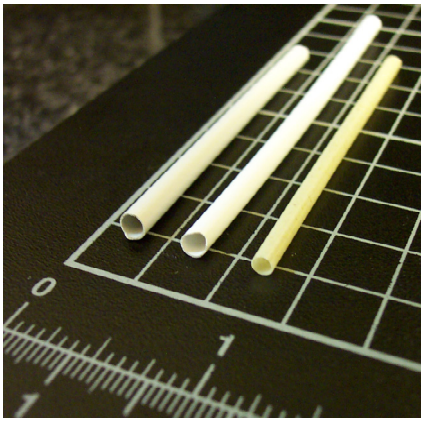
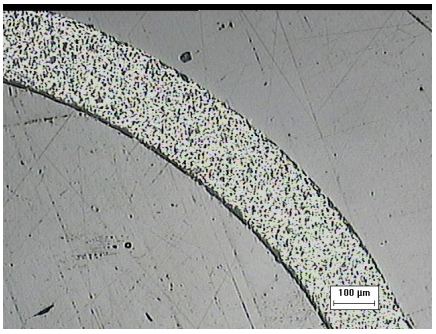


Figure 1.- Micrograph of tubes at different stages: green (left), brown (middle) and sintered (right)



~ 200 μm

Figure 2.- Optical micrograph of sintered extruded .

MULTIPLE PROFILES PROTECTIVE COATINGS FOR PIPELINES AND VESSELS

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The present work deals with the synthesis of highly effective fluorine-free anti- adhesive protective glass coatings designed for pipelines and vessels, made on the base of non-deficient complex materials.

The process of glass formation in the compositions containing technogenic raw materials was studied.

Optimal ratios of oxides were defined, which determined high physical-chemical and technological properties of protective coatings.

The created enamels and glass coatings passed industrial testing in one of the leading plants of chemical aggregates of Ukraine and these coatings were estimated positively.

The obtained enamels and glass coatings are distinguished by their low burning temperature, a wide temperature range of coating formation, high quality of surface purity, thermal shock resistance (DIN 51167), high acid and alkaline resistance (ISO 2743, ISO 2745) in extreme exploitation conditions compared to the known alternative glass coatings.

ANODE-SUPPORTED SOLID OXIDE FUEL CELLS (SOFCs) BASED ON THIN FILMS OF DOPED CERIA ELECTROLYTES

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The utilization of anode-supported electrolytes is a useful strategy to increase the electrical properties the solid oxide fuel cells, because it is possible to decrease considerably the thickness of the electroly So, cylindrical anodes were fabricated successfully using nanometric precursor powders. Mixtures powders of NiO and gadolinium doped ceria (GDC) with different particle sizes and compositions w prepared by sol-gel related techniques and pressed at 1 - 3 Tm/cm^2 to obtain precursor cylindr cermets to be used as anodes for anode-supported solid oxide fuel cells.

Nanometric doped ceria powders with different compositions ($Ce_{1-x}Gd_xO_{2-y}$) were also prepa by sol-gel related techniques to be used as electrolyte thin films (~10 - 30 µm). Then, the nanometric c based powders were deposited by dip coating on the cermet anodes at different thickness, using an prepared with the nanometric GDC powders dispersed in a commercial liquid polymer. We have optimi the temperature for the preparation of a dense thin film electrolyte on the porous NiO-GDC substrate tc used as anode. Densification of the electrolyte powders was carried out by uniaxial pressing a deposition of the ink on the cermet anodes, followed by a heat treatment at high temperature (~135 1450 ºC). Gd doped ceria was previously mixed with Co acetate at different compositions (1 - 3 % weight) in order to decrease the densification temperature of the electrolyte on the NiO-GDC anode reduction on Ar/H_2 5% at 900º C gives an optimal Ni-GDC cermet without reduction of de GDC electrol The thickness of electrolytes so fabricated was determined by SEM analysis. Porosity of the anoc composition and grain distribution was also analyzed by EDAX and SEM spectroscopy.

Finally, nanometric ceramic powders of $Ln_{1-x}M_xCoO_3$ where Ln = lantanide and M = Sr, Ba a $Ba_{0.5}Sr_{0.5}Fe_{0.2}Co_{0.8}O_{3-\delta}$ (BSFCO) for cathodes deposition were also prepared by sol-gel related methoc The powders for cathodes were deposited directly on the electrolyte thin films by dip coating usin mixture of the powders with the same commercial resin utilized for the anodes preparation. The electr properties of the fuel cells so prepared were characterized in order to optimize the preparation procedu

SOLID OXYDE ELECTROLYTE TUBULAR CELLS FOR HYDROGEN PRODUCTION

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In the aim to develop highly efficient and low cost processes for hydrogen production, steam electrolysis at high temperatures is being studied in CEA within Pan-H research program. The objective is to develop tubular solid oxide electrolysis cells for operating temperatures at 800-850°C. In a first step, suitable electrodes and electrolyte materials for SOEC applications, either electrolyte-supported or electrode-supported cells, were selected after testing various commercial cells [1]. A second step, carried out this year and presented in this paper, consisted on the elaboration of opened electrolyte support tubular cell, in the aim of large scale hydrogen production, using the as-chosen electrodes and electrolytes materials; correlation between the layers microstructures and thicknesses with electrochemical performances was performed using planar cells. Tubular cells performances will be carried out in July 2009.

Yttria-stabilized zirconia (8YSZ) is the state-of-the-art material for the electrolyte support of SOEC. Both 8YSZ planar and tubular cells were fabricated by thermal spraying. Thicknesses between 150 to 350 microns were reached for planar cells with 60 mm to 120 mm in diameter; 350 microns thickness was reached related to the following tubular cell sizes: 200 mm high and 100 mm diameter.

Screen printing and dip-coating were performed to coat respectively planar and tubular electrolyte supports by electrodes. The anode electrode consisted in YSZ-LSM composite material (outside layer on tubular cell presented in figure 1) and the cathode electrode consisted in a composition gradient CGO-NiO layer (internal layer on tubular cell). Microstructure and thicknesses were studied by scanning electron microscopy: a 35 microns porous anode and a 60 microns porous cathode seemed to be suitable according to electrochemical tests performed on planar cells. Figure 2 shows that the electrochemical performance at 850°C of a 150 microns thickness thermal sprayed 8YSZ electrolyte with suitable anode and cathode is higher than an ESC2 commercial cell (HC Starck).

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Figure 1: tubular cells

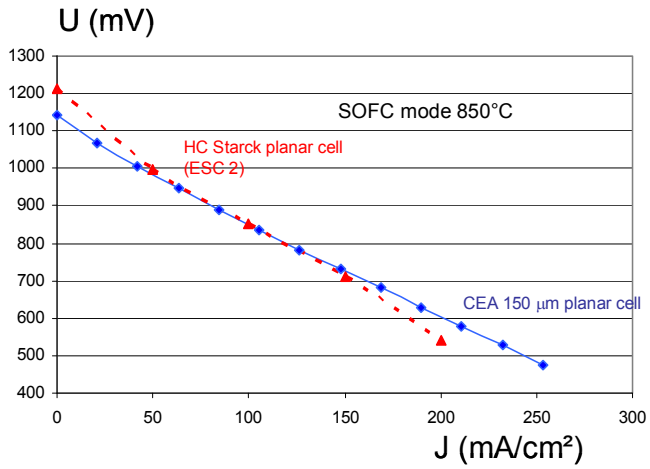

Figure 2 : electrochemical performances of a 150 microns planar cell.

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CORROSION RESISTANCE Of IRON POWDERS CLAD WITH PHOSPHORUS IN INORGANIC AND BIOMEDIA OF HUMAN ORGANISM

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Production and use of clad powdered materials are promising trends in powder metallurgy thanks to possibility of varying their technological and magnetic properties.

The properties of iron powders clad with phosphorus and their interaction with biomedia of human organism has been investigated. Iron powders of different dispersity (PZhRV 3.200.26 (Ukraine), ANS 100.29 (Sweden) and PZhV 3.342.28 (Russia)) were clad by using methods of chemical thermal decomposition of phosphorus compounds or thermochemical synthesis in a vibrating layer.

The obtained phosphorus-clad iron powders underwent corrosion tests in a 3.0 % NaCl solution. The calculation of the depth coefficient of the corrosion rate revealed that phosphorus-clad powders were characterized by much higher corrosion resistance in this solution (3-4 points by the ISO 1462-78(A) standard) as compared to the initial powder (1 point). This indicates that corrosion processes only take place on the surface of sample.

The initial iron powders of the PZhRV 3.200.26 and ANS 100.29 grades were shown to interact with human blood plasma much more intensely (by 5.8 and 7.2 times for PZhRV and ANS powders, respectively) than phosphorus-clad powders. The presence of phosphorus layers on the surface of iron particles results in forming a protective colloidal biocomplex with available blood plasma proteins due to the presence of dissociating groups that are constituents of amino acids radicals. This markedly enhances the stability of clad powders in biomedia.

As follows, cladding of iron powders with phosphorus significantly increases their chemical stability in both biomedia containing human blood plasma and in air.

THERMAL BEHAVIOUR OF KAOLINITE POWDERS: MULTI-STEP DEHYDROXYLATION AND HIGH-TEMPERATURE PHASES

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The thermal dehydroxylation of kaolinite and formation of high-temperature phases (mullite and cristobalite) from the dehydroxylated amorphous phase (metakaolinite) have been extensively studied for many years. It is very important when the manufacture of ceramics from kaolinite and clays containing kaolinite is considered. However, the results reported in the literature are not satisfactorily consistent due to several factors, such as different studied samples, particle size, impurity contents, grinding treatments and others, that influence the thermal behaviour, besides the instrumental techniques used. These all factors produce several effects on the thermal behaviour of kaolinite samples, with dehydroxylation at relative lower temperature than 540-560 ºC (endothermal maximum peak of DTA at heating rate 10 ºC/min), formation of amorphous dehydroxylated phase (metakaolinite), and mullite and cristobalite formation at different temperatures in the range 900-1500 ºC, depending on the particular sample. Several authors consider that the dehydroxylation follows a diffusion model, as well as a change in the mechanism may occur during the dehydroxylation because it is a complex reaction, with influence of heat and mass transfer phenomena. For instance, the activation energies reported in several works show disagreements between values depending on water pressure, besides which is the rate-controlling kinetic mechanism of the reaction.

In the present work, a sample of kaolinite was studied to re-examine the kinetics of kaolinite dehydroxylation using small sample weights, homogeneus particle size distribution and high-vacuum conditions that avoid the influence of heat and mass-transfer phenomena. The experiments involve the application of the technique of Controlled Rate Thermal Analysis (CRTA), which was developed to reduce pressure and temperature gradients throughout the sample. An electrobalance connected to a high vacuum system equipped with a penning gauge was used for the CRTA experiments. Kinetic analysis has been carried out on the basis of the basic kinetic equation of the reaction rate and advanced isoconversional method. The activation energy (E) was calculated and the determination of whether E varies with reaction. The kinetic model can be estimated from the analysis of the pseudoisothermals by the reducing time plots. High-temperature experiments were performed using an electric furnace, in air, from 900 to 1550 ºC using pressed samples at 40 MPa previously dried. Complementary techniques (XRD, SEM-EDX, Dilatometry, Bulk Density, Porosity measurements, etc.) have been also used.

It has been found that there are at least two different dehydroxylation stages, revealing the multi-step nature of this reaction. The activation energy for the first step, nucelation and the growth of nuclei, decreases from 100 to 75 KJ/mol. The second stage corresponds to a difussion process and the activation energy rises to 120 KJ/mol because the metakaolinite formation. This amorphous phase, with the same morphology of the original kaolinite, closes the interlamellar channels and leaves isolated patches of kaolinite from which the water escapes with difficulty.

On the other hand, the nucleation and development of mullite and cristobalite crystals from metakaolinite have been also examined. According to XRD results, low-crystalline mullite appears after firing at 1000 ºC for 0.5 h, with maximum development at 1300 ºC for 2 h. Cristobalite produced from amorphous silica crystallization is detected after firing at 1300 ºC for 1h. Single phase mullite is present in the fired sample at 1500 ºC. These results are related with the evolution of bulk density and porosity of the pellets after firing, with important microstructural changes produced by crystal growth, the increase of the glassy phase and reaction kinetic.

POWDER PROCESSING OF LAYER SILICATES BY DRY GRINDING: A BIDIMENSIONAL PARTICLE SIZE MODEL

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Wet or dry grinding are commonly used as procedures for the preparation of fine and reactive powders and further processing steps. Deagglomeration and elimination of soft aggregates are produced by wet grinding. In contrast, dry grinding is most intensive when acting on solids, in particular using equipments such as vibratory, oscillating and planetary mills. It originates mechanical stress which are quite diverse, producing important changes such as lattice distortions, amorphization, decrease in particle size, increase in surface area, etc. Thus, the reactivity of ground nanostructured solids is enhanced and, therefore, it leads to mechanochemical reactions. From this, there is a general interest in the study of the physical and chemical induced effects by grinding in solids.

The effects of dry grinding on layer silicates have been extensively studied due to their relevant importance in some applications, for instance kaolinite and talc as ceramic raw materials. In the present work, several samples of kaolinite (1:1 layer silicate) and talc (2:1) have been selected. Dry grinding experiments by planetary milling have been performed in order to examine the effects of different conditions in the resultant powders. Several techniques have been used to follow the evolution of the layer silicates, mainly XRD, nitrogen adsorption, SEM, TEM, and particle size analysis. Modifications of surface area, particle size and shape of short and prolonged grinding on the crystal structure of the layer silicates have been evaluated.

The observed changes have been related to a progressive delamination and structural breakdown during grinding, with final formation of a turbostratic-type structure characterized by a partial or complete absence of three-dimensional order of the finest particles, mainly along [00*l*]. Short grinding times resulted in the breakdown and drastic size reduction of relatively thin particles with an increase of surface area. However, the rate of size reduction (particle size and domain thickness) decreased with time and a limit is reached. Increasing grinding produced a decrease of surface area. These changes also affected the pore size distribution from nitrogen adsorption measurements. It can be also observed an increasing in amorphization as a consequence of the loss of long-range order. The particles gradually lost their original layered shape with grinding, with a more isometric particle shape, producing round-edged particles with a rugged surface, as observed by electron microscopy and calculated shape factors. Taking into account all these results, it has been developed a bidimensional model of particle size evolution and changes of particle shape on grinding these 1:1 and 2:1 layer silicates, under the present experimental conditions, for short and prolonged grinding. Relevant examples of the utility of this model in dry powder processing will be show.

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MICROSTRUCTURE DESIGN BY MECHANICAL ALLOYING

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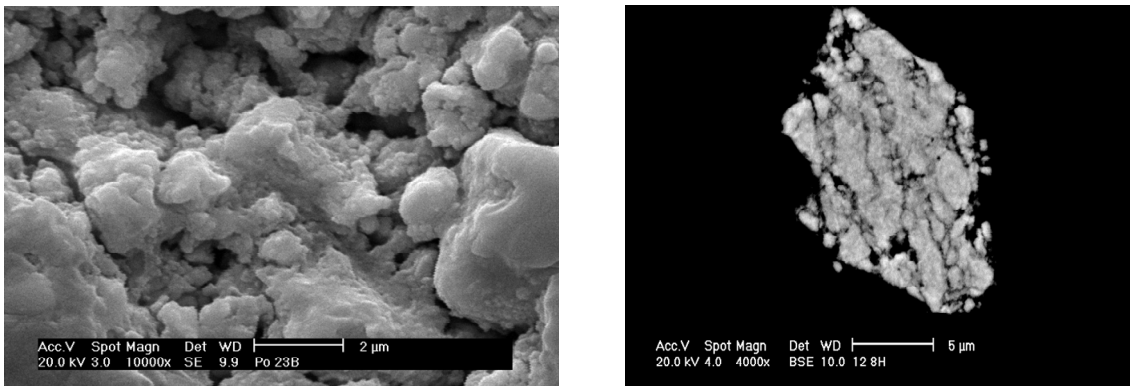
Mixing and coprecipitation processes are, often, not enough in order to reach materials holding several functional components, like selective catalysts that must work simultaneously. Even thought when a homogeneous and fine distribution of the constituents is obtained, the affinity between equal phase particles leads to coarsening during the consolidation (sintering) process, as well as on application, such as the material can loose high reactivity. The present work proposes a new consolidation route – Sintering by Activated Surface (SAS) – that employs sacrificial metal layers to avoid coarsening and to increase the diffusion profiles during sintering, once high activity surfaces are exposed during the first sintering step. Regarding limited oxygen potential is established in the sintering atmosphere, the SAS effect is engaged when a specific projected powder microstructure obtained by mechanical alloying (MA) processing is provided. The MA is driven in such a way that yields cermet powders particles with lamellar pod-of-pie like structures, as shown in the SEM image. This projected morphology comprises the ceramic round particles plated by thin metal layers or embedded on them.



SEM images revealing the MA powder morphology.

Porous nickel-zirconia based cermets are studied with Cu and some selected refractory metal additives. The refractory metals are expected to repeal Cu, which remains in pure state at the cermet. By its turn, Cu addition is postulated to prevent coking when fuel-reforming reactions are involved at the application (e.g. in solid oxide fuel cells). Furthermore, Cu is desired since it promotes shrinkage and lower the sintering temperatures. The SAS process running under argon atmospheres with controlled oxygen partial pressure is found to further reduce the sintering temperature by 100 to 300°C, for cermets final densities above 60%TD. The sintering behaviour depends on the chosen additive, being Ag, Cu and Mo the most effective ones. The resulted sintered parts attain a suitable density and phase dispersion for catalysis applications.

The authors acknowledge the research councils FAPESP, CNPq, FINEP and CAPES for the financial support.

MULTIFUNCTIONAL HETERO-MODULE COMPOSITE IN B_4C-BN-TiC-SiC-C SYSTEM

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Experiments have been carried out to develop multifunctional composites with improved properties and structure for using in air turbines, ballistic armor, cutting tools and atomic reactors.,

For optimization of sintering process, microstructure improvement and viscosity increase we used MgO, Y_2O_3 additives.

Carbon fiber (S-242 L_{av}=370μm, d=13μm) stipulated, also, the maintenance of carbide stoichiometric composition.

Sintering was done in vacuum with hot pressure (25 MPa) in the interval of 1600-1700^{0}C with dwell time 5-10 minutes.

Structural analysis proved the homogeneous structure of the composite. The properties satisfy ISO standards. HV=30 GPa, compressive strength 3300 MPa, HRA=93, bending strength – 700-800 MPa.

SHAPING AND SURFACE MODIFICATION OF METAL PARTICULATED CERAMIC-Nb POWDER COMPOSITE

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Instituto de Ciencia de Materiales de Madrid, CSIC, Cantoblanco Madrid 28049, Spain
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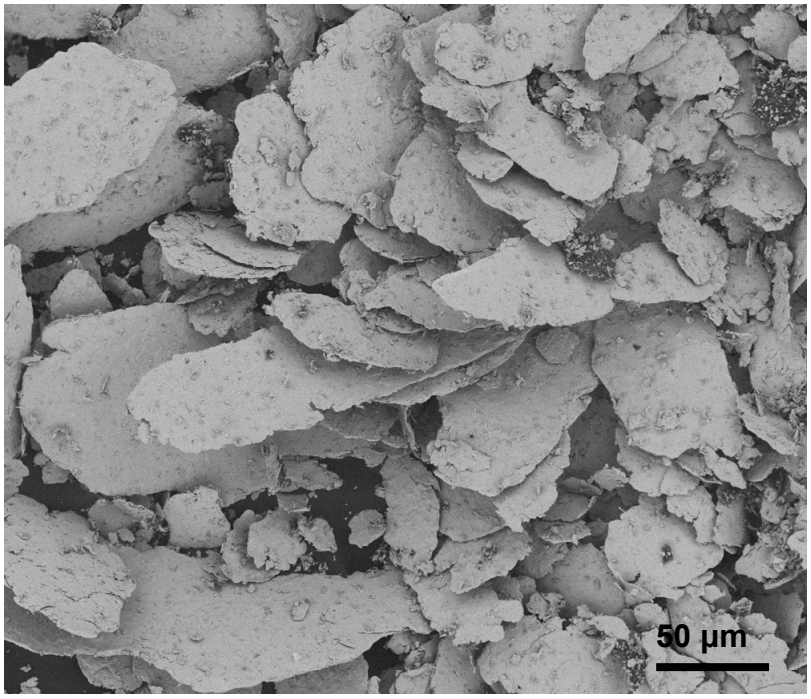
Nb commercial powder was attrition milled in isopropilic alcohol using Al_2O_3 and 3Y-TZP balls as grinding media. The morphological evaluation of the milled particles was carried out by Scanning Electron Microscopic (SEM). It was found that during milling the particle morphology of Nb powder changes from initial globular to flaky-shape particles. The flaky-shaped particles were supposed to be formed by breaking-off of sticked layer on ball surfaces and mill container walls by the impact actions of milling balls. It was also found that small fragment (< 5 μm) of broken ceramic balls have been embedded into the metal powders during the milling process. On the other hand the surface characterization of the metal powders was investigated by means of X-ray photoelectron spectroscopy (XPS). XPS measurements indicated that both Al_2O_3 and 3Y-TZP particles were present on the surface of the metal. Additionally an oxidizing passive thin layer of Nb_2O_5 ~ 3 nm in thickness was covered the milled metal particles. It was suggested that the squeeze into elongated flakes and surface modification of Nb powders induced by milling processing offers the possibility to design interface properties of ceramic matrix reinforced with flaky Nb particles. This can affect the mechanical stability and properties of final ceramic-metal composites, i.e. 3Y-TZP/Nb.



SEM micrograph of flaky shape niobium powder after wet ball milling.

POROUS CERAMICS IN COMBUSTION APPLICATIONS

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Increasing combustion efficiency and reducing harmful emissions are hot topics in gas natural combustion applications. There are some basic properties that ceramics should accomplish to be applied as radiant burners, such as low thermal expansion coefficient, chemical stability at the combustion temperature, good mechanical stability and high porosity. The pore size is a critical parameter for achieving flame stability avoiding either flame lift-off or flash-back. The pore size of burners depends fundamentally on the type of burner either surface or submerged required. The surface radiant burners need pores in the range of 1.2-1.6 mm in diameter and low thermal conductivity materials like mullite and cordierite, as they must support very high temperature gradients, about 900ºC over 1cm. Conversely, in porous submerged burners the flame is confined inside the ceramic structure and then large pores, diameters in the range of 3-5 mm, and high thermal conductivity materials are required. In the present work, different ceramic structures were fabricated using the replica method, based on the impregnation of a cellular polymeric structure with a ceramic suspension in order to produce a macroporous ceramic exhibiting the same morphology as the original porous material (Figure 1). Different types of ceramic structures varying the porosity level and the pore size were fabricated from cordierite and SiC-based ceramics, and two types of burners were designed and rig tested under different combustions conditions, using a wide range of power and different gas mixtures. In particular quite energetic and less contaminant mixtures of natural gas and hydrogen in proportions of up to 50 % were tested.

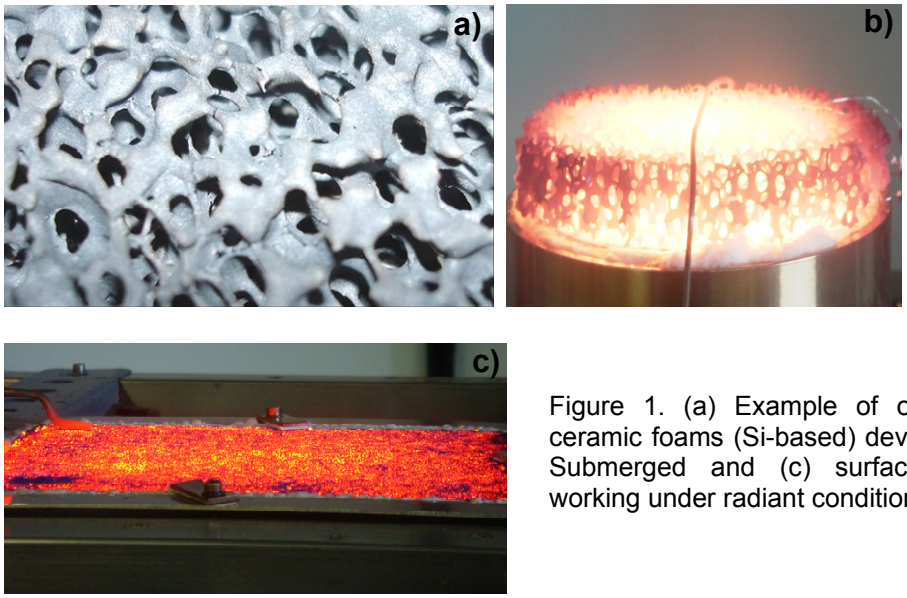

Figure 1. (a) Example of one of the ceramic foams (Si-based) developed. (b) Submerged and (c) surface burners working under radiant conditions.

ZrO_2 FOAMS FOR HEAT RECUPERATIVES IN GAS BURNERS

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Refractories porous ceramics are particularly adequated for using as thermo diffusers, gas and liquid dispersers and others technologies. In this work, ZrO_2 foams with low relative density were developed through the replication method using polyurethane foams (PUF) for application as porous radiant burners. The ceramic foams were produced by impregnation of open – cell PUF with aqueous suspensions varying solid fractions (30 wt% - 50 wt%) of raw materials. Zeta potential was calculated and flow curves were performed in order to adequate suspensions for impregnation processing. A careful calcination study performed before sinterization in order to improve mechanical strength of the ceramics foams. Ceramics with dimensions of 20x20 cm were succeeded through sinterization process at 1350ºC, showing good structural stability.

INFLUENCE OF Y_2O_3 ADDITION TO Mg-PSZ CERAMICS ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES

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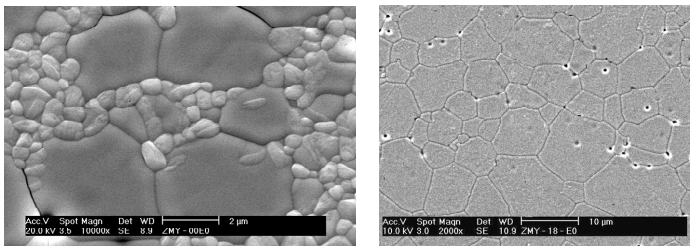
Y-TZP, Yttria doped tetragonal zirconia polycrystalline exhibit high strength and toughness. However these ceramics degrade at low temperatures, especially when exposed to humid environments, where tetragonal to monoclinic transformation occurs because of reaction of water with tetragonal grains of Y-TZP. Mg-PSZ, magnesia partially stabilized zirconia has better resistance to low temperatures, its fracture toughness is higher, but the strength of Mg-PSZ is lower than of Y-PSZ. The addition of Y_2O_3 to MgO–ZrO_2 system is known to improve mechanical properties of Mg-PSZ by decreasing eutectoid decomposition. Y_2O_3-MgO- ZrO_2 crystalline at low temperatures was prepared by co-precipitation process. The addition of 1.7mol% Y_2O_3 in MgO–ZrO_2 structure was investigated and it was compared to 9% mol MgO–ZrO_2 ceramics. Precursor powders were ground into a high energy grinder mill for 4h. Those powders were uniaxially pressed (98Mpa) into pellets with 10 mm diameter and thickness, which were sintered at 1500 ^oC for 1h. Mechanical properties were evaluated by Vickers indentation technique. The microstructure of sintered body of both ceramics has been examined by SEM. The crystalline phases of sintered ceramics where analyzed by XRD. Dense ceramics (99% of theoretical) were obtained, after sintering.



(a) (b)

SEM images of sintered MgO–ZrO_2 (a) and Y_2O_3–MgO–ZrO_2 (b) ceramics.

COMPACTION AND SINTERING PROPERTIES OF STONEWARE BASED TILES.

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Nowadays, the production of stoneware based tiles had increased because of their excellent mechanical properties and resistance to chemical attack. In addition, it presents aesthetic advantages and large formats that allow their use in technical applications as facades. Several relevant aspects are considered in the ceramic processing in order to obtain final materials with a reduction of the mass per square meter. One of the most critical steps is the compaction process because of the low modulus of rupture of green tiles that limited their thickness.

In this work we studied the effect of glass particles addition in the compaction behaviour of green tiles. For such a reason glass particles consisted of frits or recycled glass were used. The glass addition ranged from 1%wt to 20%wt and the effect of the particle size was considered. The incorporation of the glass followed a standard stoneware tile processing that consisted on a ball-milling step to form a slip that, after spray drying was uniaxially pressed and the sintered. Industrial kiln performance was compare with laboratory furnace experiments.

It was evaluated the addition of the glass on the response by using compaction curves of the powders. Density, shrinkage, porosity, water absorption, surface roughness and modulus of rupture were determined on both green and sintered tiles. Resulting properties are correlated with the composition, percentage and the particle size of the added glass.

The additions of the glass produced an increasing of the powder compaction process that allows increasing the modulus of rupture of green tiles in spite of their lower density. The flux components of the glass produced higher density in the material after sintering but with an important increasing of their pyroplastic behaviour. The stoneware tile composition was reformulated to compensate this effect.

PROCESSING AND PROPERTIES OF TUBULAR ASYMMETRIC MIXED CONDUCTING MEMBRANES

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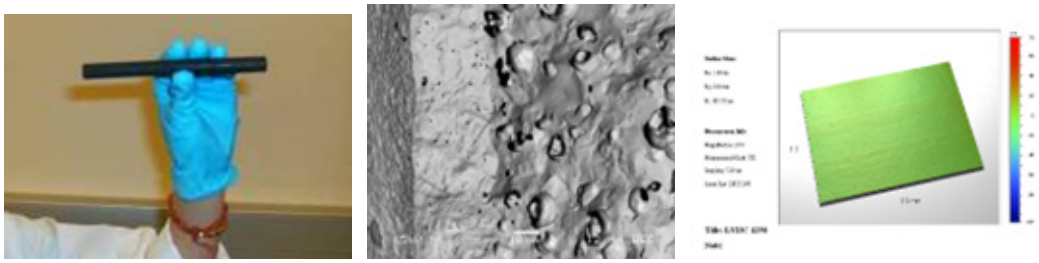
Dense inorganic membranes are prospected for the selective separation of oxygen from air for power generation. Transport of oxygen from one side of the membrane to the other occurs through bulk diffusion in the lattice of the mixed oxygen ion and electron conducting material. Both membrane thickness and kinetics of oxygen surface exchange contribute to determine the oxygen flux across the membrane. The membrane is therefore typically designed as an asymmetric structure with a thin dense layer coated on a mechanically strong porous support. For commercial deployment of this technology, low cost reliable membrane processing and compatible sealing technology for module fabrication should be developed.

In the present work, extrusion is chosen for the production of porous ceramic tubes of perovskite related materials of ca. 15 cm length and 1.5 mm wall thickness. Pyrolyzable pore formers are used to tailor their porosity. The green tubes are heat-treated in air with careful monitoring of the debinding step and to achieve sufficient mechanical strength. The thin selective layer is then deposited by dip-coating using a colloidal ceramic suspension. A final sintering step is carried out to obtain the desired asymmetric structure. Within this process, several parameters are readily adjustable to produce properties-driven membranes, including layer thickness, grain size, porosity (pore size, volume and distribution) and texture. This will be illustrated in this presentation by means of several techniques applied for the thorough characterization of the produced membranes (XRD, SEM, EDS, N2 adsorption/desorption, white light interferometry …). The results of this analysis are used to establish correlations between oxygen flux and membrane's characteristics. This further drives optimization of membrane design.



(a) (b) (c)

Picture of green tube (a). SEM micrograph (b) and surface roughness (c) of sintered asymmetric tube.

Glass ceramic seals are investigated in parallel for the development of membranes modules. The composition of the selected silicate-based glasses is tuned to match membrane material expansion during operation, and to minimize reactivity. The produced seals are studied for their thermal behaviour and structure as a function of the annealing treatment. The retained glass ceramic materials are used for the preparation of water-based polymeric glue. Seal/membrane joint experiments are carried out to define a suitable sealing procedure and to assess the compatibility between the two materials. Results of this work will also be discussed in this presentation.

The prepared asymmetric tubular membranes may also be sealed to high temperature alloy housings using silver-copper based air-brazing. This has been demonstrated for dense (symmetric) tubular membranes of La_2NiO_4, assembled in single-tube modules, which have been tested for oxygen permeation in controlled atmosphere at temperatures up to 900°C.

Acknowledgement:
This publication forms a part of the BIGCO2 project, performed under the strategic Norwegian research program Climit. The authors acknowledge the partners: StatoilHydro, GE Global Research, Statkraft, Aker Clean Carbon, Shell, TOTAL, ConocoPhillips, ALSTOM, the Research Council of Norway (178004/I30 and 176059/I30) and Gassnova (182070) for their support.

Shaping 4

Final Programme

SHAPING-4 CONFERENCE

Madrid (Spain) November 15-18, 2009

Programme

SUNDAY 15th November

16:00 - 18:00 h Registration at Conference site

18:00 - 19:00 h Opening session and Plenary Lecture (PL)

COLLOIDAL APPROACH TO RAPID SHAPE FORMING,
F. F. Lange.
Materials Department, University of California at Santa Barbara, Santa Barbara, CA 93111, USA. flange@engineering.ucsb.edu

19:00 - 21:00 h Welcome Cocktail

MONDAY 16th November

Session 1 (S1) Chair persons: J. Halloran & P.J. Sánchez-Soto

08:30 - 09:00 h Invited Lecture (IL1)

PROCESSING AND PROPERTIES OF NANOSTRUCTURED YSZ CERAMICS.
J. Binner, B. Vaidhyanathan, A. Annapoorani, S. Huang, and J. Bai.
Department of Materials, Loughborough University, Loughborough, UK.
j.binner@lboro.ac.uk

09:00 - 09:30 h Invited Lecture (IL2)

MONO- AND OLIGOSACCHARIDES IN DEFLOCCULATION PROCESS OF NANOCERAMIC POWDERS.
M. Szafran, P. Falkowski, and A. Danelska.
Warsaw University of Technology, Faculty of Chemistry, Warsaw, Poland.
szafran@ch.pw.edu.pl

09:30 - 09:45 h (S1-1) CONTROL OF MICROSTRUCTURE IN CERAMICS BY SLIP CASTING UNDER A STRONG MAGNETIC FIELD.

T. S. Suzuki, T. Uchikoshi, and Y. Sakka.
Fine Particle Processing Group, Nano Ceramics Center, National Institute for Materials Science, Tsukuba, Ibaraki, Japan. SUZUKI.tohru@nims.go.jp

09:45 - 10:00 h (S1-2) CRITICAL PARTICLE SIZE FOR SHAPING DENSE CERAMIC BODIES BY SLIP CASTING.
C. Tallon, M. Limacher, and G. V. Franks.
Department of Chemical and Biomolecular Engineering, University of Melbourne, Melbourne, Australia, tallon@unimelb.edu.au

10:00 - 10:15 h (S1-3) HYBRIDIZATION OF TEXTURE INDUCING PROCESSES.
E. Suvaci[1], K. Keskinbora[1], T. S. Suzuki[2], and Y. Sakka[2].
[1] Department of Materials Science and Engineering, Anadolu University, Eskisehir, Turkey, [2]Fine Particle Processing Group, Nano Ceramics Center, National Institute Materials Science, Tsukuba, Ibaraki, Japan, esuvaci@anadolu.edu.tr

10:15 - 10:30 h (S1-4) POWDER PROCESSING FOR TRANSPARENT POLYCRYSTALLINE ALUMINA.
M. Stuer[1,2], P. Bowen[1], and Z. Zhao[2].
[1] Powder Technology Laboratory, Material Science Institute, Swiss Federal Institute of Technology, Lausanne, Switzerland, [2] Department of Physical, Inorganic and Structural Chemistry, Arrhenius Laboratory, Stockholm University, Stockholm, Sweden, michael.stuer@epfl.ch

10:30 - 10:45 h (S1-5) DEVELOPMENT OF GRAIN ORIENTED TUNGSTEN BRONZE CERAMICS WITH MAGNETIC FIELD.
T. Kawase, E. Yaegaki, S. Tanaka, and K. Uematsu.
Department of Materials Science and Technology, Nagaoka University of Technology, Kamitomioka, Niigata, Japan, keizouematsusan@hotmail.co.jp

10:45 - 11:00 h (S1-6) REALISATION OF CERAMIC HOLLOW FIBER GAS SEPARATION MEMBRANES BY SPINNING WITH PHASE INVERSION.
F .M. M. Snijkers, C. Buysse, J. J. Luyten , M. Schillemans, and A. Buekenhoudt.
Flemish Institute for Technological Research (VITO), Mol, Belgium, frans.snijkers@vito.be

11:00 - 11:30h Coffee Break

Session 2 (S2) Chair persons: F. Rossignol & M.I. Nieto

11:30 - 12:00 h Invited Lecture (IL3)
ELABORATION OF TAILORED MILLIMETRIC POROUS CERAMIC SPHERES BY COLLOIDAL WAY.
C. Pagnoux.
SPCTS, ENSCI, CNRS, Limoges, France, cecile.pagnoux@unilim.fr

SHAPING-4 CONFERENCE. Final programme.

12:00 - 12:15 h (S2-1) PARTICLE PACKING IN PARAFFIN-WAX SUSPENSIONS USED FOR LPIM.
A. Dakskobler and T. Kosmač.
Engineering Ceramics Department, Jožef Stefan Institute, Ljubljana, Slovenija, ales.dakskobler@ijs.si

12:15 - 12:30 h (S2-2) POWDER-BINDER-SEPARATION IN INJECTION MOULDED GREEN PARTS.
A. Mannschatz, S. Höhn, and T. Moritz.
Fraunhofer Institute for Ceramic Technologies and Systems, Dresden, Germany, Anne.Mannschatz@ikts.fraunhofer.de

12:30 - 12:45 h (S2-3) AQUEOUS DISPERSION OF TUNGSTEN POWDER FOR INKJET PRINTING PROCESS.
J. Pommay, M. Lejeune, C. Dossou-Yovo, M. Mougenot, and R. Noguera.
SPCTS-UMR 6638, Limoges, France, CERADROP - ESTER Technopole, Limoges, France, judith.pommay@etu.unilim.fr

12:45 - 13:00 h (S2-4) SOLID FREEFORM FABRICATION OF Al_2O_3/TiO_2 GRADIENT MATERIALS.
C. M. Gomes[1], N. Travitzky1, P. Greil[1], O. R. K. Montedo[2], A. P. N. de Oliveira[2], and D. Hotza[2].
[1] Department of Materials Science, Institute of Glass and Ceramic, Erlangen, Germany. [2]Group of Ceramic and Glass Materials (CERMAT), Federal University of Santa Catarina (UFSC), Florianópolis, Brazil, nahum.travitzky@ww.uni-erlangen.de

13:00 - 13:15 h (S2-5) NEW FRONTIERS IN CERAMIC MICRO SHAPING TECHNOLOGIES.
Y. DeHazan, J. Heinecke, and T. Graule.
EMPA Swiss Federal Laboratories for Materials Testing and Research, Laboratory for High Performance Ceramics, Dübendorf, Switzerland, Yoram.dehazan@empa.ch

13:15 - 13:30 h (S2-6) PHOTOPOLYMERIZATION OF CERAMIC SUSPENSIONS.
J. W. Halloran and V. Tomeckova
Department of Materials Science and Engineering, University of Michigan, Michigan, USA, peterjon@umich.edu

13:30 - 15:00 h Lunch

15:00 - 15:30 h Invited Lecture (IL4)
INNOVATIVE PRODUCTION PROCESSES FOR CERAMIC MEMS/NEMS.
M. Schulz and T. Hanemann.
Forschungszentrum Karlsruhe, Institute for Materials Research III, Karlsruhe, Germany. Department of Microsystems Engineering - IMTE, University of Freiburg, Germany, Michael.Schulz@imf.fzk.de

15:30 - 17:00 h Poster Session-1 (P1)

(P1-1) CONDUCTING ALUMINA PARTICLES: EFFECT OF IONIC STRENGTH AND pH ON ZETA POTENTIAL.

R. C. D. Cruz[1,3], A. M. Segadães[2], R. Oberacker[3], and M. J. Hoffmann[3].
[1] Univ. Caxias do Sul, Dept. Mechanical Eng., 95070-560 Caxias do Sul, Brazil, [2]Univ. Aveiro, Dept. Ceramics and Glass Eng. (CICECO), 3810-193 Aveiro, Portugal [3]Univ. Karlsruhe, Inst. Ceramics in Mechanical Eng., 76131 Karlsruhe, Germany, rcdcruz@ucs.br

(P1-2) CHEMICAL STABILITY OF AQUEOUS/NONAQUEOUS (V,Zr)SiO_4 SUSPENSIONS.

E. Ozel, S. Akdemir, and E. Suvacı.
Department of Materials Science and Engineering, Anadolu University, 26480 Eskisehir, Turkey, eozel@anadolu.edu.tr

(P1-3) IMPROVEMENT OF THE DISPERSION OF THE MWCNT IN A ZIRCONIA MATRIX BY THE ADDITION OF PARTIALLY COATED MWCNT AND COLLOIDAL PROCESSING.

N. Garmendia[1], I. Santacruz[2,3], R. Moreno[2], and I. Obieta[1].
[1]Unidad de Salud, INASMET-TECNALIA, San Sebastián/Gipuzkoa, Spain. [2]Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain. [3]Departamento de Química Inorgánica, Cristalografía y Mineralogía, Universidad de Málaga, Málaga, Spain,
nere.garmendia@inasmet.es

(P1-4) KINETICS AND SYNTHESIS MECHANISM OF CORDIERITE BY KAOLIN/TALC/ALUMINA MIXTURE IN SLIP CASTED BODIES.

J. B. Rodrigues Neto[1], D. Hotza[2], and R. Moreno[3].
[1] Sociedade Educacional de Santa Catarina - SOCIESC. Rua Albano Schmith, 3333 - 89206-001 - Joinville - Brasil. [2]Universidade Federal de Santa Catarina - CTC - ENQ - Caixa Postal 476 - 88040-900 - Florianópolis - Brasil. [3]Instituto de Cerámica y Vidrio - CSIC - C/ Kelsen, 5 - 28049 - Madrid - Spain,
jbrn.ufsc@gmail.com

(P1-5) PRESSURE FILTERING AND DENSIFICATION OF FINE GRAINED MAGNESIUM ALUMINATE SPINEL.

F. Orgaz.
Instituto de Cerámica y Vidrio, CSIC. Campus de Cantoblanco. Madrid, Spain,
Felipe.orgaz@icv.csic.es

(P1-6) FORMULATION OF DIELECTRIC INK FOR FABRICATION OF HIGH POWER CERAMIC CAPACITORS BY INK-JET PRINTING PROCESS.

N. Bouvier[1], M. Lejeune[1], F. Rossignol[1], S. Guillemet[2], C. Dossou-Yovo[3], Rémi Noguera[3], and J. Sarrias[4].

[1] SPCTS-UMR 6638, 47 à 73, Avenue Albert Thomas, 87065 Limoges Cedex, France, [2]CIRIMAT-UMR 5085, Université Paul Sabatier, 118, route de Narbonne, 31062 Toulouse Cedex 9, France, [3]CERADROP, 1 avenue d'Ester, BP 36921, 87069 Limoges, France, [4]MARION Technologies, Parc Technologique Delta Sud, 09340 Verniolle, France, nicolas.bouvier@etu.unilim.fr

(P1-7) INKJET PRINTING OF FUNCTIONAL MATERIALS FOR CERAMIC ELECTRONIC APPLICATIONS.

C. Dossou-Yovo[1], M. Mougenot[1], M. Bessaudou[1], N. Bernardin[1] , F. Charifi[1], C. Coquet[1], R. Noguera[1], E. Beaudrouet[2], and M. Lejeune[2],

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(P1-8) RAPID PROTOTYPING TECHNIQUE FOR CERAMIC MINIDEVICES CONTAINING INTERNAL CHANNELS WITH ASYMMETRICAL CONTOUR.

R. F. Louh, Y. Ku, and I. Tsai.

Dept. of Materials science and Engineering, Feng China University, Taichung, Taiwan 40724, rflouh@fcu.edu.tw

(P1-9) MICRO POWDER INJECTION MOULDING OF ALUMINA DENTAL BRACKETS.

P. Thomas[1], A. Cervera[2], B. Levenfeld[1], S. Laddha[3], S. Vallury[3], G. Lingam[3], S. Atre[3], and A. Várez[1].

[1] Materials Science and Engineering Department. Universidad Carlos III de Madrid. Avda. de la Universidad, 30. 28911 Leganés. SPAIN, [2]Euroortodoncia. Polígono Industrial Urtinsa. 28923-Alcorcon. SPAIN, [3]Oregon State University. Corvallis, OR 97330. USA, pthomas@ing.uc3m.es

(P1-10) WATER DEBINDING KINETICS OF CERAMIC INJECTION MOULDING FEEDSTOCKS.

V. Dupont[1], C. Delmotte[1], J. P. Erauw[1], F. Cambier[1], T. Boulanger[2], C. Emmerechts[2], B. Guerra[2], and E. Beeckman[2].

[1] Belgian Ceramic Research Centre (BCRC), 4, Avenue Gouverneur Cornez, B-7000 Mons (Belgium), [2]Sirris, 12, Rue du Bois Saint-Jean, B-4102 Seraing, Belgium, v.dupont@bcrc.be, c.delmotte@bcrc.be

(P1-11) ELECTROHYDRODYNAMIC FORMING OF CERAMIC COMPONENTS FROM A PRECERAMIC POLYMER.

P. Colombo[2,3,1], M. Nangrejo[1], E. Bernardo[2], U. Farook[1], Z. Ahmad[1], E. Stride[1], and M. Edirisinghe[1].

[1] Department of Mechanical Engineering, University College London, Torrington Place, London WC1E 7JE, UK, [2]Department of Mechanical Engineering - Materials Division, University of Padova, 35131 Padova, Italy, [3]Department of Materials Science and Engineering, Pennsylvania State University, University Park, PA 16802, USA, paolo.colombo@unipd.it

(P1-12) PROCESSING OF YTTRIA BY GEL-CASTING.

A. L Costa[1], A. Sangiorgi[2], P. Pinasco[1], B. Ballarin[2], and A. Sanson[1].

[1] Institute of Science and Technology for Ceramics (ISTEC-CNR), Via Granarolo 64, 48018 Faenza-Italy, [2]Department of Industrial Chemistry and Materials, University of Bologna, Viale Risorgimento 4, 40136 Bologna - Italy, anna.costa@istec.cnr.it

(P1-13) FABRICATION OF INTERCONNECTED POROUS CERAMIC PARTS BY SELECTIVE LASER GELLING.

F. H. Liu[1] and Y. S. Liao[2].

[1] Department of Mechanical Engineering, Lunghwa University of Sci. and Technol., Taiwan, [2]Department of Mechanical Engineering, National Taiwan University, Taipei, Taiwan, fhliu@mail.lhu.edu.tw

(P1-14) OPTIMIZATION OF FABRICATION PARAMETERS OF CELLULAR ALUMINA STRUCTURES TO BE USED AS FILTERS.

A. M. Montes and J. A. Escobar.

Mechanical Engineering Department, Universidad de los Andes, Bogotá, Colombia, jaiescob@uniandes.edu.co

(P1-15) COLLOIDAL PROCESSING OF MAGNESIUM ALUMINATE SPINEL DENSE BODIES.

P. Pinho, A. B. Lopes, and M. M. Almeida.

Department of Ceramic and Glass Engineering, CICECO, University of Aveiro, 3810-193 Aveiro, Portugal, augusto@ua.pt

(P1-16) OPTIMIZATION OF CUPRATE BARIUM POWDER SLIP FOR TAPE CASTING.

H. Amaveda[1], M. Mora[1], A. Sotelo[1], C. Cardiel[1], L. A. Angurel[1], and R. Moreno[2].

[1] ICMA (CSIC-Universidad de Zaragoza), c/ María de Luna 3, 50018 Zaragoza, Spain, [2]Instituto de Cerámica y Vidrio, CSIC, c/ Kelsen, 5, 28049 Madrid, Spain, hippo@unizar.es

(P1-17) TAPE CASTING OF CLAY BASED COMPOSITIONS FOR TILES.

F. Rubio-Marcos[1], J. J. Reinosa[1], E. Solera[1], M.A. Bengochea[2], and J. F. Fernández[1].

[1] Electroceramic Department, Instituto de Cerámica y Vidrio, CSIC 28049 Madrid, Spain, [2]Keraben S.A. 12520 Nules Castellón, Spain, frubio@icv.csic.es

(P1-18) TWO ALTERNATIVE ROUTES FOR STARCH CONSOLIDATION OF MULLITE GREEN BODIES.

M. H. Talou and M. A. Camerucci.
Lab. de Materiales Estructurales - INTEMA, Fac. de Ingeniería - UNMdP - CONICET (7600) Mar del Plata, Argentina, mtalou@fi.mdp.edu.ar

(P1-19) UNIFORMED POWDER COMPACTS FABRICATED FROM AIR DRY FORMING METHOD USING CONDENSED SLURRIES WITH ADDITION OF GLYCEROL.

S. Tanaka, R. Furushima, and K. Uematsu.
Nagaoka University of Technology, 1603-1 Kamitomioka, Nagaoka Niigata 9402188 Japan, uematsu@vos.nagaokaut.ac.jp

(P1-20) PRODUCTION OF POROUS CERAMICS AND HOLLOW CAPSULES FROM PARTICLE-STABILIZED EMULSIONS.

E. Tervoort, I. Akartuna, A.R. Studart, and L. J. Gauckler.
ETH, Department of Materials, Wolfgang-Pauli-Strasse 10, CH-8093, Zurich, Switzerland, elena.tervoort@mat.ethz.ch

(P1-21) STUDY OF Al_2O_3-TiO_2 GRANULES OBTAINED BY FREEZEDRYING PROCESS.

M. Vicent[1], E. Sánchez[1], R. Moreno[2], and M. I. Nieto[2].
[1] Instituto de Tecnología Cerámica, Castellón, Spain, [2]Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain, monica.vicent@itc.uji.es

(P1-22) USE OF EXTRUSION TECHNOLOGY FOR OBTAINING OF DENSE TITANIUM OXIDE CERAMICS.

A. Pavlova, J. Locs, R. Neretnieks, and L.Berzina-Cimdina
Riga Technical University, Riga Biomaterials Innovation and Development Centre, Pulka Street 3/3, Riga, LV-1007, Latvia, agnese.pavlova@rtu.lv

(P1-23) CONTINUOUS EXTRUSION OF SUSPENSIONS OF NATURAL ZEOLITES.

G. Zacahua-Tlacuatl[1], J. Pérez-González[2], J. J. Castro-Arellano[1], and H. Balmori-Ramírez[1].
[1]Sección de Estudios de Posgrado, ESIQIE-IPN, Edif. 8, 3er piso, C. P. 07738, México D. F., Mexico, [2]Laboratorio de Reología, Escuela Superior de Física y Matemáticas, Instituto Politécnico Nacional, Apdo. Postal 118-209, C. P. 07051, México D. F., Mexico, hbalmori@ipn.mx

(P1-24) SYNTHESIS OF CERAMIC NANOPARTICLES BY LASER ABLATION IN LIQUIDS

M. Oujja[1], M. Sanz[1], M. Castillejo[1], G. Gómez[2], R. Moreno[2], and J.C. Fariñas[2]
[1]Instituto de Química Física Rocasolano, CSIC, 28006 Madrid, Spain, [2]Instituto de Cerámica y Vidrio, CSIC, 28049 Madrid, Spain, jcfarinas@icv.csic.es

17:00 - 17:15 h Coffee Break

17:15 - 19:00 h Student Contest (SC1)

Chair persons: A. Segadaes &. C. Baudín

(SC1-1) CHEMICAL STABILITY OF $CoAl_2O_4$ BLUE PIGMENT IN AQUEOUS SUSPENSIONS.
S. Akdemir, E. Ozel, and E. Suvacı.
Department of Materials Science and Engineering, Anadolu University, Eskisehir, Turkey, semakdemir@gmail.com

(SC1-2) MONOSACCHARIDES DERIVATIVES AS MONOMERS IN GELCASTING PROCESS.
P. Bednarek[1], M. Szafran[1], T. Mizerski[1], Y. Sakka[2].
[1] Warsaw University of Technology, Faculty of Chemistry, Warsaw, Poland, [2]Fine Particle Processing Group, Nano Ceramics Center, National Institute for Materials Science, Tsukuba, Ibaraki, Japan, bednarek@ch.pw.edu.pl

(SC1-3) WATER BASED SYNTHESIS AND DISPERSION OF $Ni(OH)_2$ NANOPARTICLES.
S. Cabanas-Polo, O. Burgos-Montes, and A. J. Sanchez-Herencia.
Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain, scabanas@icv.csic.es

(SC1-4) DEFLOCCULATION OF NANOZIRCONIA POWDERS BY MEANS OF MONOSACCHARIDES ADDITION.
A. Danelska, M. Szafran, and E. Bobryk.
Warsaw University of Technology, Faculty Of Chemistry, Warsaw, Poland, anna.danelska@gmail.com

(SC1-5) SPRAY DRYING OF TiO_2 NANOPARTICLES INTO REDISPERSIBLE GRANULES.
B. Faure[1], J. S. Lindeløv[2], M. Wahlberg[2], N. Adkins[3], P. Jackson[3], and L. Bergström[1].
[1] Materials Chemistry Research Group, Department of Physical, Inorganic and Structural Chemistry, Arrhenius Laboratory, Stockholm University, Stockholm, Sweden, [2]Niro A/S, Gea Process Engineering Division, Søborg, Denmark, [3]CERAM Research Ltd., Penkhull, Stoke-On-Trent, Staffordshire, United Kingdom, bertrand@inorg.su.se

(SC1-6) ELECTROCONDUCTED ASSEMBLY OF NANO-YSZ AND SCANDIA - DOPED YSZ PREPARED BY MILD - HYDROTHERMAL SYNTHESIS.
I. Gonzalo-Juan, M. T. Colomer, and B. Ferrari.
Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain, igonzalo@icv.csic.es

(SC1-7) POWDER CONDITIONING FOR THERMAL SPRAYING PROCESSES.
J. Guimarães, E. Garcia, P. Miranzo, and M. I. Osendi.
Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain, joana.queiroz@icv.csic.es

(SC1-8) HOLLOW SPHERICAL TiO_2-BASED PHOTOCATALYSTS PREPARED BY SPRAY PYROLYSIS.
B. Haugen[1], C. Simon[2], I. Kumakiri[2], and M. A. Einarsrud[1].
[1] Department of Materials Science and Engineering, Norwegian University of Science and Technology, Trondheim, Norway, [2]SINTEF Materials and Chemistry, Oslo, Norway, astribjo@stud.ntnu.no

(SC1-9) RHEOLOGICAL BEHAVIOUR OF ZIRCONIA AND TITANIA SUSPENSIONS TO SYNTHESIZE ZIRCONIUM TITANATE-BASED COMPOSITES.
E. López-López, C. Baudín, and R. Moreno.
Instituto de Cerámica y Vidrio, CSIC, C/ Kelsen 5, 28049, Madrid, Spain, emilioll@icv.csic.es

(SC1-10) WATER BASED PROCESSING OF NANO Y_2O_3 DISPERSED HYDROXYAPATITE COMPOSITES.
P. Parente[1], O. Burgos-Montes[2], M. A. Auger[1], M. A. Monge[1], and A. J. Sánchez-Herencia[2].
[1] Physic Department, Universidad Carlos III Madrid, Leganés, Madrid, Spain,
[2] Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain, pparente@fis.uc3m.es

(SC1-11) SLIP PREPARATION FOR BIOCERAMICS CONTAINING MACROPORES.
N. Pawlak[1], M. Kelleher[1], and S. Hampshire[2].
[1] School of Manufacturing and Design Engineering, Dublin Institute of Technology, Dublin, Ireland. [2]Materials and Surface Science Institute, University of Limerick, Limerick, Ireland, natalia.pawlak@student.dit.ie

(SC1-12) RHEOLOGY OF UV CURABLE COLLOIDAL SILICA DISPERSIONS FOR RAPID PROTOTYPING APPLICATIONS.
M. Wozniak[1,2], Y. Hazan[1] T. Graule[1], and D. Kata[2].
[1] EMPA, Swiss Federal Laboratories for Materials Testing and Research, Laboratory for High Performance Ceramics, Dübendorf, Switzerland, [2]University of Science and Technology, Department of Technology of Ceramics and Refractories, Krakow, Poland, wozniak@agh.edu.pl

TUESDAY 17th November

Session 3 (S3) Chair persons: J.P. Eraw &. I. Santacruz

08:30 - 09:00 h Invited Lecture (IL5)
POROUS CERAMICS FOR GAS AND BIOMOLECULE SEPARATION.
L. Bergström.
Materials Chemistry Research Group, Department of Physical, Inorganic and Structural Chemistry, Arrhenius Laboratory, Stockholm University, Sweden, lennartb@inorg.su.se

SHAPING-4 CONFERENCE. Final programme.

09:00 - 09:30 h Invited Lecture (IL6)
NOVEL PROCESSING AND FORMING OF BIOMATERIALS.
M. Edirisinghe.
Department of Mechanical Engineering, University College London, UK, m.edirisinghe@ucl.ac.uk

09:30 - 09:45 h (S3-1) AN X-RAY TOMOGRAPHY STUDY OF AGGLOMERATE BREAKDOWN DURING PASTE FLOW.
P. McGuire, S. Welch, K. Harrison, Y. O. Ayanlowo, S. Odukogbe, and S. Blackburn.
IRC in Materials Processing and School of Chemical Engineering, University of Birmingham, Edgbaston, Birmingham, UK, s.blackburn@bham.ac.uk

09:45 - 10:00 h (S3-2) SYNTHESIS, COLLOIDAL STABILITY, PHOTOCATALYTIC AND ANTIMICROBIAL PROPERTY OF Ag-DEPOSITED TiO_2 COMPOSITE NANOPARTICLES.
C. N. Chen, W. C. Lin, and W. J. Tseng.
Department of Materials Science and Engineering, National Chung Hsing University, Taichung, Taiwan, wenjea@dragon.nchu.edu.tw

10:00 - 10:15 h (S3-3) ELECTROPHORETIC DEPOSITION OF ADVANCED FUNCTIONAL CERAMICS INTO COMPLEX SHAPES AND CONFIGURATIONS.
J. Ma.
School of Materials Science and Engineering, Nanyang Technological University, Singapore, asjma@ntu.edu.sg

10:15 - 10:30 h (S3-4) CO-EXTRUSION OF MULTILAYERED CERAMIC MICRO-TUBES.
J. Powell[1] and S. Blackburn[2].
[1] Department of Metallurgy and Materials Science. [2]Department of Chemical Engineering, University of Birmingham, Edgbaston, Birmingham, UK, jdapowell@hotmail.com

10:30 - 10:45 h (S3-5) POROUS ALUMINA CERAMICS PREPARED WITH WHEAT FLOUR.
E. Gregorová, W. Pabst, and Z. Živcová.
Institute of Chemical Technology (ICT Prague), Prague, Czech Republic, pabstw@vscht.cz

10:45 - 11:00 h (S3-6) OSMOTIC DRYING OF GELCASTED BODIES PREPARED FROM FINE ALUMINA POWDER.
M. Trunec.
Department of Ceramics and Polymers, Brno University of Technology, Brno, Czech Republic, trunec@fme.vutbr.cz

11:00 - 11:30 h Coffee Break

SHAPING-4 CONFERENCE. Final programme.

Session 4 (S4) Chair persons: P. Colombo &. M.T. Colomer

11:30 - 12:00 h — Invited Lecture (IL7)
CAPSULES AND FOAMS FROM NANOPARTICLES.
L. J. Gauckler, I. Akartuna, E. Teervort, and U. Gonzenbach.
Inorganic Nonmetallic Materials, ETH Zurich; ,Department Materials Science, Zurich, Switzerland, ludwig.gauckler@mat.ethz.ch

12:00 - 12:15 h — (S4-1) PREPARATION AND PROPERTIES OF ULTRAHIGHLY POROUS SILICON CARBIDE.
M. Fukushima, Y. Zhou, and Y. Yoshizawa.
Advanced Manufacturing Research Institute, National Institute of Advanced Industrial Science and Technology (AIST), Moriyama-ku Nagoya, Japan, manabufukushima@aist.go.jp

12:15 - 12:30 h — (S4-2) ICE TEMPLATING :A VERSATILE PROCESS TO PRODUCE FUNCTIONALLY GRADED CERAMIC FILTER MEDIA.
C. Delmotte, M. Tabata, G. Bister, J.P. Erauw, and F. Cambier.
Belgian Ceramic Research Centre (BCRC), Mons, Belgium, c.delmotte@bcrc.be

12:30 - 12:45 h — (S4-3) POROUS ECOMATERIALS BASED ON INDUSTRIAL WASTE AND WOOD TO BUILDING MATERIALS.
E. Prud'homme, P. Michaud, and S. Rossignol.
Groupe d'Etude des Matériaux Hétérogènes (GEMH-ENSCI) Ecole Nationale Supérieure de Céramique Industrielle, Limoges, France, sylvie.rossignol@unilim.fr

12:45 - 13:00 h — (S4-4) POROUS POLYMER DERIVED CERAMIC COMPOSITES DECORATED WITH IN-SITU GROWN NANO-STRUCTURES.
C. Vakifahmetoglu[1], J. Woltersdorf[2], E. Pippel[2], and P.Colombo[1].
[1] University of Padova, Dipartimento di Ingegneria Meccanica - Settore Materiali, Padova, Italy. 2Department of Materials Science and Engineering, The Pennsylvania State University, PA, USA. 2Max Planck Institut für Mikrostrukturphysik, Halle, Germany, cekdar@unipd.it

13:00 - 13:15 h — (S4-5) POLYMER DERIVED CERAMICS FOR BEARING APPLICATIONS.
L. Schlier[1], M. Steinau[1], N. Travitzky[1], J. Gegner[2], and P. Greil[1].
[1] Department of Materials Science, Institute of Glass and Ceramics, Erlangen, Germany. [2]SKF GmbH, Schweinfurt, Germany, nahum.travitzky@ww.uni-erlangen.de

13:15 - 13:30 h — (S4-6) JOINING OF PRECERAMIC PAPERS FOR THE PRODUCTION OF FILTER SYSTEMS.
B. Gutbrod[1], N. Travitzky[1], C. Sorg[2], A. Hofenauer[2], and P. Greil[1].
[1] Department of Materials Science, Institute of Glass and Ceramics, Erlangen, Germany. [2]Paper Technology Specialists (PTS), Munich, Germany, nahum.travitzky@ww.uni-erlangen.de

13:30 - 15:00 h Lunch

SHAPING-4 CONFERENCE. Final programme.

Session 5 (S5) Chair persons: G.L. Messing &. A.J. Sánchez-Herencia

15:00 - 15:30 h — Invited Lecture (IL8)
POROUS CERAMIC STRUCTURES AS A TOOL FOR MANY APPLICATIONS.
J. Luyten, S. Mullens, F. Snijkers, M. Snel, and P. Nuyts.
Materials Technology, VITO, Mol, Belgium, jan.luyten@vito.be

15:30 - 15:45 h — (S5-1) TAPE CAST POROSITY-GRADED PIEZOELECTRIC CERAMICS.
E. Mercadelli, A. Sanson, P. Pinasco, E. Roncari, and C. Galassi.
Institute of Science and Technology for Ceramics, National Research Council, CNR-ISTEC, Faenza, Italy, elisa.mercadelli@istec.cnr.it

15:45 - 16:00 h — (S5-2) A NOVEL TEMPLATED GRAIN GROWTH APPROACH FOR THE PROCESSING OF 〈 001〉 -TEXTURED PMN-PT CERAMICS.
H. Amorín[1], J. Ricote[1], I. Santacruz[2,3], R. Moreno[2], J. Holc[4], M. Kosec[4], P. Ramos[5], D. Chateigner[6], and M. Algueró[1].
[1] Instituto de Ciencia de Materiales de Madrid, CSIC, Madrid, Spain. [2]Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain. [3]University of Málaga, Málaga, Spain. [4] Jozef Stefan Institute, Ljubljana, Slovenia. [5]Dpto. de Electrónica, Universidad de Alcalá, Alcalá de Henares, Spain. [6]Laboratoire de Cristallographie et Sciences de Matériaux, ENSICAEN, Caen, France, hamorin@icmm.csic.es

16:00 - 16:15 h — (S5-3) PROCESSING AND CHARACTERIZATION OF TEXTURED MULLITE CERAMICS FROM PHYLLOSILICATES.
S. Deniel[1], N. Tessier-Doyen[1], C. Dublanche-Tixier[2], D. Chateigener[3], and P. Blanchart[1].
[1] GEMH, ENSCI, Limoges, France, [2]ENSIL- SPCTS UMR CNRS, Limoges, France, [3] CRISMAT- ENSICAEN and IUT Caen, Université de Caen Basse Normandie, Caen, France, sarah.deniel@etu.unilim.fr

16:15 - 16:30 h — (S5-4) ELABORATION OF $La_{1-X}Sr_XFe_{1-Y}Ga_YO_{3-\Delta}$ MULTILAYER MEMBRANE WITH Sr SUBSTITUTION GRADIENT BY TAPE-CASTING AND CO-FIRING.
P.M. Geffroy[1], A. Vivet[1,2], A. Julian[1,2], E. Juste[1,2], V. Coudert[1], P. Del Gallo[2], N. Richet[2], and T. Chartier[1].
[1] CNRS-ENSCI, SPCTS, Limoges, France. [2]Air Liquide, Centre de Recherche Claude-Delorme, Les Loges-en-Josas, Jouy-en-Josas, France, pierre-marie.geffroy@unilim.fr

16:30 - 16:45 h — (S5-5) PROCESSING ROUTE TO GENERATE AND DIRECTLY TAPE CAST NANO-SIZED α-Al_2O_3 POWDERS.
P. Vozdecky[1], A. Roosen[1], C. Knieke[2], and W. Peukert[2].
[1] University of Erlangen-Nuremberg, Department of Materials Science, Institute of Glass and Ceramics, Erlangen, Germany. [2]University of Erlangen-Nuremberg, Department of Chemical and Biological Engineer-ing, Institute of Particle Technology, Erlangen, Germany, pavel.vozdecky@ww.uni-erlangen.de

16:45 - 17:00 h (S5-6) A MULTI-SCALE SIMULATION MODEL FOR TAPE CASTING.
A. Wonisch, T. Kraft, M. Moseler, and H. Riedel.
Fraunhofer Institute for Mechanics of Materials, Freiburg, Germany,
andreas.wonisch@iwm.fraunhofer.de

17:00 - 17:15 h Coffee Break

17:15 - 19:00 h Student Contest (SC2)

Chair persons: C. Galassi &. C. Baudín

(SC2-1) SHAPING AND DENSIFICATION OF β-TRICALCIUM PHOSPHATE BIOCERAMICS.
E. Constantin-Rguiti[1,2,3], J-C. Hornez[1,2], M. Poorteman[4], J. Lu[3], F. Cambier[4], M. Descamps[1,2], and A. Leriche[1,2].
[1]Université Lille Nord de France, Lille, France. [2]Laboratoire des Matériaux et Procédés, Université Lille Nord de France, Maubeuge, France. [3]Biocétis SARL, Cournonsec, France. [4]Belgian Ceramic Research Centre, Mons, Belgium,
emmanuelle.rguiti@univ-valenciennes.fr

(SC2-2) PROCESSING OF CARBON NANOTUBES CONTAINING SILICON NITRIDE NANOCOMPOSITES.
J. González-Julián, P. Miranzo, M.I. Osendi, and M. Belmonte.
Institute of Ceramics and Glass, CSIC, Madrid, SPAIN, jgonzalez@icv.csic.es

(SC2-3) BINDER DISTRIBUTION DURING WICK-DEBINDING OF CERAMIC PARTS PREPEARED BY LPIM.
L. Gorjan, A. Dakskobler, and T. Kosmač.
Institut Jožef Stefan, Ljubljana, Slovenia, lovro.gorjan@gmail.com

(SC2-4) OPEN-CELL CERAMIC FOAM STRUCTURES PRODUCED BY DIRECT FREEZE FOAMING.
A. Müller and T. Moritz.
Fraunhofer Institute for Ceramic Technologies and Systems, Dresden, Germany,
Axel.Mueller@ikts.fraunhofer.de

(SC2-5) ELECTROPHORETIC SHAPING WITH COAXIAL ELECTRODES.
A. Nold and R. Clasen.
Saarland University, Saarbruecken, Germany, a.nold@nanotech.uni-saarland.de

(SC2-6) INCORPORATION OF SEPIOLITE FIBER CONTAINING t-ZIRCONIA NANOPARTICLES TO A CERAMIC GLAZE.
R. Pina-Zapardiel[1], A. Esteban-Cubillo[2], J. F. Bartolomé[1], C. Pecharromán[1], and J.S.Moya[1].
[1] Instituto de Ciencia de Materiales de Madrid, CSIC, Madrid, Spain. [2]Tolsa S.A.,
raul.pina@icmm.cisc.es

(SC2-7) PROGRESS IN THE ELECTROPHORETIC DEPOSITION (EPD) OF BIOACTIVE GLASS AND BIOACTIVE GLASS-BIOPOLYMER COMPOSITE COATINGS.
F. Pishbin, A. Simchi, M. P. Ryan, and A. R. Boccaccini.
Department of Materials, Imperial College London, London, UK, fatemehsadat.pishbin07@imperial.ac.uk

(SC2-8) EFFECT OF SHAPING TECHNIQUE ON SINTERED DENSITIES OF SILICON CARBIDE.
K. Rade, S. Novak, and S. Kobe.
Jožef Stefan Institute, Ljubljana, Slovenia, katja.rade@ijs.si

(SC2-9) PROCESSING AND TAPE CASTING FROM COLLOIDAL SUSPENSIONS OF DOPED LANTHANUM CHROMITE SYNTHESIZED BY COMBUSTION SYNTHESIS.
L.F.G. Setz[1], S.R.H. Mello-Castanho[1], I. Santacruz[2,3], M.T. Colomer[2], and R. Moreno[2].
[1]Instituto de Pesquisas Energéticas e Nucleares - IPEN/CNEN - Brasil. [2]Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain, [3]University of Málaga, Málaga, Spain, lfsetz@yahoo.com.br

(SC2-10) NOVEL STRATEGIES TO PRODUCE HIGH SPECIFIC SURFACE AREA CERAMIC FOAMS FROM PRECERAMIC POLYMERS.
C. Vakifahmetoglu and P. Colombo.
Dipartimento di Ingegneria Meccanica - Settore Materiali, Università di Padova, Padova, Italy, cekdar@unipd.it

(SC2-11) ZnO-BASED THIN FILMS BY EPD.
M. Verde, M. Peiteado, A. C. Caballero, M. Villegas, and B. Ferrari.
Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain, mverde@cv.csic.es

21:00 - 23:30 Conference Dinner

Restaurante El Espejo.
Paseo de Recoletos, 31, 28004 Madrid
www.restauranteelespejo.com

SHAPING-4 CONFERENCE. Final programme.

WEDNESDAY 18th November

Session 6 (S6) Chair persons: K. Uematsu &. E. Sánchez-Vilches

08:30 - 09:00 h — Invited Lecture (IL9)
COMPLEX SHAPE FORMING USING CROSSLINKABLE POLY-VINYL ALCOHOL (PVA).
G. V. Franks.
Chemical and Biomolecular Engineering, University of Melbourne, Australia, gvfranks@unimelb.edu.au

09:00 - 09:30 h — Invited Lecture (IL10)
MICROSTRUCTURAL REQUIREMENTS AND IN SITU PROCESSING FOR ALUMINA MATRIX NANOCOMPOSITES.
R. I. Todd and A. Mukhopadhyay.
University of Oxford, Department of Materials, Parks Road, Oxford OX1 3PH, UK, richard.todd@materials.ox.ac.uk

09:30 - 09:45 h — (S6-1) TEMPLATED GRAIN GROWTH AND PROPERTIES OF BIOINSPIRED CERAMIC MICROSTRUCTURE COMPOSITES.
R.J. Pavlacka and G.L. Messing.
Pennsylvania State University, University Park, PA USA, messing@matse.psu.edu

09:45 - 10:00 h — (S6-2) PREPARATION OF HIGH SOLIDS CONTENT NANOTITANIA SUSPENSIONS FOR ATMOSPHERIC PLASMA SPRAYING.
M. Vicent[1], E. Sánchez[1], R. Moreno[2], I. Santacruz[2], M. D. Salvador[3], and V. Bonache[3].
[1]Instituto de Tecnología Cerámica (ITC) - Asociación de Investigación de las Industrias Cerámicas (AICE). Universitat Jaume I. Castellón, Spain. [2]Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain. [3]Instituto de Tecnología de Materiales, Universidad Politécnica de Valencia, Valencia, Spain, monica.vicent@itc.uji.es

10:00 - 10:15 h — (S6-3) MULLITE COATINGS ON CERAMIC SUBSTRATES: STABILISATION OF Al_2O_3-SiO_2 SUSPENSIONS FOR SPRAY DRYING OF COMPOSITE GRANULES SUITABLE FOR REACTIVE PLASMA SPRAY.
A. Schrijnemakers[1], S. André[2], G. Lumay[3], N. Vandewalle[3], F. Boschini[1], R. Cloots[1], and B. Vertruyen[1].
[1] LCIS/CMI, Chemistry Institute B6, University of Liège, Liège Belgium. [2]BCRC, Belgian Ceramic Research Center, Mons Belgium. [3]APTIS/GRASP, Physic Institute B5, University of Liège, Liège Belgium, a.schrijnemakers@student.ulg.ac.be

10:15 - 10:30 h — (S6-4) MEASUREMENT OF BULK DENSITY DISTRIBUTION IN LARGE CERAMIC TILES BY A NON-DESTRUCTIVE METHOD.
J. L. Amorós, J. Boix, D. Llorens, G. Mallol, I. Fuentes, and C. Feliu.
Instituto de Tecnología Cerámica. Asociación de Investigación de las Industrias Cerámicas. Universitat Jaume I. Campus Universitario Riu Sec. Castellón (Spain), jboix@itc.uji.es

10:30 - 10:45 h (S6-5) DESIGNING PARTICLE SIZING AND PACKING FOR FLOWABILITY AND SINTERED MECHANICAL STRENGTH.
A. P. Silva[1], D. G. Pinto[1], A. M. Segadães[2], and T.C. Devezas[1].
[1] Dept. Electromechanical Eng., University of Beira Interior, Covilhã, Portugal. [2]Dept. Ceramics and Glass Eng. (CICECO), University of Aveiro, Aveiro, Portugal, abilio@ubi.pt

10:45 - 11:00 h (S6-6) STUDY OF SHAPING CONDITIONS OF ALUMINOSILICATES BASED SLABS.
L. Esposito and A. Salomoni.
Centro Ceramico Bologna, Bologna, Italy, salomoni@cencerbo.it

11:00 - 11:30 h Coffee Break

Session 7 (S7) Chair persons: J. Heinrich &. A.C. Caballero

11:30 - 12:00 h Invited Lecture (IL11)
FABRICATION OF HIGHLY STRUCTURE CONTROLLED CERAMICS THROUGH ADVANCED COLLOIDAL PROCESSING.
Y. Sakka, T. S. Suzuki, and T. Uchikoshi.
Nano Ceramics Center, National Institute for Materials Science, Tsukuba, Ibaraki, Japan, SAKKA.Yoshio@nims.go.jp

12:00 - 12:15 h (S7-1) PRESSURELESS SINTERING OF Ti_3SiC_2 POWDER.
B. B. Panigrahi[1], J. J. Gracio[1], M. Chu[2], and S. Cho[2].
[1] Center for Mechanical Technology and Automation, Department of Mechanical Engineering, University of Aveiro, Aveiro, Portugal, [2]Division of Advanced Technology, Korea Research Institute of Standards and Science, Yuseong, Daejeon, Republic of Korea, panigrahi14@yahoo.com

12:15 - 12:30 h (S7-2) SPARK PLASMA SINTERING OF Si-BASED CERAMICS.
M. Belmonte, J. Gonzalez-Julian, P. Miranzo, and M. I. Osendi.
Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain, mbelmonte@icv.csic.es

12:30 - 12:45 h (S7-3) POROUS MULLITE MATERIALS WITH VERY LOW THERMAL CONDUCTIVITY.
P. Miranzo, E. Garcia, and M.I. Osendi.
Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain, miosendi@icv.csic.es

12:45 - 13:00 h (S7-4) PRODUCTION OF β-SiAlON CERAMICS WITH LOW AMOUNT OF ADDITIVE AT LOW SINTERING TEMPERATURE.
O. Eser[1], S. Kurama[1], and G. Gunkaya[2].
[1] Graduate School of Sciences, Department of Advanced Technologies, Anadolu University, Eskisehir, Turkey. [2]Department of Materials Science and Engineering, Anadolu University, Eskisehir, Turkey, skurama@anadolu.edu.tr

13:00 - 13:15 h (S7-5) SINTERING OF POWDERS IN THE $CrSi_2$-Ti (Ta)Si_2 SYSTEMS DEPENDING ON THE METHODS OF SYNTHESIS.

I. Uvarova, I.Kud', L. Yeremenko, L. Lykhodid, and D. Ziatkevich
I.Frantsevych Institute for Problems of Materials Science of NAS, Kyiv, 03142 Ukraine, uvarova@ipms.kiev.ua

13:15 - 13:30 h (S7-6) THE FABRICATION OF POROUS CERAMIC ELECTRODES FOR APPLICATIONS IN ELECTROCHEMISTRY.

E. Chavez, L. Jones, J. A. Diez, J. Etxeberria.
CIDETEC -Centre for electrochemical technologies, Parque tecnológico de San Sebastián, Spain; CEIT, San Sebastián, Spain, echavez@cidetec.es

13:30 - 15:00 h Lunch

Session 8 (S8) Chair persons: A. Boccaccini &. B. Ferrari

15:00 - 15:30 h Invited Lecture (IL12)
POWDER PROCESSING WITH LASERS AS ENERGY SOURCE.

J. Günster[1], C. Oelgardt[2], X. Tian[2], and J. G. Heinrich[2].
[1] CIC Ceramic Institute Clausthal GmbH, Clausthal-Zellerfeld, Germany, [2]Clausthal University of Technology, Clausthal-Zellerfeld, Germany,
jens.guenster@oerlikon.com

15:30 - 15:45 h (S8-1) PROGRESS IN THE ELECTROPHORETIC DEPOSITION OF CARBON NANOTUBES (CNT) AND CNT/ NANOPARTICLES COMPOSITES.

A. R. Boccaccini.
Department of Materials, Imperial College London, London, UK and Institute of Biomaterials, University of Erlangen-Nuremberg, Erlangen, Germany,
a.boccaccini@imperial.ac.uk

15:45 - 16:00 h (S8-2) ELECTROPHORETIC DEPOSITION OF STRUCTURED COATINGS.

R. Clasen.
Saarland University, Campus C6 3, D-66123 Saarbruecken, Germany,
r.clasen@nanotech.uni-saarland.de

16:00 - 16:15 h (S8-3) ELECTROPHORETIC DEPOSITION OF SIALON PHOSPHOR PARTICLES FOR PACKAGING OF FLAT PSEUDO-WHITE LIGHT EMITTING DEVICES.

T. Uchikoshi[1], T. Kitabatake[1,2] F. Munakata[2], Y. Sakka[1], and N. Hirosaki[1].
[1] Nano Ceramics Center, National Institute for Materials Science, Tsukuba, Ibaraki, Japan. [2]Department of Energy Science and Nuclear Engineering, Tokyo City University. Tokyo, Japan, uchikoshi.tetsuo@nims.go.jp

16:15 - 16:30 h (S8-4) ELECTRIC FIELD ASSISTED FORMING OF CNT-SiC_f/SiC COMPOSITE.
S. Novak[1], K. König[1], A. Ivekovič[1], and A.R. Boccaccini[2].
[1]Department for Nanostructured Materials, Jožef Stefan Institute, JLjubljana, Slovenia. [2]Department for Materials, Imperial College, London, UK, sasa.novak@ijs.si

16:30 - 16:45 h (S8-5) ELECTROSTATIC AND KINETIC ASPECTS OF ELECTROPHORETIC DEPOSITION OF CERAMIC MATERIALS.
C. Baldisserri, D. Gardini, and C. Galassi.
ISTEC - CNR, via Granarolo 64, 48018 Faenza (RA), Italy, carlo.baldisserri@istec.cnr.it

16:45 - 17:00 h (S8-6) LEAD MAGNESIUM NIOBATE-LEAD TITANATE THICK FILMS PREPARED BY ELECTROPHORETIC DEPOSITION
D. Kuscer and M. Kosec.
Jožef Stefan Institute, Jamova 39, SI-1000 Ljubljana, Slovenia, danjela@ijs.si

17:00 - 17:15 h (S8-7) ELECTRICALLY DRIVEN ARRANGEMENT OF SILICA MESO-POROUS COATINGS.
Y. Castro, M. Servera, A. Duran, and B. Ferrari.
Instituto de Cerámica y Vidrio, CSIC, Madrid, Spain, castro@icv.csic.es

17:15 - 18:45 h Poster Session-2 (P2)

(P2-1) MINERALOGICAL AND IONIC CONDUCTIVITY STUDY OF BENTONITE.
M. Ayadi[1] and N. K. Ariguib[2].
[1]Faculté des Sciences de Bizerte, 7021 Zarzouna Bizerte, Tunisia, [2]Laboratory of Materials, Centre de Recherches et des Technologies de l'Energie, B.P. 95, 2050 Hammam-Lif, Tunisia, mounirayadi@yahoo.fr

(P2-2) SELECTIVE LASER HEAT TREATMENTS FOR REALIZING COATINGS AND THIN ELECTRIC COMPONENTS - PART 1: SILVER CONDUCTOR FABRICATION ON GLASS AND ALUMINA SUBSTRATES.
F. Petit[1], C. Ott[1], F. Cambier[1], N. Basile[2], and M. Gonon[2].
[1]Belgium Ceramic Research Centre, 4, Avenue du Gouverneur Cornez, B- 7000 Mons, [2]Faculté Polytechnique de Mons, Service de Science des Matériaux, 56, Rue de l'Epargne, B-7000 Mons, f.petit@bcrc.be, c.ott@bcrc.be

(P2-3) SELECTIVE LASER HEAT TREATMENTS FOR REALIZING COATINGS AND THIN ELECTRIC COMPONENTS - PART 2: SINTERING OF $BATiO_3$ BY SLS (SELECTIVE LASER SINTERING).
N. Basile[1], M. Gonon[1], C. Ott[2], F. Petit[2], and F. Cambier[2].
[1]Faculté Polytechnique de Mons, Service de Science des Matériaux, 56, Rue de l'Epargne, B-7000 Mons, [2]Belgium Ceramic Research Centre, 4, Avenue du Gouverneur Cornez, B- 7000 Mons, maurice.gonon@umons.ac.be, natanael.basile@umons.ac.be

(P2-4) SELECTIVE LASER HEAT TREATMENTS FOR REALIZING COATINGS AND THIN ELECTRIC COMPONENTS - PART 3: MELTING AND CRYSTALLIZATION OF A PIEZOELECTRIC GLASS CERAMIC.

N. Basile[1], M. Gonon[1], C. Ott[2], F. Petit[2], and F. Cambier[2].

[1] Faculté Polytechnique de Mons, Service de Science des Matériaux, 56, Rue de l'Epargne, B-7000 Mons, [2]Belgium Ceramic Research Centre, 4, Avenue du Gouverneur Cornez, B- 7000 Mons, maurice.gonon@umons.ac.be, natanael.basile@umons.ac.be

(P2-5) STABILIZATION OF AQUEOUS $BaCO_3$ SLURRIES AND COATING ON DENSE Y-TPZ SUBSTRATES FOR MAKING $BaZrO_3$ LAYERS BY THERMAL TREATMENT.

F. Boschini[1], A. Rulmont[1], B. Vertruyen[1], R. Cloots[1], and R. Moreno[2].

[1] Laboratory of Structural Inorganic Chemistry, Department of Chemistry -University of Liège, Allée de la chimie, 3-B6a, B-4000 Liège, Belgium, [2]Instituto de Cerámica y Vidrio, CSIC, c/ Kelsen, 5, Campus de Cantoblanco, E-28049 Madrid, Spain, frederic.boschini@ulg.ac.be

(P2-6) UTILIZATION OF NIGERIAN SANDS FOR GLASS CERAMIC COATINGS.

P. Chukwu[1], M. Muntean[2], and O. Dumitrescu[2].

[1] Anambra State University, Department of Pure and Industrial Chemistry P.O.Box 2 ,Uli. Anambra State, Nigeria, [2]Politehnical University of Bucharest, Department of Material Oxide 1 - 3 Polizu Bucharest, Romania, phimmainvestment@yahoo.co.uk

(P2-7) PREPARATION OF $YBa_2Cu_3O_{7-x}$ SUPERCONDUCTING THICK FILMS ON METALLIC SUBSTRATES BY THE ELECTROPHORETIC DEPOSITION (EPD) TECHNIQUE.

R. Closset[1,2], F. Boschini[1], B. Vertruyen[1], M. Dirickx[2], and R. Cloots[1].

[1] Laboratory of Structural Inorganic Chemistry, Department of Chemistry, University of Liège, Sart-Tilman, B-4000 Liège, Belgium, [2] Royal Military Academy, CISS department, Brussels, Belgium, Raphael.Closset@ulg.ac.be

(P2-8) MULTILAYER COATINGS OBTAINED BY COMBINATION OF PVD, CVD AND DIP-COATING TECHNIQUES.

A. Díaz-Parralejo[1], J. Sánchez-González[1], M. A. Díaz-Díez[1], A. Macías-García[1], and E. M. Cuerda-Correa[2].

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[2] Dpto. Química Inorgánica. Univ. de Extremadura. Spain, adiazpar@unex.es

(P2-9) ZIRCONIA COATINGS PREPARED BY EPD FROM SOL-GEL SOLUTIONS.

A. Díaz-Parralejo[1], J. Sánchez-González[1], J. L. Pantoja-Pertegal[1], J. M. González-Moreno[1], and E. M. Cuerda-Correa[2].

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(P2-10) MANUFACTURING OF (THICK) Ni-YSZ ANODE AND (THIN) YSZ ELECTROLYTE FOR TUBULAR SOFC BY POWDER THERMOPLASTIC EXTRUSION MOULDING.

T. Jardiel, B. Arias, G. Matula, B. Levenfeld, and A. Várez
Departamento de Ciencia e Ingeniería de Materiales. Universidad Carlos III de Madrid. Avda. de la Universidad 30, 28911, Leganés, Spain, jardiel@icv.csic.es, alvar@ing.uc3m.es

(P2-11) MULTIPLE PROFILES PROTECTIVE COATINGS FOR PIPELINES AND VESSELS.

Z. Kovziridze and I. Berdzenishvili.
Georgian Technical University, 77 st. Kostava, Tbilisi, Georgia, kowsiri@gtu.ge

(P2-12) ANODE-SUPPORTED SOLID OXIDE FUEL CELLS (SOFCs) BASED ON THIN FILMS OF DOPED CERIA ELECTROLYTES.

M. Morales[1,2], M. Segarra[2], and S. Piñol[1].
[1] Institut de Ciència de Materials de Barcelona (CSIC), Campus de la UAB, Bellaterra E-08193, Barcelona, Spain, [2]Departament de Ciencia de Materials i Enginyeria Metal.lùrgica, Facultat de Química. Universitat de Barcelona, Diagonal 647, E-08028, Barcelona, Spain., salva@icmab.es

(P2-13) SOLID OXYDE ELECTROLYTE TUBULAR CELLS FOR HYDROGEN PRODUCTION.

T. Piquero, B. Vergne, J. Vulliet, K. Wittmann-Teneze, N. Caron, and F. Blein.
CEA / Le Ripault, BP16, 37260 Monts, France, franck.blein@cea.fr

(P2-14) CORROSION RESISTANCE OF IRON POWDERS CLAD WITH PHOSPHORUS IN INORGANIC AND BIOMEDIA OF HUMAN ORGANISM.

N. Boshytska, L. Apininska, L. Protsenko, O. Budilina, and I. V. Uvarova.
I.Frantsevych Institute for Problems of Materials Science of NAS, Kyiv, 03142 Ukraine, uvarova@ipms.kiev.ua

(P2-15) THERMAL BEHAVIOUR OF KAOLINITE POWDERS: MULTI-STEP DEHYDROXYLATION AND HIGH-TEMPERATURE PHASES.

F. J. Gotor, M. Macías, A. Ortega, and P. J. Sánchez-Soto
Instituto de Ciencia de Materiales de Sevilla, Centro Mixto Consejo Superior de Investigaciones Científicas (CSIC)-Universidad de Sevilla (US), c/Américo Vespucio 49, 41092-Isla de la Cartuja, Sevilla, Spain, pedroji@icmse.csic.es

(P1-16) POWDER PROCESSING OF LAYER SILICATES BY DRY GRINDING: A BIDIMENSIONAL PARTICLE SIZE MODEL

P. J. Sánchez-Soto[1]*, M. Raigón[2], E. Garzón[3], I. G. García-Rodríguez[3] and A. Ruiz-Conde[1]
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(P2-17) MICROSTRUCTURE DESIGN BY MECHANICAL ALLOYING.
T. A. G. Restivo and S. R. H. Mello-Castanho.
Nuclear and Energetic Research Institute - IPEN -Av. Lineu Prestes 2242 - Cidade Universitária - 05508000 - São Paulo - SP Brazil, guisard@dglnet.com.br

(P2-18) MULTIFUNCTIONAL HETERO-MODULE COMPOSITE IN B_4C-BN-TiC-SiC-C SYSTEM.
Z. Kovziridze, N. Nizharadze, G. Tabatadze, Z. Mestvirishvili, and V. Kinkladze, and E. Nikoleishvili.
Georgian Technical University, 77 st. Kostava, Tbilisi, Georgia, kowsiri@gtu.ge

(P2-19) SHAPING AND SURFACE MODIFICATION OF METAL PARTICULATED CERAMIC-Nb POWDER COMPOSITE.
J. F. Bartolomé, C. F. Gutierrez, F. J. Palomares, and J.S. Moya.
Instituto de Ciencia de Materiales de Madrid, CSIC, Cantoblanco Madrid 28049, Spain, jbartolo@icmm.csic.es

(P2-20) POROUS CERAMICS IN COMBUSTION APPLICATIONS.
P. Miranzo[1], M. A. Sainz[1], M. I. Osendi[1], R. Marín[2], and J. Fernández[3].
[1] Institute of Ceramics and Glass (ICV, CSIC), Kelsen, 5; 28049 Madrid. Spain,,
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(P2-21) ZrO_2 FOAMS FOR HEAT RECUPERATIVES IN GAS BURNERS.
A. C. Silva, S. C. Santos , L. F. G. Setz, and S. R. H. Mello-Castanho.
Nuclear and Energy Research Institute - IPEN/São Paulo, Brazil, silascs@ipen.br, dasilva.ac@uol.com.br, lfgsetz@ipen.br, srmello@ipen.br

(P2-22) INFLUENCE OF Y_2O_3 ADDITION TO Mg-PSZ CERAMICS ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES.
C. Yamagata, S. R. H. Mello-Castanho, and J. O. A. Paschoal.
Instituto de Pesquisas Energéticas e Nucleares - Av. Professor Lineu Prestes, 2242, Cidade Universitária, São Paulo, 05508-000, SP, Brazil, yamagata@net.ipen.br

(P2-23) COMPACTION AND SINTERING PROPERTIES OF STONEWARE BASED TILES.
J. J. Reinosa[1], F. Rubio-Marcos[1], I Lorite[1], M. A. Bengochea[2], and J. F. Fernández[1].
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(P2-24) PROCESSING AND PROPERTIES OF TUBULAR ASYMMETRIC MIXED CONDUCTING MEMBRANES
M. L. Fontaine, P. I. Dahl, O. Paulsen, Y. Larring, and R. Bredesen.
SINTEF Materials and Chemistry, PO Box 124 Blindern, NO 314 Oslo Norway, Marie-Laure.Fontaine@sintef.no

18:45 - 20:15 h Closure and Cocktail

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