



4th International Conference and Expo on

Ceramics and Composite Materials

May 14-15, 2018 | Rome, Italy

Scientific Tracks & Abstracts Day 1

Ceramics 2018

Composite Materials | Advanced Ceramic Materials

Session Chair

David G Calatayud

Instituto de Ceramica y Vidrio – CSI, Spain

Session Introduction

Title: Effect of the homogenization treatment on the microstructure and the electrical conductivity of 3YTZP/graphene nanoplatelet composites

Rosalía Poyato, CSIC-Universidad de Sevilla, Spain

Title: Performance of granite dust as filler in ordinary portland cement

Atiemo E, CSIR-Building and Road Research Institute, Ghana

Title: Development of lead-free perovskite ceramics with tunable optical and magnetic properties at room temperature

Santiranján Shannigrahi, Institute of Materials Research and Engineering, Singapore

YRF: Role of sintering method on graphene/3YTZP composites

Cristina López-Pernía, Universidad de Sevilla, Spain

YRF: Process development for the ceramic injection molding of oxide short fiber reinforced CMCs

Hasan Metin Tülümen, Karlsruhe Institute of Technology, Germany

Title: Composite materials from clays and clay mineral: Synthesis, characterization and electroanalytical applications

Tonle Kenfack Ignas, University of Dschang, Cameroon

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Effect of the homogenization treatment on the microstructure and the electrical conductivity of 3YTZP/graphene nanoplatelet composites

R. Poyato¹, C. López-Pernía^{1,2}, C. Muñoz-Ferreiro², C. González-Orellana, A. Morales-Rodríguez² and A. Gallardo-López²¹Instituto de Ciencia de Materiales de Sevilla, ICMS, CSIC-Universidad de Sevilla, Spain²Departamento de Física de la Materia Condensada, Universidad de Sevilla-ICMS (CSIC), Spain

Nowadays, graphene nanoplatelets (GNPs) are being considered as an emerging class of nanomaterials. GNPs are composed of ten or more graphene layers, with thickness up to 100 nm, and present unusual mechanical and electrical properties^{1,2}. These characteristics have motivated a great interest in incorporating these nanostructures as second phase in a ceramic matrix, in order to enhance the mechanical and functional properties of the final composite³⁻⁵. However, one of the main obstacles to be overcome is obtaining a good dispersion of the GNPs into the ceramic matrix.

In this work, 3YTZP based composites with different GNP contents were prepared using different powder homogenization methods and electric pulsed discharge sintering (also called spark Plasma Sintering or SPS). Composite powders were prepared by tip-sonication in isopropanol and/or by planetary ball milling under dry or wet conditions. The effect of milling and ultrasonic agitation on GNP integrity and particle size has been evaluated by Raman spectroscopy and laser granulometry. Microstructure of the composites has been analysed by electron microscopy, X-ray diffraction and Raman spectroscopy to assess the integrity of the GNPs, their degree of dispersion or agglomeration in the ceramic matrix and the stabilization of the tetragonal phase in the 3YTZP matrix. The relationships between GNP content and dimensions, microstructure and electrical conductivity, as well as the conduction mechanisms of the composites, have been analysed and discussed.

Recent Publications

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2. Kaiser AB, Gómez-Navarro C, Sundaram RS, Burghard M, Kern K (2009) Electrical conduction mechanism in chemically derived grapheme monolayers. *Nano Letters* 9:1787-1792.
3. Ramirez C, Miranzo P, Belmonte M, Osendi MI, Poza P, Vega-Diaz SM, Terrones M (2014) Extraordinary toughening enhancement and flexural strength in Si₃N₄ composites using graphene sheets. *Journal of the European Ceramic Society* 34:161-169.
4. Román-Manso B, Domingues E, Figueiredo FM, Belmonte M, Miranzo P (2015) Enhanced electrical conductivity of silicon carbide ceramics by addition of graphene nanoplatelets. *Journal of the European Ceramic Society* 35: 2723-2731.
5. Gallardo-López A, Márquez-Abril I, Morales-Rodríguez A, Muñoz A, Poyato R (2017) Dense graphene nanoplatelet/yttria tetragonal zirconia composites: Processing, hardness and electrical conductivity. *Ceramics International* 43: 11743-11752.

Biography

Dr. Rosalía Poyato completed her B.Sc. in Physics at the University of Seville and received her PhD in Applied Physics at the Autònoma University of Madrid. After obtaining PhD, she developed her research at University of Connecticut and Ohio State University (USA), as a postdoctoral research Fulbright fellow. At this moment, she is a Tenured Researcher at the Materials Science Institute of Seville and her current research interests include processing and characterization of ceramic composites including carbon nanostructures (carbon nanotubes and multi-layered graphene structures). She has co-authored over 50 papers in international journals. Dr. Poyato is now co-leader of a research project funded by the Spanish National Science Foundation and the European Feder Funding: Processing and microstructural, mechanical and electrical characterization of ceramic-graphene composites (MAT2015-67889-P).

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Performance of granite dust as filler in ordinary portland cement

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Granite dust, a waste from stone crushing accounts for 25% of the final product from stone quarrying in Ghana. In the absence of alternative uses, granite dust which is released directly into the environment has a high tendency of causing environmental pollution. To reduce the impact of the quarry dust on environment and humans, granite dust could be put to alternative uses such as admixture in the production of Portland cement. In this work the physical, chemical, mineralogical, mechanical and durability properties of Portland cement containing varying quantities of granite dust are evaluated. The experimental results indicate that the chemical compositions of all the cement samples are within acceptable limits according to EN 197-1. The raw granite dust also passed the standard chemical composition requirements for pozzolans prescribed by ASTM C 618. Water demand, setting times and soundness of the blended cements containing 5-25% of granite dust were all within acceptable limits per EN 197-1. Compressive strength results indicate that granite dust could be used to replace ordinary Portland cement up to an optimum of 15% for class 32.5 cement. Durability studies after 90 days proved that even though strength decreased as granite dust content increased, the cement samples containing granite dust are stable and can withstand aggressive environments (in terms of percentage strength reduction) comparable to Class 42.5N cement.

Key words: Granite dust, Ordinary Portland cement, Pozzolans, Soundness, setting times, Mineralogical, Durability.

Biography

Dr. Eugene Atiemo is a Chief Research Scientist and Director of CSIR-Building and Road Research Institute, Ghana. He holds a Ph.D in Civil Engineering and M.Phil in chemical Engineering from Kwame Nkrumah University of Science and Technology, Ghana. He is a member of Ghana Institution of Engineers.

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Development of lead-free perovskite ceramics with tunable optical and magnetic properties at room temperature

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In recent days, there have been extensive interests in developing transparent lead free ceramics as the alternatives to toxic lead-based ceramics. Transparent ceramics are fundamental to applications in lasers, sensors, IR optics, and transducers. In this work, La doped sodium potassium niobate based perovskite (ABO_3) ceramics, more specifically $[(1-x)(\text{K}_{0.5}\text{Na}_{0.5})\text{xLa}]\text{Nb}_{(1-2x/5)}\text{O}_3$ ($0 < x < 0.1$) have been developed. Among these, the material at 5 mol% La doping appears with an unusually high density up to 99% of its theoretical value, while densities of the parent $\text{K}_{0.5}\text{Na}_{0.5}\text{NbO}_3$ (KNN) ceramics typically only reach ~ 70 to 89% of the theoretical limit. The obtained ceramics display distinctive properties. The developed ceramics in their circular disk form with thickness 0.8 mm show certain transparency and most importantly, this transparency is electrically tunable up to 52%, which is quite remarkable and makes it suitable for optical device applications such as optical filters, sensors and smart windows. Moreover, under UV illumination, the ceramics display a photo-darkening which is reported for the first time for KNN based ceramics. The photodarkening phenomenon is reversible and is time-temperature-transformation phenomena. Additionally, the material shows extraordinary sensitivity of its magnetic and optical properties to UV irradiation. These materials transform to a metastable and reversible state that exhibits graded color change from clear to deep blue and shows considerable increase of magnetization under UV exposure. This feature makes the developed ceramics attractive for the fabrication of new generation devices; e.g. powerless UV detectors as well as protectors, since it absorbs UV completely. Based on first-principles calculations, we developed a model that attributes these observations to the occupancy of La fxyz orbitals induced by UV excitations. The model also indicates that UV induced absorption in the visible range and magnetism are related.

Biography

Santiranjana Shannigrahi is currently working as a senior scientist in the Institute of Materials Research and Engineering, A*STAR (Agency for Science, Technology and Research), Singapore. He is also associated with National University of Singapore as an adjunct Assoc. professor. His research expertise include design, fabrication and characterization of different types of advanced smart materials (mainly oxide electric and magnetic ceramics in their various form factor) for UV, electro-optic, magneto-optic sensing as well as high frequency electromagnetic interference (EMI) shielding applications. He has published more than 95 papers in referred international journals and 10 patents among which 4 technologies have been licensed. He is a regular reviewer of University Ph.D. thesis as well as several international journals including Nature Nanotechnology, J. of Appl. Phys., Appl. Phys. Lett., Ceramics International, etc. He is a member of Materials Research Society-Singapore (MRS-S) and served as one of the symposium chairperson in the International Conference on Materials for Advanced technologies (ICMAT) 2017.

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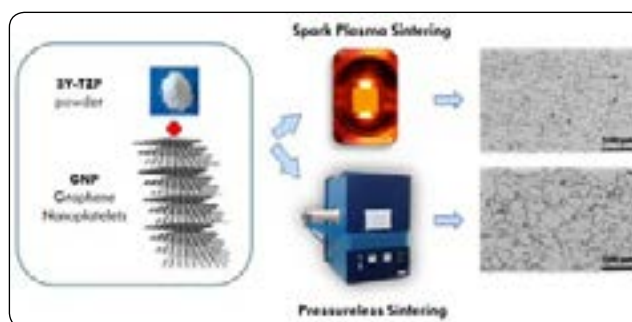
Ceramics and Composite Materials

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Role of sintering method on graphene/3YTZP composites

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Graphene in the form of graphene nanoplatelets (GNP), graphene oxide or few layer graphene has become an ideal filler in fabrication of different polymer, metal or ceramic composites. Recently, the fabrication of ceramic matrix composites with graphene-based materials has attracted a special interest due to the potential improvement of mechanical and functional properties. Amongst ceramic matrices, 3 mol% yttria tetragonal zirconia (3YTZP) presents outstanding mechanical properties and with the addition of GNP can become electrically conductive. The properties of the materials depend not only on the composition, but also on the microstructure. In the case of ceramics, the processing method has a great importance from the point of view of the final properties. Graphene / ceramic composites are typically prepared through wet powder processing followed by a pressure assisted sintering technique, such as Spark Plasma Sintering (SPS) or Hot Pressing (HP). SPS advantages over HP include lower sintering temperatures and shorter sintering times. However, it requires expensive equipment and produces highly anisotropic materials. Conventional pressureless sintering (PLS) is a simpler and cheaper sintering method that produces composites with lower anisotropy. Therefore, the study of graphene / ceramic composites prepared by PLS compared to SPS sintered ones is very interesting. The main objective of this work is to make a direct comparison of the effects of these two sintering techniques (PLS and SPS) on the microstructural features, mechanical and electrical properties of composites of 3YTZP with different contents of GNPs.



Recent Publications

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3. A. Gallardo-López, I. Márquez-Abril, A. Morales-Rodríguez, A. Muñoz, and R. Poyato, "Dense graphene nanoplatelet/yttria tetragonal zirconia composites: Processing, hardness and electrical conductivity," *Ceram. Int.*, vol. 43, no. 15, pp. 11743–11752, Oct. 2017.
4. C. Ramirez, P. Miranzo, M. Belmonte, M. I. Osendi, P. Poza, S. M. Vega-Díaz and M. Terrones, "Extraordinary toughening enhancement and flexural strength in Si₃N₄ composites using graphene sheets", *J. Eur. Ceram. Soc.*, 34, pp. 161–169, 2014.
5. F. Chen, D. Jin, K. Tyeb, B. Wang, Y. H. Han, S. Kim, J. M. Schoenung, Q. Shen, and L. Zhang, "Field assisted sintering of graphene reinforced zirconia ceramics," *Ceram. Int.*, vol. 41, no. 4, pp. 6113–6116, 2015.

Biography

Cristina López Pernía is a doctoral candidate at the Department of Condensed Matter at Universidad de Sevilla. She graduated with a Bachelor of Materials Engineering from Universidad Politécnica de Madrid and holds a Master's Degree in *Advanced Materials* from Universidad Autónoma de Madrid. Currently she focuses her work on graphene-ceramic composites.

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Process development for the ceramic injection molding of oxide short fiber reinforced CMCs

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Ceramic matrix composites (CMCs) are created by adding fibers with variety of fiber structure into various ceramic materials to provide condition / task adapted properties. On the other hand, ceramic injection molding is an automated net shaping process that can produce the ceramic parts with complex geometry and good surface quality without the requirement for a post-processing step. In this study, we produced aluminum oxide CMCs (Al_2O_3 powder: TMDAR, Taimicron; Al_2O_3 chopped fibers: 3M Nextel 610) by using ceramic (μ -) injection molding process. Each step such as feedstock preparation, molding step, debinding or sintering, has significant effect on the final properties of the CMCs parts. Measurement of density as a function of time, temperature, fiber content or -orientation coupled with the examination of the final microstructure is a useful method to evaluate the ceramic parts. The relative density of sintered CMC parts (sintered from 1150 to 1350 °C) decreased at about 35% with increasing amount of oxide chopped fibers from 0 to 25 Vol. % in feedstock including polymer binding system. The reason of such a low sintering temperature for aluminum oxide based material is to prevent the grain growth in the fibers [1]. In addition, the form of the injection mold design defines the fiber orientation that differentiate the density and mechanical properties of the sintered parts. The difference between tensile specimen (higher orientation) and disc form (random orientation) is about from 1 to 10% depending on temperature and the amount of fiber in the feedstock. On the other hand, whether it is hard to define the mechanical characteristic because of highly deformed sintered parts, our first impression on the results is that the fiber reinforced injection molded parts have lower fracture toughness than common CMC parts with endless fibers or fabric.



Figure 1: Fiber orientation and distribution in the neck region of a sintered (at 1250 °C) tensile specimen.

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1. Tülümen M, Hanemann T, Hoffmann M, Oberacker R, Piotter V. (2017). Process Development for the Ceramic Injection Molding of Oxide Chopped Fiber Reinforced Aluminum Oxide. *Key Engineering Materials*. 742. 231-237. DOI: 10.4028
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Biography

Metin Tülümen is a PhD Student at University Freiburg, Germany and working as a research associate in institute of applied materials at Karlsruhe Institute of Technology, working with Prof. Dr. Thomas Hanemann. He earned his Master of Science on materials science and with specialization of glass and ceramics at Clausthal University of Technology, Germany. In his PhD thesis he is developing and characterize new molding compounds containing short ceramic fibers for powder injection processes.

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Composite materials from clays and clay mineral: Synthesis, characterization and electroanalytical applications

Ignas K. Tonlé, Francis M. M. Tchieno, Ervice Yméle, Sherman Z. Jiokeng and Emmanuel Ngameni
University of Dschang, Cameroon

Scientific research devoted to the exploitation of clays and clay minerals for the preparation of nanohybrid materials has gained growing for the past decade. The resort to clays minerals as inorganic parent support for such materials is due to their surface reactivity and ability to immobilize guest organic molecules, either within their structure or on their surface. In fact, clay-based nanohybrid materials offer a wide range of applications in environmental protection, catalysis, polymer science and in analytical electrochemistry. In the last mentioned application, the development of amperometric sensors useful in the analysis and determination of various pollutants (e.g. heavy metals, dyes, pesticides) in natural media represents a daily challenge. Yet, the inherent toxic character of these compounds, combined to the relative low degradation rate of some of them and the non-biodegradable character of others made them persistent in living organisms where they participate in metabolism processes, thereby inducing the monitoring and traceability of toxic pollutants is an ethic duty that concerns the whole scientific community and challenges researchers working in several areas covering analytical chemistry, environmental science, pollution control and chemistry of materials. In this communication, some typical clay-based nanohybrid materials exploited as electrode modifiers for preconcentration electroanalysis will be exposed.

Recent Publications

1. Kenne D G, Nguelo B B, Tonle I K, Ngameni E, Detellier C (2017) Molecular Control of the functional and spatial interlayer environment of kaolinite by the grafting of selected pyridinium ionic liquids. *Applied Clay Science* 143: 445 – 451.
2. Jiokeng Z S L, Dongmo M L, Yméle E, Ngameni E, Tonle I K (2017). Sensitive stripping voltammetry detection of Pb(II) at a glassy carbon electrode modified with an amino-functionalized attapulgite. *Sensors and Actuators: B. Chemical* 242: 1027-1034.
3. Ngassa P G B, Tonle I K, Ngameni E (2016) Square wave voltammetric detection by direct electroreduction of Paranitrophenol (PNP) using an organosmectite film-modified glassy carbon electrode. *Talanta* 147: 547-555.
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Biography

Ignas Kenfack Tonle is Professor of Analytical Chemistry at the Department of Chemistry of the University of Dschang (Cameroon) where he leads a research group working on the development of sensors and biosensors based on inorganic porous materials and lignocellulosic materials. In collaboration with the Electrochemistry and Analytical Chemistry research group of Dr Alain Walcarius (University of Lorraine, Nancy- France), his PhD thesis was defended in 2004 at the University of Yaounde 1 (Cameron) under the supervision of Prof Emmanuel Ngameni. The research was focussed on the grafting of organosilanes onto the surface of smectite-type clay minerals, followed by the application of the obtained organoclays as electrode materials in electroanalysis. In 2008, he moved to Prof Christian Detellier's group (University of Ottawa, Canada) for a one year postdoctoral position. Since 2013, he is a Georg Foster Senior Scientist Fellow to the 'Elektroanalytik & Sensorik' group headed by Prof Dr Wolfgang Schuhmann at the Ruhr-Universität Bochum (Bochum, Germany).

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Sessions:

Day 1 May 14, 2018

Ceramics applications | Porous Ceramics | Ceramic Industry and Environment

Session Chair

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Session Introduction

Title: **Fabrication and characterisation of high-frequency ultrasonic transducers based on piezoelectric thick films and porous backing**

Danjela Kuscer, Jozef Stefan Institute, Slovenia

Title: **Preparation of zirconium-based porous ultra-high temperature ceramics via sol-gel precursors**

Xiao Huang, Shanghai Institute of Ceramics, China

Title: **Practical approaches to energy efficiency improvement in SiC-based ceramics processing**

Maria Vartanyan, D. Mendeleev University of Chemical Technology of Russia, Russia

YRF: **3D-printing of ceramic-based porous structures using stereolithography**

Dieraert Axel, CEA, DAM, France

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Fabrication and characterisation of high-frequency ultrasonic transducers based on piezoelectric thick films and porous backing

Danjela Kuscer¹, Tina Bakarič¹, Silvo Drnovšek¹, Barbara Malič¹, Julien Bustillo² and Franck Levassort²¹Jozef Stefan Institute, Slovenia²François Rabelais University, France

The properties of the high-frequency transducer (>20 MHz), in particular its operating frequency, sensitivity and resolution, are defined by the geometry, microstructure and characteristics of piezoelectric material and backing. For the backing, acoustical impedance and attenuation coefficient are predominant properties to be determined. In this work, we proposed a novel method for the in-situ measuring of these backing's properties at the operating frequency of the transducer.

We report on the processing and characterization of lead-zirconate-titanate based (PZT) piezoelectric thick films on a porous backing with tailored amount, size and shape of the pores. As a porous backing we used ~5mm-thick ceramic with nominal composition $\text{Pb}(\text{Zr}_{0.53}\text{Ti}_{0.47})\text{O}_3$ (PZT). The ceramic was prepared by hetero-coagulation process of PZT and polymethylmetacrylate in water at pH 8 followed by sintering the powder compacts at 1080 °C. Ceramic exhibited homogeneous microstructure with 15 % porosity and spherical, ~1 and ~10 µm-sized pores, respectively. The PZT thick films, screen-printed onto the electroded backing and sintered at 900 °C, had a thickness of ~25 µm, porosity of 20 % and thickness coupling coefficient of 45 %.

This integrated piezoelectric structure allows direct acoustic measurements of transducer components. The PZT thick film is electrically excited to measure the electroacoustic response in water and also the back-wall echoes coming from the backing if its thickness is sufficiently thin. The thickness of the backing was successively reduced and the measurements were repeated.

In the frequency range 15-25 MHz, the attenuation coefficients of backings with 1- and 10-µm- sized pores were 0.7 dB/mm/MHz and 4 dB/mm/MHz, respectively, the group velocities were ~3400 m/s which results in the acoustic impedance of ~22 MRa. The high attenuation in backing with 10 µm-sized pores and moderate acoustical impedance enable substantial miniaturisation of high-resolution ultrasonic imaging transducers.

Recent Publications

1. Kuscer D, Rojac T, Belavič D, Santo Zarnik M, Bradeško A, Kos T, Malič B, Boerrigter M, Martin DM, Faccini M (2017). »Integrated piezoelectric vibration system for fouling mitigation in ceramic filtration membranes«. J. Membr. Sci., 540:277-284.
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Biography

Asst. Prof. Danjela Kuscer, PhD in material science at the University of Ljubljana, Slovenia (1999). Current position: senior researcher at Jožef Stefan Institute and Assistant Professor at Jožef Stefan International Postgraduated School, Ljubljana, Slovenia.

Research: synthesis and characterisation of complex-composition ceramic applicable in electronics, including synthesis of (nano) powders by mechanically-assisted and solid state synthesis, synthesis of ceramic with tailored microstructure, patterning of thick film structures using water- and organic-based suspensions by electrophoretic deposition, screen- and inkjet printing, and their structural, microstructural and functional characterisation.

Publications: 120 publications and 150 technical reports. She holds one Slovenian, two USA patents and three PCT patent applications. She participated in 31 projects, of which she leads 7 EU and 5 national applied projects. Between 2014 and 2017 she won six national awards for the innovation in the field of ceramics, the most important being Puh recognition for 2015, the highest Slovenian science award for important applied work.

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Preparation of zirconium-based porous ultra-high temperature ceramics via sol-gel precursors

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Ultra high temperature ceramics (UHTCs) are a class of inorganic materials that have melting point over 3000 °C and are typically borides, carbides and nitrides of early transition metals. UHTCs are considered as the promising candidate used in the extreme environment involved with the hypersonic aviation thermal protective system. Synthesis of UHTC based materials can be divided into solid based and solution based protocols according to the state of the raw materials. Sol-gel process is one of the solution based protocols for the preparation of UHTC based materials, which involves the hydrolysis, condensation of the metal organic and/or metal inorganic compounds, gelation and the post high temperature treatment of the dried gels. Here, we present the preparation of several zirconium-based porous UHTCs. The formation and manipulation of the pore structure and the correlation to their properties are also discussed.

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Practical approaches to energy efficiency improvement in SiC-based ceramics processing

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Statement of the Problem: Glass and ceramic industries fall into the category of energy-intensive industries and since 2014 when Integrated Pollution Prevention and Control was introduced in Russia these sectors have been closely monitored with regards to resource efficiency and environmental performance. Methodology & Theoretical Orientation: Traditional sub-sectors including tile and brick manufacturing in 2003-2013 participated in pilot projects intended to evaluate possibilities of implementing Best Available Techniques (BATs) in Russian industries. Based on the results of these projects BATs have been identified and national Information and Technical Reference Books developed. In manufacturing technical ceramics existing sector-specific BATs comprise mostly emissions control. However, considering materials for advanced applications such as alumina, zirconia or carborundum, where these levels are determined by strict process parameters, a generally accepted practice is to reduce energy consumption by adjusting firing temperature. This allows to suggest candidate BATs: batch composition adjustment, the use of eutectic sintering aids, and liquid-phase sintering. Findings: The present research addresses a combination of these techniques in production of SiC-based structural ceramics including selection of additives based on their physico-chemical properties (melting point, surface interaction) and the use of pre-fabricated sintering aids with enhanced reactivity. The effects of the additives on sintering behavior, phase composition, microstructure, and mechanical properties were studied. Conclusion & Significance: The combination of ultrafine SiC with 25 vol. % of a eutectic sintering aid in MgO-Al₂O₃-Y₂O₃ system allowed to produce ceramics with bending strength of 450 MPa, fracture toughness of 4.0 MPa·m^{1/2}, and elasticity modulus of 380 GPa. Sintering temperature for this material didn't exceed 1900°C, which makes this technique practically suitable to reduce emissions and the impact on the environment. This research was carried out with financial support from the Ministry of Education and Science of the Russian Federation under State order, contract No. 10.6309.2017/BCh.

Recent Publications

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3. Reference Document on Best Available Techniques in the Ceramic Manufacturing Industry. European IPPC Bureau, 2007.
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5. S.V. Zhitnyuk, D.Yu. Zhukov, D.O. Lemeshev, N.A. Makarov (2015) Modification of Silicon Carbide Based Ceramic Properties by Granulometric Composition Regulation. Glass and Ceramics. 72(7-8):245-253.
6. Mararakin M.D., Vartanyan M.A., Makarov N.A., Sazhin I.V. (2017) Sol-gel synthesis of eutectic additives for silicon carbide-based ceramics. Glass and Ceramics. 9:25-27 (in Russian).

Biography

Maria Vartanyan is an expert in ceramics manufacturing, energy efficiency and environmental performance in production of ceramics materials based on oxide and non-oxide compounds.

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4th International Conference and Expo on

Ceramics and Composite Materials

May 14-15, 2018 | Rome, Italy

3D-printing of ceramic-based porous structures using stereolithography

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Stereolithography-based additive manufacturing (AM) is increasingly becoming the technology of choice for the small series or single unit production. It also allows the material-by-design fabrication, prime concern for most material chemists. State-of-the-art of three-dimensional (3D) structured organic-inorganic hybrid materials will be described and focus onto combined 3D printing technology and sol-gel ceramic-based material preparation will be emphasized. Although the spectrum of available 3D-printed materials has been widened in recent years, there is still a lack of ceramic-based materials which can be processed with stereolithography on a routine basis. The extremely high melting point of many ceramics adds challenges to additive manufacturing as compared with metals and polymers. Because ceramics cannot be cast or machined easily, three-dimensional (3D) printing enables a big leap in geometrical flexibility and microstructured architecture. We report work on synthesis new ceramics from preceramic monomers or inorganic nanopowder loaded resins that are cured with ultraviolet light in a stereolithography 3D printer. After a thermal debinding and sintering step the part turns into a dense ceramic open structure and gains its final properties, with uniform shrinkage and porosity control. The paper discusses the critical process parameters that influence polymerization uniformity and structure quality. Currently it is possible to print 3D-structures with a spatial resolution down to 40 μm , with complex shape and cellular architecture. The photosensible resin formulation is a key parameter to control the printing resolution, so the geometry of the final ceramic. Highly complex three-dimensional open microstructures have been theoretically designed to lead to the best agreement between thermal insulation properties and mechanical toughness. Experimental characterization and performances of the AM ceramic parts will be discussed with regard to high temperature super insulation material application, exhibiting high-stiff properties. Finally, scale-up of this combined approach is carefully considered.



Recent Publications

1. Eckel, Z. C., et al., *Additive manufacturing* of polymer-derived ceramics. *Science* 2016, 351, 6268.
2. Hundley, J. M., et al., Geometric Characterization of Additively Manufactured Polymer Derived Ceramics. *Additive Manufacturing* 2017.
3. Zanchetta, E., et al., Stereolithography of SiOC Ceramic Microcomponents. *Advanced Materials* 2016, 28, (2), 370-376.
4. De Hazan, Y.; Penner, D., SiC and SiOC ceramic articles produced by stereolithography of acrylate modified polycarbosilane systems. *Journal of the European Ceramic Society* 2017.
5. Schmidt, J.; Colombo, P., Digital light processing of ceramic components from polysiloxanes. *Journal of the European Ceramic Society* 2017.

Biography

Axel Dieraert obtains his Master's degree in inorganic chemistry in 2016 at the University of Montpellier, in France (major of promotion, specialty "Porous materials, divided systems and thin layers"). Since 2017, he's PhD student at CEA Le Ripault ("Commissariat à l'Energie Atomique et aux Energies Alternatives"), under the direction of Philippe Belleville (CEA Le Ripault – PhD director) and Clement Sanchez (UPMC Paris - scientific adviser). The purpose of this thesis is to develop new materials for high temperature insulation based on stereolithography of ceramic materials.

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4th International Conference and Expo on

Ceramics and Composite Materials

May 14-15, 2018 | Rome, Italy

Scientific Tracks & Abstracts Day 2

Ceramics 2018

Sessions:

Day 2 May 15, 2018

Functional Ceramics and Inorganics | Materials and Innovative Processing Ideas | Ceramics and Art | Ceramics and Glasses

Session Chair

Ghatu Subhash

University of Florida, USA

Session Chair

Julian Reinos

Institute of Ceramics and Glass (CSIC), Spain

Session Introduction

Title: Ceramics in the system $\text{ZrO}_2\text{-Al}_2\text{O}_3$ with eutectic additives

Nikolay Makarov, D.Mendeleev University of Chemical Technology of Russia, Russia

Title: Homogeneous precipitation of ceramic powders preparation from sulfate-sulfite-ammonia system

Satoshi Sugita, University of Guanajuato, Mexico

Title: Use of ceramic materials in street art

Fidan Tonza, Ondokuz Mayıs University, Türkiye

Title: Usage of boron waste in transparent glazes

Fidan Tonza, Ondokuz Mayıs University, Türkiye

Title: *In Vivo* behaviour of Ag^+ ion doped calcium phosphate based ceramic powder coating on $\text{Ti}_6\text{Al}_4\text{V}$ implants

Ceren Peksen, Ondokuz Mayıs University, Türkiye

Title: Development and characterisation of Ox-Ox CMCs components for high temperature insulation application

Virtudes Rubio, NCC, UK

4th International Conference and Expo on

Ceramics and Composite Materials

May 14-15, 2018 | Rome, Italy

Ceramics in the system $\text{ZrO}_2\text{-Al}_2\text{O}_3$ with eutectic additives

Nikolay A. Makarov and Dmitry A. Antonov

D. Mendeleev University of Chemical Technology, Russia

Statement of the Problem: The $\text{ZrO}_2\text{-Al}_2\text{O}_3$ system is key for the synthesis of various structural materials; in particular, it is promising for manufacturing of wear-loaded items like friction pairs, cutting tools, etc. One of the most important issues in zirconia ceramics technology is that ZrO_2 is subject to intensive recrystallization at temperatures exceeding 1100-1200°C. This phenomenon leads to a martensitic transformation, accompanied by a significant drop in mechanical strength.

Methodology & Theoretical Orientation: An effective way to prevent recrystallization is to affect the grain of zirconia with an external compressive load, since in the stressed state the tetragonal solid solution does not decompose. It is possible to create an external load by introducing a second phase into the material, the role of which is taken up by alumina. The aim of the work is to create ceramic materials in the $\text{ZrO}_2\text{-Al}_2\text{O}_3$ system, with predominant zirconium dioxide, having a sintering temperature of 1400-1550 °C, and high mechanical properties. An attempt was made to reduce sintering temperature by the addition of eutectic aids to the batch. The influence of various types of alumina (commercial, obtained by chemical precipitation) on the structural properties, mechanical strength and microstructure parameters was analyzed. Zirconia was obtained by chemical precipitation, partial stabilization was carried out using yttrium chloride. To control the structure and properties of ceramics, modifiers were used in $\text{CaO-Al}_2\text{O}_3\text{-SiO}_2$ and MnO-TiO_2 systems.

Findings: It was found that ceramics with eutectic additives $\text{CaO-Al}_2\text{O}_3\text{-SiO}_2$ and MnO-TiO_2 in the ratio 1: 1 possesses the greatest mechanical strength - 750 ± 35 MPa. **Conclusion & Significance:** This ceramic material is characterized by the highest microhardness of 1200 N/mm². The material is promising for use as elements of stop valves, mill bodies, cutting tools; wear-resistant structural parts, etc.

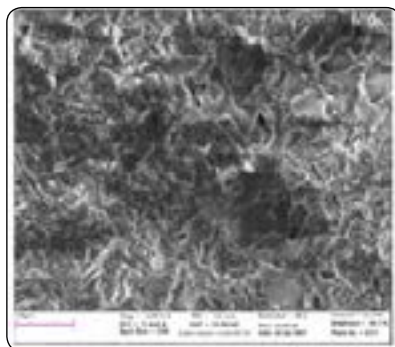


Figure 1: Microstructure of samples from partially stabilized zirconia and alumina with eutectic additives in $\text{CaO-Al}_2\text{O}_3\text{-SiO}_2$ and MnO-TiO_2 systems

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2. Makarov N.A. (2006) Features of sintering corundum ceramics, modified with eutectic additives. Glass and Ceramics. 4:16-18 (in Russian).
3. Lukin E.S., Anufrieva E.V., Makarov N.A., Popova N.A. (2008) New generation of oxide ceramics and the field of its application. Glass and Ceramics. 10:27-31 (in Russian).
4. Lukin E.S., Kozlov A.I., Makarov N.A., Popova N.A. (2009) Nanopowders for the production of oxide ceramics of the new generation. Refractories and Industrial Ceramics. 11:29-34 (in Russian).
5. Makarov N.A., Sverdlikov V.L. (2005) Composite material of the corundum - zirconia system - sintering additive. Glass and Ceramics. 11:16-18 (in Russian).

Ceramics and Composite Materials

May 14-15, 2018 | Rome, Italy

Biography

N. Makarov's scientific activity is dedicated to the creation of new types of ceramic materials, the study of their properties and makes a significant contribution to the development of the physico-chemistry of the processes underlying the technology of ceramics for special purposes, including nanomaterials. Scientific interests lie in the field of chemistry and technology of materials with a controlled structure and given properties based on aluminum and zirconium oxides, as well as oxygen-free compounds; directed control of the formation of the structure of ceramics from oxides and anoxic compounds; development of the theory and mechanism of sintering of ceramic materials modified by additives of various nature; development of energy- and resource-efficient technologies of ceramic materials possessing a high level of physico-mechanical properties and a low temperature of sintering.

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Notes:

4th International Conference and Expo on

Ceramics and Composite Materials

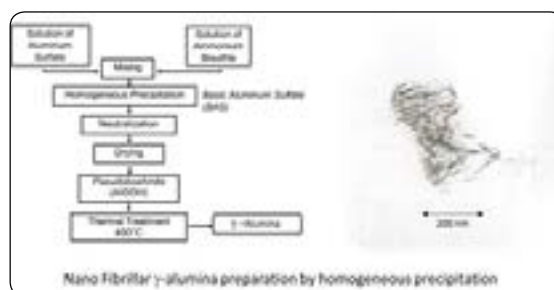
May 14-15, 2018 | Rome, Italy

Homogeneous precipitation of ceramic powders preparation from sulfate-sulfite-ammonia system

Satoshi Sugita

University of Guanajuato, Mexico

High quality ceramic powders can be produced by homogeneous precipitation. This method permits to control purity, particle size, shape, distribution and homogeneity, as known these factors play a very important role in the properties of finished ceramic materials. Alumina nano-sized particles could be obtained starting from the admixture of solutions of aluminum sulfate and ammonium bisulfite. The homogeneous precipitation carried out at pH 4.2, when the mixture of solutions was heated to the decomposition and boiling temperature. Nano-, spherical with narrow size distribution, and soft agglomerated, the basic aluminum sulfate (BAS) was easily produced. The powder was heated at 950 °C to decompose sulfate and then was calcined at 1250°C to transform to α -alumina. Otherwise, treating BAS with aqueous ammonium solution, the precipitate could be transformed to nano-sized fibrillar pseudoboehmite, which could be transformed to α -alumina by calcining. On the other hand, to prepare mullite precursor, initially the silica nano powder (Aerosil 200, Degussa) was dispersed in water and mixed with the solutions of aluminum sulfate and ammonium bisulfite. The admixture was heated to the decomposition and boiling temperature, in which a precipitate of silica covered with BAS was produced. This precursor was transformed to mullite heating at 1250°C. The other application example of this homogeneous precipitation is used to prepare in the fabrication of basic chromium sulfate. Starting from the admixture of solutions of chromium sulfate and ammonium bisulfite, the basic chromium sulfate can be precipitated. But in the last case it is not easy for all chromium precipitate since chromium forms a water soluble complex with ammonia. Even though, almost every chromium can be precipitated. In this presentation, a simple useful technique of homogeneous precipitation for preparing the basic sulfate salts of metals starting from the admixture of metal ion-sulfate-sulfite-ammonia aqueous system is discussed.



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2. López FJ, Sugita S, Kobayashi T (2014) Cesium-adsorbent geopolymer foams based on silica from rice husk and metakaolin. *Chem. Lett.* 43:128-30.
3. Reyes SY, Serrato J, Sugita S (2013) Microstructural characterization of sanitaryware, the relationship spinel and mullite, *Journal of Ceramic Processing Research* 14, 4:492-497.
4. Zamorategui A, Alatorre A, Ibanez J, Garcia MG, Nosaka Y, Kobayashi T, Sugita S (2013) Thermodynamic and electrochemical study on the mechanism of formation of $\text{Ag}(\text{OH})_4^-$ in alkaline media, *Electrochimica Acta* 111:268-74.
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6. Zamorategui A, Martinez JM, Soto JA, Sugita S (2013) Ammonium bisulfite as a precipitant agent to synthesize pseudoboehmite ($\gamma\text{-AlOOH}$), *International Journal of Engineering and Innovative Technology* 3, 5:401-6.
7. Tovar-Carrillo KL, Sugita S, Tagaya M, Kobayashi T (2013) Fibroblast compatibility on scaffold hydrogels prepared from Agave Tequilana Weber bagasse for Tissue Regeneration, *Ind. Eng. Chem. Res.* 52, 33:11607-11613.

Ceramics and Composite Materials

May 14-15, 2018 | Rome, Italy

Biography

Satoshi Sugita has his expertise in ceramic powder processing. His homogeneous precipitation method to prepare the precursors of ceramic powders provides the opportunity to obtain nano-sized basic metal salts, which converts easily soft agglomerated ceramic powders, i.e. nano fibrillar γ -alumina, spherical α -alumina, spherical and fibrous mullite, etc. With this homogeneous precipitation, ammonium bisulfite is used as precipitant, and the control of the reaction conditions are very simple. This technique has the great possibility of preparing different types of ceramic powders.

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Notes:

4th International Conference and Expo on

Ceramics and Composite Materials

May 14-15, 2018 | Rome, Italy

Use of ceramic materials in street art

Fidan Tonza¹ and Sevim Çizer²¹Ondokuz Mayıs University, Turkey²Dokuz Eylül University, Turkey

Street in the public space, as the area belonging to everybody, comes out as the most important place where the pulse of the city is thrown. The heterogeneous structure that emerges with urbanization comes to the forefront with the multitude of voices it creates and is the voice of the discourse of the individual's living space. Public spaces and frame Political structure have shaped from past to the present-day in public space in the context of social relationships. The street art, which has positioned the street as a public space in the center, but with the timeframe of the freedom of expression of its own field, has existed directly in this area. The street art that transcends the boundaries and becomes widespread continues to show its diversity of working examples in different disciplines today and forms a space for the formation of new discourses together with elements belonging to the street. The use of ceramic materials in the public space, on the street, provides important practices for opening new areas in street art. At the forefront of these approaches that change the perception of traditional materials, street art has a dynamic connection with art, especially with the use of ceramics and the viewer factor that directly relate to it. Contrary to the fast performance process of street art, ceramic material contains slow and different application items, also points to the existence of a planned production process with the approach of different disciplines in street art. Apart from the gallery and museum concept of exhibition space, the use of ceramics, which are familiar to view, is emphasized in this work. Furthermore, except of the use of ceramic material as a building material in urban aesthetics, it also focused on its existence as an artistic discourse. The aim of this work is to develop and exemplify different expansions in the name of ceramics art in the direction of the experiences and practices of the unexpected dynamics of the street in the process from the production process to the exhibition on the street as a public place.

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1. Riggle N. A. (2010) Street Art: The Transfiguration of the Commonplaces, Journal of Aesthetics and Art Criticism 68 (3):243-257
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5. Hunter, Garry (2012) Street Art: From Around the World, London: Arcturus.
6. Ket (2011) Street Art: The Best Urban Art From Around the World, London: Michael O'Mara Books Limited.

Biography

Fidan Tonza graduated from Dokuz Eylül University, Faculty of Fine Arts, and Department of Ceramics in 2007. She completed her Master of Arts Degree at the same university on the subject of "Research of Eastern Mysticism, Anatolian Sufism and Ceramic Applications" in 2012. She completed her Ph.D. at Dokuz Eylül University on the subject of "The Association of Street Art With Ceramic Material". She has participated in several international and national exhibitions. She has also received 3 awards. In 2011 she founded her own studio "Fidan Tonza Art Studio" in Izmir. In 2017, she started to work as an assistant professor at Samsun Ondokuz Mayıs University of Fine Arts Faculty, Ceramic-Glass Department.

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4th International Conference and Expo on

Ceramics and Composite Materials

May 14-15, 2018 | Rome, Italy

Usage of boron waste in transparent glazes

Fidan Tonza¹, Ceren Peksen² and Erhan Ayas³^{1,2}Ondokuz Mayıs University, Türkiye³Anadolu University, Türkiye

The recycling of waste materials contribution and usage as a raw material are important in terms of conservation of natural balance and energy saving. Boron raw materials are the most important natural sources for many industrial applications such as ceramic, agriculture and construction and their values become increased with the finding of boron usage in ceramic industry. Boron is mined in Eskişehir Kirka region of Türkiye which has the biggest boron deposition of the World. Instead of recreating the raw materials, the use of boron wastes has gained importance in ceramic glaze preparation because of preserving the reserves. In the present study wastes of Eti Maden Kirka Plant were used in the production of ceramic glazes which was suitable for different types of ceramic bodies. Boron waste added transparent glazes compositions were applied on the biscuit fired ceramic samples and single firing was employed. The effects of the glaze compositions prepared with the different additions of the boron waste on the ceramic surfaces were evaluated. It was seen that the boron waste added glaze is formed on the ceramic bodies.

Recent Publications:

1. Kaman D, Koroglu L, Ayas E, Guney Y (2017) The effect of heat-treated boron derivative at 600 C on the mechanical and microstructural properties of cement mortar, *Construction and Building Materials*, 154:743-7541.
2. Barutca B, Koroglu L, Ayas E, Koparal T (2018) In vitro cytotoxicity of monticellite based bioactive ceramic powder synthesized from boron derivative waste, *Ceramics International*.
3. Koroglu L, Butev E, Esen Z, Ayas E (2017), A novel approach for synthesis of monticellite based bioactive ceramic powders from boron derivative waste, *Materials Letters*, 209: 315-318.
4. Uslu T, Arol A (2004) Use of boron waste as an additive in red bricks, *Waste Management*, 24:217-220.
5. Kavas T (2006) Use of boron waste as a fluxing agent in production of red mud brick, *Building and Environment*, 41:1779-1783.

Biography

Fidan Tonza graduated from Dokuz Eylül University, Faculty of Fine Arts, and Department of Ceramics in 2007. She completed her Master of Arts Degree at the same university on the subject of "Research of Eastern Mysticism, Anatolian Sufism and Ceramic Applications" in 2012. She completed her Ph.D. at Dokuz Eylül University on the subject of "The Association of Street Art With Ceramic Material". She has participated in several international and national exhibitions. She has also received 3 awards. In 2011 she founded her own studio "Fidan Tonza Art Studio" in İzmir. In 2017, she started to work as an assistant professor at Samsun Ondokuz Mayıs University of Fine Arts Faculty, Ceramic-Glass Department.

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4th International Conference and Expo on

Ceramics and Composite Materials

May 14-15, 2018 | Rome, Italy

In Vivo behaviour of Ag⁺ ion doped calcium phosphate based ceramic powder coating on Ti₆Al₄V implants

Ceren Peksen¹, Remzi Caylak², Nusret Kose³ and Aydin Dogan⁴¹Ondokuz Mayıs University, Türkiye²Private Ortopedia Hospital, Türkiye³Osmangazi University, Türkiye⁴Anadolu University, Türkiye

Long-term survival and favorable outcome of orthopaedic implant use are determined by bone-implant osseointegration and absence of infection near the implants. To enhance resistance to colonization, implant materials may be modified with antimicrobial coatings. The mechanism of the antimicrobial action of silver ions is closely related to their interaction with thiol groups. In this study, Ag⁺ ion doped calcium phosphate based ceramic nanopowder coated Ti₆Al₄V implants were evaluated to prevent implant-related infection by comparing hydroxyapatite (HA) coated and uncoated titanium implants in vivo. Ag⁺ ion doped calcium phosphate based nano-powder were deposited on Ti₆Al₄V implants by using electrospray deposition technique. Electrospray coated samples were sintered under high vacuum by RF (radio frequency). After sintering process surface morphologies of implants were observed with scanning electron microscope. Prior to surgery, rabbits were randomised to receive either coated implants or uncoated implants. First group of the implants were coated with Ag⁺ ion doped nano size calcium phosphate based ceramic powder. Second group of the implants were coated with hydroxyapatite(HA), and the remaining implants (Group 3) were used without any coating. Implants were inserted left femurs of animals from knee regions with retrograde fashion. Before implantation of implants 50 µl solution containing 10⁶ CFU/ml methicillin resistance *Staphylococcus aureus* (MRSA) injected intramedullary canal. Rabbits were monitored for 10 weeks. At the end of the 10 weeks animals were sacrificed and rods were extracted in a sterile fashion. Swab cultures were taken from intramedullary canal. Bacteria on titanium rods were counted. Histopathological evaluation of bone surrounding implants was also performed. In conclusion, Ag⁺ ion doped calcium phosphate based ceramic nano powder coated Ti₆Al₄V implants may prevents bacterial colonisation and infection compared with those for implants without coating and HA coated implants.

Recent Publications:

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5. Zhao, L et al. (2009) Antibacterial coatings on titanium implants, Journal of Biomedical Materials Research Part B: Applied Biomaterials 91: 470-480.

Biography

Ceren Peksen received the B.E. degree in ceramic engineering from the Dumlupınar University and the MSc and PhD degrees in ceramic engineering from the Anadolu University Institute of Science, Türkiye, in 2006 and 2012, respectively. In 2014, she joined the Department of Ceramic and Glass, Ondokuz Mayıs University, as a lecturer and became an Assistant Professor. Her current research interests include ceramic powder synthesis, ceramic surface coatings, bioceramics, antibacterial materials and materials characterization.

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