

# 15<sup>TH</sup> ECerS CONFERENCE FOR YOUNG SCIENTISTS IN CERAMICS

# **BOOK OF ABSTRACTS**

October 11-14, 2023 Faculty of Technology Novi Sad Novi Sad, Serbia

# **15<sup>th</sup> ECerS CONFERENCE for YOUNG SCIENTISTS in CERAMICS**

# PROGRAMME and BOOK OF ABSTRACTS

October 11-14, 2023 Novi Sad, Serbia Programme and Book of Abstracts of The ECerS 15<sup>th</sup> Conference for Young Scientists in Ceramics (CYSC-2023) publishes abstracts from the field of ceramics, which are presented at traditional international Conference for Young Scientists in Ceramics.

*Editors-in-Chief* Prof. Dr. Vladimir V. Srdić Dr. Soňa Hříbalová

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### Preface

Dear colleagues and guests we are delighted to welcome you all to Novi Sad, Serbia and the 15<sup>th</sup> ECerS Conference for Young Scientists in Ceramics. This biannual event is once again jointly organized by the Faculty of Technology Novi Sad, University of Novi Sad and the Young Ceramists Network (YCN) of the European Ceramic Society (ECerS).

The ECerS Conference for Young Scientists in Ceramics is celebrating its 25<sup>th</sup> anniversary since it started back in 1998 as a national event and now it gathers scientists from all over the world. During all these 25 years the conference has been growing constantly and we are proud to say that it became one of the trademark events in the field of ceramics in Europe.

During the four days of the Conference we will have an opportunity to hear 104 oral presentations given by young scientists together with 12 invited talks and 5 plenary lectures of the more experienced scientists and experts from 29 countries. In addition, we will host a satellite event "Workshop on atomistic calculations in materials science", thoughtfully designed to introduce fundamental computational methods that are accessible to beginners in this field. Thus, we continue to be the venue for the vivid exchange of ideas and knowledge intertwined with fruitful discussions about the one topic that gathers us all - ceramic materials and all its subfields. Young scientists especially have the opportunity to meet with their peers and senior colleagues to promote their work and make new connections that can benefit them throughout their carrier. We have to emphasize that the feedback from our past conferences, which we get from former participants and guests, is more than positive and gives us ever new energy to endure in our mission of bringing young people involved in ceramics closer together. This is why we are confident that you will enjoy your stay in Novi Sad and be able to broaden your knowledge since topics covered by the conference include various aspects of the ceramics including processing, characterisation and application of advanced and traditional ceramics but also cutting edge results in advance manufacturing, high entropy oxides, computer modelling and physics of the ceramic materials and structures.

Our deepest gratitude goes to our sponsors and co-organizers since we would not be able to organize this conference without them. Once again, the JECS Trust Fund of the European Ceramic Society has recognized the significance of the CYSC and became our greatest financial benefactor. Also, we are thankful to the Serbian Ministry of science and technological development which once again endorsed the conference financially. At the end, we would like to thank to all the people in the local organizing committee and colleagues from YCN who participated in the preparations of the Conference.

Editors

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# 15<sup>th</sup> ECerS Conference for Young Scientists in Ceramics, CYSC-2023 Novi Sad, Serbia, October 11-14, 2023

PROGRAMME

# WEDNESDAY, OCTOBER 11, 2023.

- 09.00 11.00 h Registration (Conference desk)
- 11.00 11.30 h Oppening (Rectorate-Amphitheater)
- 12.00 12.45 h PL-1 Plenary lecture (Rectorate-Amphitheater) Tadej Rojac, Slovenia, Origins and mechanistic aspects of the high piezoelectricity of lead-based relaxor-ferroelectric ceramics
- 12.45 14.00 h Welcome Party (Rectorate-Ceremonial Hall)

## 14.00 - 16.00 h - Section 1

*Synthesis of ceramic powders - 1* (Rectorate-Amphitheater)

14.00 – 14.15 h	OA-1 - Vlad-Alexandru Lukacs, Romania
	Comparative analysis of BaTiO <sub>3</sub> nanoceramics derived from
	cuboidal and equiaxed nanoparticles
14.15 – 14.30 h	OC-2 - Marcell Bohus, Hungary
	Investigation of CNT/oxide composites in the application of nanofluids
14.30 – 14.45 h	OA-3 - Eliška Virágová, Czech Republic
	Development of ceramic suspensions for lithography based ceramics manufacturing (LCM)
14.45 – 15.00 h	OA-4 - Emilija Nidžović, Serbia
	High-entropy spinel oxides: Fundamentals, synthesis and
	characterization
15.00 – 15.15 h	OA-5 - Nida Khan, India
	Development of porous hyper-stoichiometric lithium titanate
	$(Li_2TiO_3)$ for tritium breeder application
15.15 – 15.30 h	OA-6 - Gamze Yüksel, Turkey
	Critical parameters for growth of oriented ZnO nanowire
	arrays during hydrothermal synthesis
15.30 – 15.45 h	OA-7 - Aleksandra Milojkovic, Germany
	Tuning the properties of the magnetostrictive cobalt ferrite – A
	promising candidate for wireless neural stimulation application
15.45 – 16.00 h	OA-8 - Pavlína Šárfy, Czech Republic
	Optimization and bioactivity evaluation of silica-doped
	hydroxyapatite scaffolds for bone tissue engineering: A direct
	foaming approach

#### 16.00 – 16.15 h – Coffe Break

16.15 – 17.00 h – PL-2 Plenary lecture (Rectorate-Amphitheater) Markus Winterer, Germany, Combining Reverse Monte Carlo analysis of X-ray scattering and extended X-ray absorption fine structure of small nanoparticles

#### 17.00 - 19.00 h - Section 2

#### *Ceramic processing - 1* (Rectorate-Amphitheater)

17.00 – 17.15 h	OA-9 - Álvaro Sández, Spain
	Combining freeze casting with pressure-less spark plasma
	sintering for the manufactu-ring of bulk ultra-high temperature
	ceramics
17.15 – 17.30 h	OA-10 - Radoslaw Zurowski, Poland
	Ceramic microbeads fabricated via UV curing assisted drop- casting method
17.30 – 17.45 h	OA-11 - Alisa Tatarinova, Russia
	Features of sintering nanopowders of metastable aluminum
	oxide doped with stabilized zirconium dioxide
17.45 – 18.00 h	OA-12 - Anna Maria Wieclaw-Midor, Poland
	Photocurable, aqueous ceramic dispersions for 3D printing
	techniques
18.00 – 18.15 h	OA-13 - Joanna Tanska, Poland
	DLP printing method in obtaining of ceramic-metal
	composites
18.15 – 18.30 h	OC-14 - Oksana Baranovska, Ukraine
	Features of the phase and structure formation of powder
	composites of the Al-Ti-C system reinforced with finely
	dispersed $Al_2O_3$ obtained by the method of thermal synthesis
18.30 – 18.45 h	OA-15 - Heloise Orihuel, France
	Solution-based deposition of ceramics on glass substrates for
	alkali-metal vapor cells
18.45 – 19.00 h	OC-16 - Łukasz Rakoczy, Poland
	Microstructure and selected properties of the metal-ceramic
	nanocomposites for the aerospace applications

# THURSDAY, OCTOBER 12, 2023.

## 09.15 - 11.00 h - Section 3

Engineering and	refractory ceramics - 1 (Rectorate-Amphitheater)
09.15 – 09.30 h	OC-17 - Alexander V. Maletskyi, Ukraine
	Features of structure formation of ZTA ceramics doped with
	zirconia
09.30 – 09.45 h	OC-18 - Derya Arslan, Turkey
	Effect of metal-organic framework (MOF) incorporation on
	the properties of $\alpha/\beta$ -SiAlON matrix ceramic composites
09.45 – 10.00 h	OA-19 - Zofia Kucia, Poland
	Synthesis and structural analysis of polysilazane-derived SiCN
	with tuneable carbon content
10.00 – 10.15 h	OA-20 - Anastasia Kucheryavaya, Austria
	Tailoring the properties of zirconium oxycarbides and
	oxycarbonitrides by adjusting their chemical compositions
10.15 – 10.30 h	OC-21 - Dawid Kozień, Poland
	UHTCs composites based on the boron carbide with
	intermetallic additives from Ti-Si system
10.30 – 10.45 h	OC-22 - James Alexander, United Kingdom
	Production of functionally gradient ceramic-metal
	interpenetrating composites via pressureless infiltration for
	ballistic applications
10.45 – 11.00 h	OA-23 - Lucie Kotrbová, Czech Republic
	Predictions of the grain size dependence of thermal
	conductivity for La <sub>2</sub> Zr <sub>2</sub> O <sub>7</sub> and other pyrochlore ceramics

*Synthesis of ceramic powders - 2* (Rectorate-Multimedia Room)

09.15 – 09.30 h	<b>OA-24 - Nyemaga Malima, Tanzania</b> Composition tunable $Ni_{1-x}Mg_xFe_2O_4$ ( $0 \le x \le 1$ ) ceramic nanocatalysts for enhanced hydrogen evolution and oxygen
	evolution reactions
09.30 – 09.45 h	OA-25 - Jakub Aleksandrowicz, Poland
	Optimization of phenyl ladder-like systems by sol-gel synthesis
09.45 – 10.00 h	OA-26 - Milena Dojčinović, Serbia
	Magnesium substitution with nickel and its influence on the sensing properties of $MgFe_2O_4$
10.00 – 10.15 h	OA-27 - Jan Słomiński, Poland
	Synthesis of ternary boride Cr <sub>3</sub> AlB <sub>4</sub> by solid-state reaction
10.15 – 10.30 h	OA-28 - Hamza Boussebha, Turkey
	AiON powder via dynamic chemical method

10.30 – 10.45 h	OA-29 - Tijana Vlašković, Serbia
	Preparation, synthesis and characterization of nanometric
	$Ca_{0.9}Er_{0.1}MnO_3$
10.45 – 11.00 h	OA-30 - Elvira Toth, Serbia
	Vanadium-carbide MXene synthesis and characterization for
	SERS sensors

#### 11.00 - 11.15 h – Coffe Break

- 11.15 11.45 h IL-2 Invited lecture (Rectorate-Amphitheater) Julian Walker, Norway, Ionic molecular systems – Next generation "ceramics" for electronic and thermal energy storage applications
- 11.45 12.30 h PL-3 Invited lecture (Rectorate-Amphitheater) Thomas Graule, Switzerland, Mysteries and pitfalls in ceramics processing
- 12.30 13.30 h Lunch
- 13.30 14.15 h PL-4 Invited lecture (Rectorate-Amphitheater) Erkka Frankberg, Finland, Quest for room temperature ductility in ceramics
- 14.15 14.45 h IL-3 Invited lecture (Rectorate-Amphitheater) Maria Canillas, Spain, Laser induced forward transfer for shaping ceramics

#### 14.45 - 16.00 h - Section 4

*Bioceramics - 1* (Rectorate-Amphitheater)

14.45 – 15.00 h	OA-31 - Shaista Ilyas, Germany
	Drug conjugates for tumor-specific localization and superior
	therapeutic action
15.00 – 16.15 h	OA-32 - Sergio Moreno-Martínez, Spain
	Fabrication of bioinspired structures for dental applications by
	indirect DLP
15.15 – 15.30 h	OC-33 - Muthusundar Kumar, Belgium
	Cold sintering process for developing hydroxyapatite ceramic
	and polymer composite
15.30 – 15.45 h	OC-34 - Miljana Mirković, Serbia
	Synthesis and characterization of cellulose-hydroxyapatite
	composite material with proper antimicrobial properties
15.45 – 16.00 h	OC-35 - Łukasz Wilk, Poland
	Composite Ni/SiO <sub>2</sub> scaffolds obtained by DIW 3D printing

## 14.45 - 16.00 h - Workshop

*Workshop on atomistic calculations in materials science* (Rectorate-Multimedia Room)

14.45 – 15.25 h	PL-5 Plenary lecture
	Ivano E. Castelli, Denmark, Computational workflows for an
	accelerated design of novel materials and interfaces
15.25 – 15.45 h	IL-11 Invited lecture
	Stevan Armaković, Serbia, Introducing fundamental
	concepts of atomistic calculations with tools of
	atomistica.online – online molecular modeling platform
15.45 – 16.00 h	Discussion

## 16.00 - 16.15 h - Coffe Break

## 16.15 - 18.30 h - Section 5

Electroceramic -	<i>1</i> (Rectorate-Amphitheater)
16.15 – 16.30 h	OA-36 - Jixi Chen, Denmark
	Post-lithiation: A way to control the ionic conductivity of
	solid-state thin film electrolyte
16.30 – 16.45 h	OC-37 - Azim Uddin, China
	Thermally stable silicone elastomer composites based on
	MoS <sub>2</sub> @biomass-derived carbon with high dielectric constant
	and ultralow loss for flexible microwave electronics
16.45 – 17.00 h	OC-38 - Ekatarin A. Didenko, Russia
	Electrical properties of nanostructured systems FeSe-CuInSe <sub>2</sub>
	and MnSe-CuInSe <sub>2</sub> under conditions of varying moisture and
	lighting
17.00 – 17.15 h	OC-39 - Buse Muslu Kop, Turkey
	Design of BaTiO <sub>3</sub> within the shapes of equiaxed, platelet and
	nanowire based flexible nano generators
17.15 – 17.30 h	OC-40 - Barbara Repič, Slovenia
	Screen printed graphite-glass composite electrodes for
15.00 15.151	detection of neonicotinoid pesticides
17.30 – 17.45 h	OA-41 - Monika Łazor, Poland
	Impact of the steel pre-oxidation on the quality of MC11- and
17.45 10.001	CMF-based protective layers for SOC interconnects
17.45 – 18.00 h	OA-42 - Rui Pinto, Portugal
	Exploring PrVO <sub>y</sub> -CaVO <sub>y</sub> oxides: Characterization and
10.00 10.151	performance in solid oxide fuel cells
18.00 – 18.15 h	OA-43 - Abdelmajid Agnaou, Morocco
18.15 – 18.30 h	Structural and electrical studies of silicon-doped $Bi_4V_2O_{11}$
18.13 - 18.30 II	OA-44 - Danica Piper, Serbia
	Processing and characterization of ultrathin epitaxial LaMnO <sub>3</sub>
	based films by chemical solution deposition

#### 16.15 - 17.30 h - Workshop

*Workshop on atomistic calculations in materials science* (Rectorate-Multimedia Room)

16.15 – 16.35 h	Hands-on molecular modeling with atomistica.online
16.35 – 16.55 h	IL-12 Invited lecture
	Dejan Zagorac, Serbia, Advanced ceramic materials:
	bridging the gap between theory and experiment
16.55 – 17.30 h	<b>Open discussion – participants' questions</b>

## 17.30 – 18.30 h – Presentation of Industry

*Presentation of Industry* (Rectorate-Multimedia Room) 17.30 – 18.30 h

20.00 h – Conference dinner - Restaurant "Fontana" Meeting of Young Ceramists Network of the European Ceramic Society -

# FRIDAY, OCTOBER 13, 2023.

#### 09.15 - 11.20 h - Section 6

Optics - 1 (TF-Blue Hall)

09.15 – 09.30 h	OA-45 - Pavlina Bancheva, Bulgaria
	Synthesis and investigating the properties of pure and doped
	ZnO thin films obtained by spray pyrolysis
09.30 – 09.45 h	OA-46 - Andrzej Kruk, Poland
	Effect of RE <sup>3+</sup> doping on the magneto-optical and luminescent
	properties of $Y_2O_3$
09.45 – 10.00 h	OA-47 - Dariia Chernomorets, Italy/Ukraine
	Solubility of ZrO <sub>2</sub> in yttrium oxide and its influence on
	transparent ceramics properties
10.00 – 10.15 h	OA-48 - Dániel A. Karajz, Hungary
	Structural possibilities of inverse opals
10.15 – 10.30 h	OA-49 - Larisa O. Fedorova, Russia
	Advanced optical ZnS and MgF <sub>2</sub> ceramics: Modification of the
	surface by carbon nanotubes

## Catalysts-1 (TF-Room 10)

09.15 – 09.30 h	OC-50 - Aicha Elaouni <u>,</u> Morocco/Hungary
	Preparation of ZnO/Bi <sub>2</sub> WO <sub>6</sub> heterostructures via surfactant-
	assisted hydrothermal method: Characterization and
	photocatalytic activity
09.30 – 09.45 h	OC-51 - Natalija Milojković, Serbia
	Photocatalytic degradation of Reactive Orange 16 dye using
	TiO <sub>2</sub> /PPy nanocomposites under simulated solar light
09.45 – 10.00 h	OA-52 - Mechouet Mourad, Algeria
	Obtaining a hybrid electrode based on imidazonium
	ionterminated and metallic nano-clusters and its catalytic
	activity toward HER
10.00 – 10.15 h	OA-53 - Jana Petrović, Serbia
	Acid treated $g-C_3N_4$ photocatalysts for the photocatalytic
	reduction of Cr(VI)
10.15 – 10.30 h	OA-54 - Irmak Su Okten, Turkey
	Preparation of Pt based hydrotalcite derived Mg(Al)O shaped
	catalysts via wet impregnation for propane dehydrogenation
	reaction

## 10.30 - 11.00 h - IL-4 Invited lecture (TF-Blue Hall)

Jan Hostaša, Italy, Transparent ceramic composites: Macro and micro, the "hows" and "what fors"

#### 11.00 - 11.15 h - Coffe Break

#### 11.15 - 12.30 h - Section 7

### Porous ceramics and membranes - 1 (TF-Blue Hall)

11.15 – 11.30 h	OA-55 - Manuel A. García-Galán, Spain
	Evaluating the mechanical integrity and reliability of multi-
	channelledflat-sheet ceramic membranes for filtration
	applications
11.30 – 11.45 h	OA-56 - Miguel Vieira, Portugal
	Hydrothermally activated ceramic membranes for oxygen
	separation
11.45 – 12.00 h	OA-57 - Radu Stefan Stirbu, Romania
	Simulation of properties of anisotropic porous ceramics based
	on 3D reconstructed microstructures
12.00 – 12.15 h	OC-58 - Zalán István Várady, Hungary
	Synthesizing SiO <sub>2</sub> -ZnO composite nanoparticles for
	application of nanofluids
12.15 – 12.30 h	OT-59 - Stjepan Šarić, Croatia
	Relationship between bentonite internal and external surface
	area and its performance in wine clarification

#### Catalysts - 2 (TF-Room 10)

11.15 11.001	
11.15 – 11.30 h	OA-60 - Vesna Miljić, Serbia
	Visible light driven photocatalytic ceramic based nano-
	composites with antibacterial activity
11.30 – 11.45 h	OA-61 - Maria M. Savanović, Serbia
	Photocatalytic performance of TiO <sub>2</sub> -coated aluminum foil for
	degradation of Rhodamine B in water
11.45–12.00 h	OA-62 - Francis Oseko, Poland
	Leveraging defects to promote dual exsolution on (Ba,La) <sub>1</sub> .
	<sub>x</sub> Ag <sub>x</sub> CoFeO <sub>6-δ</sub>
12.00 – 12.15 h	OA-63- Sara Joksovic, Serbia
	Carbon nanotubes-based thin films prepared by low-cost
	technique for biosensing applications
12.15 – 12.30 h	OA-64 - Milinko Peric, Serbia
	Synthesis of $Ti_3C_2T_x$ and its potential use in water purification
	processes
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#### 12.30 – 13.00 h – IL-5 Invited lecture (TF-Blue Hall) Martin A. Schroer, Germany, Nanostructure formation revealed by X-ray scattering methods

## 13.00 - 14.00 h - Lunch

# 14.00 – 14.30 h – IL-6 Invited lecture (TF-Blue Hall)

David Rafaja, Germany, The role of interfaces in ceramic materials

#### 14.30 - 16.00 h - Section 8

## *Electroceramics - 2* (TF-Blue Hall)

14.30 – 14.45 h	OA-65 - Ivana Goričan, Slovenia
	Energy storage properties of (1-x)Pb(Fe <sub>0.5</sub> Nb <sub>0.5</sub> )O <sub>3</sub> -xBiFeO <sub>3</sub>
	bulk ceramics and ceramic thick films
14.45 – 15.00 h	OA-66 - Touraj Karimpour, Germany
	Impact of magnetic field strength on the catalytic activity of
	chemical vapor deposition (CVD) synthesized CoFe <sub>2</sub> O <sub>4</sub> thin
	films for electrochemical oxidation of nitrogen
15.00 – 15.15 h	OA-67 - Anass Chrir, France
	Effect of post-annealing on microstructure and ferroelectric
	properties of lead-free BaTiO <sub>3</sub> thick films elaborated by
	aerosol deposition method
15.15 – 15.30 h	OA-68 - Mariam Osman, Romania
	Porosity effects on the functional properties and piezoelectric
	harvesting performances of BCTZ ceramics
15.30 – 15.45 h	OA-69 - Imane Anasser, Morocco
	Hydrothermal synthesis of SBN Aurivillius ceramics for
	ferroelectric application
15.45 – 16.00 h	OA-70 - Mihai-Alexandru Grigoroscuta, Romania
	Magneto-orientation of bulk MgB2 superconductor

Engineering and refractory ceramics - 2 (TF-Room 10)

14.30 – 14.45 h	OC-71 - Victor Zamora, Spain
	Novel boron carbide composites sintered at low temperature
14.45 – 15.00 h	OC-72 - Maria Sajdak, Poland
	Composites from the TiB <sub>2</sub> -MoSi <sub>2</sub> -C system
15.00 – 15.15 h	OC-73 - Nikhil Bhootpur, Slovenia
	Rapid pressureless sintering of cellulose nanofibre based
	ceramic matrix composites
15.15 – 15.30 h	OC-74 - Adrian Graboś, Poland
	Oxidation and thermal properties of Inconel 625 – niobium
	carbide system
15.30 – 15.45 h	OA-75 - Salomão M. da Silva Junior, Brazil
	Electroless Ni-P film deposition: Surface treatment on alumina
	and polyamide
15.45 – 16.00 h	OA-76 - Jesús López-Arenal, Spain
	An all-carbide triplex particulate ceramic composite for
	tribological applications

#### 16.00 - 16.15 h - Coffe Break

J	<b>L-7 Invited lecture</b> (TF-Blue Hall) <b>Invited lecture</b> (TF-Blue Ha
16.45 – 18.15 h – S	Section 9
<b>Bioceramics</b>	• 2 (TF-Blue Hall)
16.45 – 17.00 h	<b>OA-77 - Sumiya Iqbal, Germany</b> Tailoring silica nanocarriers to overcome hydrophobic drug
	challenges: An investigation into enhanced molecular weight and hydrophobicity modulation
17.00 – 17.15 h	<b>OA-78 - Hakim Firas Ibrahim, Germany</b> Fluorometric determination of lysozymes with aptamer
	modified silica nanoparticles
17.15 – 17.30 ł	<b>OC-79 - Slobodanka Stanojević-Nikolić, Serbia</b> Biosilica derived from agricultural and industrial waste for development of nano-silica/polymer composites for applications in various fields
17.30 – 17.45 h	A modified photo-activation protocol of a highly-filled dental composite using optical fibers
17.45 – 18.00 h	
18.00 – 18.15 h	
Coramic proc	<i>essing - 2</i> (TF-Room 10)
16.45 - 17.00 h	

16.45 – 17.00 h	OA-83 - Izabela Rutkowska, Poland
	Aluminum oxide layers deposited using pulse direct current
	electrophoretic deposition
17.00 – 17.15 h	OA-84 - Justyna Grygierek, Poland
	Sol-gel synthesis of metal-ion modified preceramic polymers
	for DLP 3D printing
17.15 – 17.30 h	OA-85 - Moussouni Hocine, Algeria
	Investigating the reactivity of surface functionalization with
	ionterminated using scanning electrochemical microscopy
	(SECM)
17.30 – 17.45 h	OA-86 - Wojciech Wieczorek, Poland
	Synthesis and structural analysis of sol-gel derived SiFeOC
	layered
17.45 – 18.00 h	OA-87 - Weronika Bulejak, Poland
	Photocurable ceramic dispersions used in the preparation of
	composite materials
	-

18.00 – 18.15 h **OA-88 - Jakub Marchewka, Poland** Preceramic polymers for the preparation of 3D silicon oxycarbide structures by digital light processing

# SATURDAY, OCTOBER 14, 2023.

09.15 – 09.45 h –	IL-8 In	nvited lecture	e (TF-Blu	e Hall)				
	Paweł	Pęczkowski,	Poland,	Degradation	of	HTS	tapes	by
	irradiati	ion with noble	gas ions ar	nd aging				

# 09.45 - 11.15 h - Section 10

### Traditional ceramics (TF-Blue Hall)

09.45 – 10.00 h	OT-89 - Tariq Labbilta, Morocco
	Eco-friendly fertilizer glasses: Controlled nutrient release for
	wheat plants
10.00 – 10.15 h	OT-90 - Ömer Furkan Ötken, Turkey
	High-temperature alkaline corrosion behaviour of [CaO, SrO,
	BaO]–Na <sub>2</sub> O–B <sub>2</sub> O <sub>3</sub> –SiO <sub>2</sub> enamel coatings on metallic
	substrates
10.15 – 10.30 h	OT-91 - Marija Kovač, Serbia
	Multi-analytical non-invasive methods as a tool for pigment
	characterization
11.30 – 10.45 h	OT-92 - Yurii Delikhovskyi, Poland
	The influence of coal fly ash additive on evolution of clay-
	cement mortars
11.45 – 11.00 h	OT-93 - Nurullah Çöpoğlu, Turkey
	Nano-copper oxide-induced surface improvements in
	(Na,Li) <sub>2</sub> O-ZnO-P <sub>2</sub> O <sub>5</sub> -B <sub>2</sub> O <sub>3</sub> -SiO <sub>2</sub> glass-ceramic coatings
11.00 – 11.15 h	OT-94 - Dunja Djukić, Serbia
	The influence of brushite-metakaolin geopolymer materials on
	phytostabilization of lead ions by Festuca rubra

## Engineering and refractory ceramics - 3 (TF-Room 10)

09.45 – 10.00 h	OC-95 - Abdelhamid Oufakir, Morocco
	Study of structural and surface changes of SiO <sub>2</sub> flint aggre-2
	gate under thermal treatment for potential valorization
10.00 – 10.15 h	OA-96 - John Wanjala, Hungary
	SIC particle size effect on cermic thermomechanical properties
10.15 – 10.30 h	OA-97 - Olga Chudinovych <u>.</u> Ukraine
	Phase equilibria in the La <sub>2</sub> O <sub>3</sub> -Lu <sub>2</sub> O <sub>3</sub> -Ho <sub>2</sub> O <sub>3</sub> system at 1500 and
	1600 °C
10.30 – 10.45 h	OC-98 - Anastasiya Kruglyak, Ukraine
	Influence of hafnium oxide on the structure and properties of
	powders and ceramics of the YSZ-HfO <sub>2</sub> composition
10.45 – 11.00 h	OA-99 - Iva Toković, Serbia
	DFT study of bulk and epitaxial LaMnO <sub>3</sub> film

#### 11.15 – 11.30 h – Coffe Break

#### 11.30 – 12.00 h – IL-8 Invited lecture (TF-Blue Hall) Henrik Haspel, Hungary, Decarbonizing the chemical industry: Ceramics in the ammonia economy

## 12.00 - 13.00 h - Section 11

#### *Ceramic processing - 3* (TF-Blue Hall)

12.00 – 12.15 h	OA-100 - Tina Tasheva, Bulgaria
	Investigation of the microstructure and magnetic properties of
	silicate glass-ceramics with high iron oxide concentration
12.15 – 12.30 h	O-101 - Sanita Ahmetović, Serbia
	Investigating the effects of Zr doping on the titanium dioxide
	nanofibers
12.30 – 12.45 h	OT-102 - Talha Doğan Özerdem, Turkey
	Comprehensive study on characterization, leaching behavior,
	and agricultural performance of glass frit as a slow-release
	fertilizer
12.45 – 13.00 h	OA-103 - Aleksandra Pavlović, Serbia
	Novel porous organosilica nanoparticles for UV protection
13.00 – 13.15 h	OC-104 - Derya Akbulut, Turkey
	Effects of pyrolysis conditions on the production of activated
	carbon from olive seeds

#### 13.15 - 13.45 h - IL-10 Invited lecture (TF-Blue Hall)

**Mtabazi G. Sahini**, **Tanzania**, Thermo-chemical stability aspects of Mixed Ionic-Electronic Conducting (MIEC) ceramic membrane materials

#### 13.45 - 14.00 h - Closing (TF-Blue Hall)

# 15<sup>th</sup> ECerS Conference for Young Scientists in Ceramics, CYSC-2023 Novi Sad, Serbia, October 11-14, 2023

PLENARY & INVITED TALKS

Plenary Lecture, PL-1

#### ORIGINS AND MECHANISTIC ASPECTS OF THE HIGH PIEZOELECTRICITY OF LEAD-BASED RELAXOR-FERROELECTRIC CERAMICS

Mojca Otonicar<sup>1</sup>, Mirela Dragomir<sup>1</sup>, Hana Ursic<sup>1</sup>, Matija Arzensek<sup>1</sup>, Urh Tos<sup>1</sup>, Sarūnas Svirskas<sup>2</sup>, Silvo Drnovsek<sup>1</sup>, <u>Tadej Rojac<sup>1</sup></u>

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The subgroup of lead-based ferroelectric perovskite materials with disordered relaxor behavior, exemplified by the  $Pb(Mg_{1/3}Nb_{2/3})O_3$ – $PbTiO_3$  (PMN–PT) solid solution, is known to exhibit exceedingly high dielectric and piezoelectric properties. This is particularly the case for Sm-doped PMN–PT ceramics, in fact, the most recent discovery revealed that Sm doping can increase the piezoelectric response of PMN–PT (piezoelectric d<sub>33</sub> coefficient ~1500 pC/N) to the highest level ever recorded for any polycrystalline material. Naturally, this has triggered at least two important questions, both touching the very nature of these chemically disordered materials. First, why are relaxor ferroelectrics so efficient and how much their relaxor nature has to do with the high efficiency, especially when compared to non-relaxor systems such as the widely used Pb(Zr,Ti)O<sub>3</sub> (PZT)? Second, what is so special about Sm dopant to boost the piezoelectric response of PMN-PT for more than 100%?

In this contribution I will present our view on these two key questions based on years of studies of undoped and Sm-doped PMN–PT. After an initial introduction to the field, I will present the results on the nonlinear piezoelectric response of PMN–PT and compare it with that of PZT to reveal the key differences between the two systems. The data obtained over large portion of the two respective phase diagrams show a clear contribution of domain walls in PMN-rich PMN–PT compositions with strongest relaxor behavior, which is distinctly different from domain-wall contributions in PZT. A clear correlation was found between this relaxor-related nonlinear response and the multiscale structure of PMN–PT as it evolves with PT content over atomic, nano- and micro-meter scale. Similarly, detailed piezoelectric and dielectric analyses as a function of Sm doping content in PMN–PT clearly show that Sm has multiple effects. No single mechanism, recently discussed in the literature, can explain the effect of Sm in enhancing the piezoelectricity of PMN–PT. The large response can only be understood by considering multiple mechanisms, such as donor dopant effects, increased local structural disorder and stabilization of tetragonal phase, all of which are induced by Sm.

Plenary Lecture, PL-2

### COMBINING REVERSE MONTE CARLO ANALYSIS OF X-RAY SCATTERING AND EXTENDED X-RAY ABSORPTION FINE STRUCTURE OF VERY SMALL NANOPARTICLES

Markus Winterer

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Although experiments investigating the atomistic structure of disordered materials are facile today through modern instruments for (X-ray) scattering (XS) and spectroscopy (EXAFS) it is still a challenge to extract the relevant information especially in case of very small nanoparticles. In our contribution we will introduce X-ray scattering and X-ray absorption spectroscopy as tools to investigate local and crystal structure of materials and present recent advances in data analysis of this inverse problem using Reverse Monte Carlo (RMC) analysis.

RMC simulations enable the analysis of XS data as well as EXAFS spectra data via partial pair distribution (pPDF) functions obtained from a physical, structural model. In case of nanoparticles and scattering data this approach suffers from the termination of the pPDF's due to the finite size of the particles. This produces artifacts in the computed

scattering intensity due to the longrange probing distance of scattering which are eliminated by using the Debye scattering equation (DSE) for computing the scattering intensity particle from а model. Computational efficiency is provided by binning the distance distribution atom pairs the DSE. of in Simultaneous refinement of XS data of and EXAFS spectra small nanoparticles are thus enabled using a mutual structural model. This method allows the self-consistent extraction of complementary information on local structure contained in EXAFS and long-range order in XS data. We describe this novel method using XS and EXAFS data of nanocrystalline LaFeO<sub>3</sub> [1] and SnO<sub>2</sub> [2] are presented.

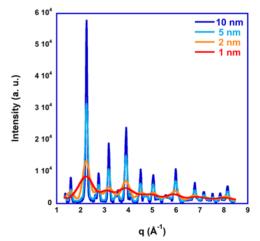


Figure 1. Computed scattering intensity for LaFeO<sub>3</sub> of different crystal diameter using the binned Debye scattering equation approach

#### 15<sup>th</sup> ECerS Conference for Young Scientists in Ceramics, CYSC-2023 Novi Sad, Serbia, October 11-14, 2023

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Plenary Lecture, PL-3

#### **MYSTERIES AND PITFALLS IN CERAMICS PROCESSING**

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Within my talk I will discuss general aspects and pitfalls in Advanced Shaping Techniques: Focusing first on powder selection: nano, submicron or micron sized powders, select the best!

Discussing then the important parameters to properly characterize powder properties: specific surface area BET, being a relevant parameter for sintering! Or could BET be a misleading number?

Dealing then with pitfalls in particle size characterization and the problem of powder segregation.

Furthermore, I will summarize state of the art knowhow how to select surfactants and who to link efficiency of surfactants with absorption and chemical composition Finally I will deal with some risk of contamination originating from impurities, supplied by the powder supplier and contamination created during powder processing, organic/polymeric binder removal and sintering steps. Plenary Lecture, PL-4

#### QUEST FOR ROOM TEMPERATURE DUCTILITY IN CERAMICS

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Ceramics, including inorganic glasses, are an integral part of the modern world, but their usefulness in engineering is limited due to their characteristic brittleness at room temperature.

As an exception to the rule, amorphous aluminum oxide  $(a-Al_2O_3)$  is a rare diatomic glassy ceramic exhibiting significant nanoscale plasticity at room temperature [1]. More recently, the room temperature plasticity of  $a-Al_2O_3$  was shown to extend to microscale and high strain rates using in situ micropillar compression [2]. Large-scale molecular dynamic simulations and finite elements simulations align with the main experimental observations and verify the plasticity mechanisms at the atomic scale and mesoscale [3]. The experimental strain rates are expanded to an order of magnitude observed in typical impact loading scenarios, such as hammer forging, with strain rates up to the order of 1 000 s<sup>-1</sup>, and the total  $a-Al_2O_3$  sample volume is expanded by 5 orders of magnitude from previous observations. Current discovery is consistent with the theoretical prediction that the plasticity mechanism observed in  $a-Al_2O_3$  can extend to macroscopic bulk glasses if the material is free of processing flaws and suggests that amorphous oxides show significant potential to be used as light, high-strength, and damage-tolerant engineering materials. Results also indicate that it is feasible to experimentally produce such flaw free samples at a bulkier scale.

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Plenary Lecture, PL-5

# COMPUTATIONAL WORKFLOWS FOR AN ACCELERATED DESIGN OF NOVEL MATERIALS AND INTERFACES

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The development of automated computational tools is required to accelerate the discovery of new functional materials, to speed up the transition to a sustainable future. Here, I address this topic by designing new electrodes with controlled interfaces for different applications which accelerate the transition to a sustainable future. These workflows are implemented in the framework of Density Functional Theory, using MyQueue and the Atomistic Simulation Environment (ASE). In the first part, I describe a fully autonomous workflow, which identifies materials to be used as intercalation electrodes in batteries, based on thermodynamic and kinetic descriptors like adsorption energies and diffusion barriers [1]. A substantial acceleration for the calculations of the kinetic properties has been obtained due to a recent implementation of the Nudged Elastic Bands (NEB) method, which takes into consideration the symmetries of the system to reduce the number of images to calculate. Moreover, we have established a surrogate model to identify the transition states, which can further reduce the computational cost to at least one order of magnitude [2,3]. We have applied this workflow to discover new cathode materials for Mg batteries as well as solid state electrolytes for Li, Na, and Mg all-solid-state batteries [1,3]. In the second part of my talk, I discuss how engineering the interface can positively impact surface properties. I show this concept using two examples. In the first one, I use nanostructure materials to increase the Li-storage capacity in C-anodes or to adjust the change in volume during charge/discharge in Si-anodes for Li-ion batteries [4]. In the second example, I apply strain engineering and external stimuli to switch material's polarization to decrease the reaction overpotential in oxynitride materials for the oxygen evolution reaction [5, 6].

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Invited Lecture, IL-1

# PROCESSING OF SUPER-HARD CERAMICS BASED B<sub>4</sub>C AND TiCN AT LOW TEMPERATURES

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The feasibility of manufacturing super-hard  $B_4C$  and TiCN by spark plasma sintering (SPS) with metal disilicides (MeSi<sub>2</sub>) and silicon (Si) aids, respectively, at low temperatures is explored. It is shown that both MeSi<sub>2</sub> and Si and act as reactive sintering additives that promote densification by transient liquid phase sintering. Specifically, MeSi<sub>2</sub> reacts with  $B_4C$  forming *in-situ* SiC, the corresponding metal diboride (MeB<sub>2</sub>), and Si, that melts at ~1411 °C and later forms more SiC. The densifiability is studied as a function of the increasing proportion of MeSi<sub>2</sub> aids, demonstrating that a broad spectrum of full-dense  $B_4C$ -MeB<sub>2</sub>-SiC composites can be obtained at increasingly lower SPS temperatures. TiCN is also fully densifiable at smoother SPS temperature with small amounts of Si aids, leading to the *in situ* formation of SiC, TiSi<sub>2</sub> and N-rich TiCN second phases dispersed in the TiCN matrix. Finally, it is shown that these multiparticulate  $B_4C$ and TiCN composites have fine-grained microstructures and that they are superhard and tougher than monolithic  $B_4C$  and TiCN ceramics, respectively, which together with their easier fabricability makes them very appealing for structural applications.

Invited Lecture, IL-2

# IONIC MOLECULAR SYSTEMS – NEXT GENERATION "CERAMICS" FOR ELECTRONIC AND THERMAL ENERGY STORAGE APPLICATIONS

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Ionic molecular systems, also called molecular ceramics, are materials where covalently bonded molecular components are bound together into a crystalline lattice by weaker bonding interactions like electrostatic, hydrogen and halogen bonding. The use

of molecules instead of single element ions means that molecular size, charge and shape can be used to engineer material properties, while the weak bonding interactions give rise to many mechanical and functional properties that allow these materials to fill intermediate parameter spaces between existing materials. These materials can possess useful functional behaviors like ferro and piezoelectricity and often possess a high entropy solid-solid phase transitions that are useful for storing thermal energy.

The goal of our work has been to understand what aspects of these materials are critical to their functional and phase transition properties and how they can be engineered. We have included computational investigations to understand the role of molecular rotation in piezoelectric responses and begun to explore the use and limitations of solid solution formation to engineer functional properties and phase transition properties. The solid solution approach is a corner stone of ceramic engineering and has shown to be effective for altering transition temperatures in molecular ceramics, while having a yet to be understood role on the functional properties. We focus on the use of in sit u X-ray diffraction techniques as a means of studying structural changes as a function of composition and temperature to successfully map out the phase diagrams of some novel material systems based on quaternary ammonium cations together with iron tetrahalide anions.

Invited Lecture, IL-3

#### LASER INDUCED FORWARD TRASFER FOR SHAPING CERAMICS

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Laser Induced Forward Transfer (LIFT) is a direct writing technique able to print a wide range of materials such as metallic and ceramics. The main advantage of the technique, if compared with other mature printing technologies like inkjet or extrusion, is the capacity to transfer material in an enormous range of viscosities and rheological behavior, from solid-state to low-density inks. It allows to work with inks characterized by a high solid content and for hence higher densities after densification. Most LIFT approaches use a pulsed laser beam to transfer material from a donor substrate to an acceptor surface. The donor substrate consists of a transparent substrate with a thin layer of ink previously deposited onto the surface. The laser energy irradiates the interface between the transparent substrate and the material to be transferred. Conversion of laser energy into kinetic energy of part of the material allows the transfer of small volumes

with high spatial accuracy, providing a printing method with spatial resolution in the order of 1  $\mu$ m. The dynamics of the process are strongly dependent on the physical properties and thickness of the material, the gap between acceptor and donor substrate, the configuration of the donor substrate, and the parameters used in the laser irradiation process such us energy pulse and scan speed.

This technology commonly used in 2D is been developed to the 3D. In the last years, 3D structures of polymeric ceramics, has been developed for medical implants also with bioprinting perspectives. In addition, the bioprinting by laser shows interesting benefits for cell and has open new applications.

Another interesting point is the possibility to combine a LIFT with other laser devices, for example, for Selective Laser Sintering (SLS). In the recent years, it has allowed the 3D printing of multimaterial structures. It is a challenge in the spotlight of the 3D technologies, especially combining ceramics with metallic materials.

Invited Lecture, IL-4

## TRANSPARENT CERAMIC COMPOSITES - MACRO AND MICRO, THE "HOWS" AND "WHAT FORS"

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Transparent ceramics stand as cutting-edge class of materials that benefit from the shaping possibilities of ceramic technology and from the crystalline structure that offers superior performance compared to glasses. Transparency may be obtained only when the material is free of defects that scatter light, viz. pores or secondary phases. Mostly, this means a requirement of a fully dense, single-phase, defect-free microstructure. However, the impact of small scatterers on transparency diminishes as their size decreases, allowing the development of multiphase materials, composites, that are transparent in the IR and even in the visible range for nanometric grain sizes. Conversely, another important topic in the field of transparent ceramics are macroscopic composites. The increasing optical quality of transparent ceramics in the past years has ignited a growing interest, especially in optics and photonics [1]. Transparent ceramics stand as counterparts to more traditionally used single crystals, which may have the same composition, but are obtained by different processes, mostly based on growth from melt. This process is time- and energy-consuming, and above all imposes significant limitations on the final shape of the components, which is obtained by machining. Transparent ceramics, in contrast, take advantage of the shaping flexibility of ceramic processing, in particular to produce composite or gradient structures without the need of

post-processing and bonding [2]. Unlike the nanocomposites mentioned above, these composites are macroscopic, mostly with relatively small differences in chemical composition among the different parts. In the case of simple shapes and planar interfaces such structures may be obtained by diffusion bonding of polished single crystals, but the process is demanding and expensive. Ceramic processing allows us to shape such structures in the green state with a high degree of freedom, avoiding intermediate cutting, polishing and bonding steps. The aim of the presentation is to illustrate the possibilities and benefits of transparent ceramics with a particular emphasis on multimaterial components, spanning both nano- and macro-composites. The application potential will be illustrated on specific examples involving structures for lasers or protective domes and the shaping possibilities will be discussed.

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Invited Lecture, IL-5

#### THE ROLE OF INTERFACES IN CERAMIC MATERIALS

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Internal interfaces between different compounds, individual phases and/or neighboring grains play always an important role in the design of bulk ceramic materials and thin films, because these microstructure features can help in adjusting and enhancing the intrinsic properties of the functional materials. Epitaxial growth at the interfaces improves adhesion of the counterparts. Thus, it can be used to control the lattice strains and crystal structure defects. In this talk, the capabilities of the materials design via 'interface engineering' will be illustrated on the examples of hard nanocomposites, multifunctional filters for the metal melt filtration and AlGaN multilayer stacks for optoelectronic applications.

The part of the talk devoted to the hard nanocomposites will comprise transition metal nitride coatings supersaturated with aluminum nitride, (TM,Al)N, and bulk boron nitride. For supersaturated (TM,Al)N with TM = Ti, Cr, Zr, it will be shown how the internal interfaces can be utilized to inhibit the decomposition of the oversaturated solid solution and to hinder the phase transition of metastable cubic aluminum nitride to the thermodynamically stable wurtzitic AlN. The bulk BN nanocomposites were selected to show how the internal interfaces can enhance the hardness of hard nanocomposites and to illustrate the role of the crystal structure defects like stacking faults in the phase

transformation process from h-BN to c-BN. The benefits of the hetero-epitaxial growth for a better adhesion of the counterparts will be illustrated on the functionalized corundum-based filters developed for the metal melt filtration and on the metallic coatings that were deposited by cold-gas spraying on ceramic substrates. The capabilities of the defect engineering at the internal interfaces will be demonstrated on the AlGaN multilayer stacks, in which a tailored composition gradient was used to control the lattice strain and to affect the defect density. Presented results are based on the results of the microstructure analyses using X-ray and electron diffraction, X-ray and electron spectroscopy, transmission electron microscopy, on the hardness and adhesion measurements and on the micromechanical modelling.

Invited Lecture, IL-6

# NANOSTRUCTURE FORMATION REVEALD BY X-RAY SCATTERING METHODS

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In order to obtain a full understanding of the structure of nano-systems and their evolution, it is essential to determine it at all relevant length scales – ideally following formation and transition processes under *in situ* and *operando* conditions. A method that fulfills these requirements is small angle X-ray scattering (SAXS). SAXS does not only yield access into lengths scales from several Ångstrom up to hundreds of nanometers, but also allows probing the structural evolution by time-resolved studies under realistic formation conditions [1].

I will demonstrate how SAXS can be used as a tool to explore the structure and structural evolution of various types of nano-systems (Fig. 1). I will give examples how to probe structure formation and transitions in hard, soft and biological matter for different aggregation states, highlighting the broad versatility of small angle scattering.

In particular, I will show how to follow nanoparticle formation within chemical vapor synthesis reactions [2], employing the high potential of modern synchrotron radiation facilities, as well as how to determine the structure on nanoparticle composites.

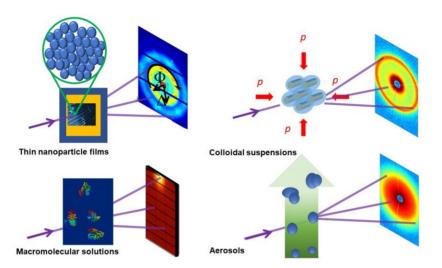


Figure 1. The wide range of applications for SAXS to study nanostructures at modern synchrotron facilities. Clockwise: Investigation of thin granular films using X-ray nanobeams, colloidal suspensions under high pressure, following nanoparticle formation in aerosols, and macromolecular solutions of proteins in solution.

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Invited Lecture, IL-7

# DEVELOPMENT AND CHARACTERIZATION OF COMPOSITES FOR BONE TISSUE ENGINEERING WITH THE AID OF BIOMIMETIC BIOREACTORS

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Developing new biomaterials for bone tissue engineering is of vital importance due to the increasing demand for bone transplants resulting from various causes, such as trauma, tumors, infection, and genetic conditions. To address this persistent need, biomaterials for bone tissue engineering must fulfil several critical requirements, such as mimicking the hierarchical structure of complex bone tissue composed of organic and inorganic components, possessing suitable mechanical properties, pore size, and porosity, as well as an appropriate degradation rate.

Our research group has successfully developed two promising biomaterials for bone tissue engineering, utilizing a combination of bioactive glass (BAG) or  $\beta$ -tricalcium phosphate ( $\beta$ -TCP) - undoped or doped with Mg<sup>2+</sup>, and different natural polymers, such as gellan gum (GG) and alginate (ALG). Obtained scaffolds were assessed in terms of porosity and pore size distribution, and mechanical properties under physiological levels of mechanical compression (337.5 µm/s loading rate, 10% strain) using a dynamic compression bioreactor coupled with medium perfusion over a 14-day period. The mechanical properties of the  $\beta$ -TCP-ALG scaffolds were influenced by the composition of calcium phosphate fillers, with improvements observed as Ca<sup>2+</sup> concentration increased while Mg<sup>2+</sup> concentration decreased. These properties were significantly higher compared to the mechanical properties of BAG-GG scaffolds.

Bioactivity of the scaffolds was evaluated under static and biomimetic conditions in the perfusion bioreactor, with a continuous flow of simulated body fluid (SBF) at a superficial velocity of 400  $\mu$ m/s. The formation of hydroxyapatite (HAp) within the BAG-based scaffolds was studied for 14 days, while for  $\beta$ -TCP-based scaffolds, the studies continued for 28 days. The results revealed a remarkable increase in HAp formation under bioreactor conditions compared to static controls in both types of scaffolds, with HAp crystals exhibiting a more uniform distribution and a distinctive cauliflower-like morphology throughout the scaffolds. However, bioactivity was more profound for the BAG-GG scaffolds compared to  $\beta$ -TCP-ALG scaffolds due to additional gelation of alginate around the incorporated fillers.

Physiologically relevant characterization with the aid of biomimetic bioreactors has unveiled the promising potential of novel composite scaffolds for bone tissue engineering, while also highlighting the importance of carefully balancing different scaffold characteristics, such as mechanical strength and bioactivity, to develop optimal scaffolds tailored for specific applications. Invited Lecture, IL-8

# DEGRADATION OFHTS TAPES BY IRRADIATION WITH NOBLE GAS IONS AND AGING

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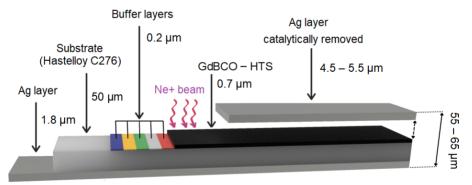
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The irradiation effect with noble gas ions on high-temperature superconducting tapes 2G HTS SF 12050 (based on GdBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub>, where  $\delta$  is the oxygen factor) will be discussed. HTS tapes are already utilized in devices operating under extreme radiation conditions, and there is also the opportunity of employing them in the space industry. In deep space, the HTS tapes covering various cosmic equipment could be exposed to radiation types, mainly protons ((90% of particles) [1],  $\alpha$  particles - He atom (9% of particles) [2], and other element ions, including noble gases: Ne<sup>+</sup> [3], Ar<sup>+</sup> [4], Kr<sup>+</sup> [4] or Xe<sup>+</sup> [4]. Ionizing radiation, including heavy ion bombardment, may involve different defects (e.g., Schottky, Frenkel) in the tape microstructure, leading to superconductivity deterioration.

A silver protective layer of 4.5–5.5  $\mu$ m was removed in a catalytic process to determine the Ne ion fluence effect on GdBCO-based tape superconductivity with a critical current density ( $J_c$ ) of 3.7 MAcm<sup>-2</sup> at 77 K (Fig. 1).



*Figure 1. The cross-section of the 2G HTS SF 12050 superconducting tape by SuperPower Inc.* [5]. *The superconducting GdBCO layer was irradiated with Ne<sup>+</sup> ions after the Ag layer removal.* 

Then, Ne<sup>+</sup> ions with energy of 250 keV and different fluences of  $10^{12}$ ,  $5 \cdot 10^{12}$ ,  $10^{13}$ , and  $10^{14}$  Ne<sup>+</sup>/cm<sup>2</sup> were uniformly implanted [3]. Structural, magnetic, and electric

studies of irradiated tapes confirm that superconductivity deterioration retains a microscopic origin, mainly due to oxygen deficiency and microstructural defects (Fig. 2).

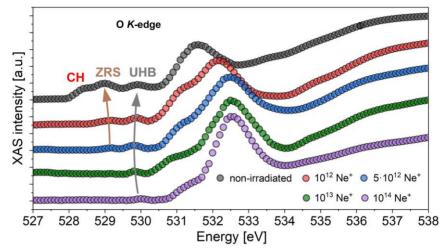


Figure 2. The X-ray absorption spectra (XAS) for the GdBCO 2G HTS tapes: O K-edge. CH – chain holes; ZRS – Zhang-Rice singlet; UHB – Upper Hubbard band. Measurement data were obtained during the experiment on the PIRX (former PEEM/XAS) beamline at the Solaris Synchrotron Radiation Center in Kraków, Poland

Critical current densities ( $J_c$ ) were reduced by 33% – 60% (depending on the external magnetic field), and weakening in critical temperature ( $T_c$ ) of 1.5–2.0 K for irradiated tapes ( $T_c$  for tape non-irradiated 93.5 K).

The last part of the presentation will also discuss the effects related to the aging of HTS-2G SF 12050 tapes based on GdBCO.

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Invited Lecture, IL-9

# DECARBONIZING THE CHEMICAL INDUSTRY – CERAMICS IN THE AMMONIA ECONOMY

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Mitigating the effect of carbon dioxide in the global climate change requires the reduction of its atmospheric concentration by transforming carbon-intensive industries "green". The so-called "green" hydrogen, i.e., hydrogen produced in a carbon-neutral way, allows the significant reduction of the ecological footprint of ammonia, fertilizer, and steel production, to name a few. Hydrogen generated immense expectations for realizing a carbon-free economy. However, issues related to storage, distribution, and the lack of infrastructure delay its full implementation. Ammonia, on the other hand, could serve as an efficient energy vector, and means of transporting hydrogen, possessing high energy density with an existing infrastructure capable of overcoming hydrogen's key drawbacks. Current ammonia production is, however, based on the energy- and carbonintensive Haber-Bosch process, where hydrogen is generated by steam-methane reforming (SMR) and water-gas shift reaction (WGSR) and then purified for highpressure ammonia synthesis. Renewable ammonia could be produced in the future based on the current Haber-Bosch process (a) with a subsequent  $CO_2$  sequestration or (b) using renewable sources of hydrogen. Nevertheless, the traditional technology could be revolutionized by the direct electrochemical conversion of N-sources to  $NH_3$ , closing the nitrogen cycle by employing selective catalytic oxidation of ammonia (NH<sub>3</sub>-SCO) to NO<sub>x</sub> compounds or N<sub>2</sub>. In this lecture, following a short overview of "Ammonia Economy", the potential roles of ceramics at different levels of this concept will be covered to envisage the route to the transition of ammonia as the next sustainable energy vector.

Invited Lecture, IL-10

# THERMO-CHEMICAL STABILITZ ASPECTS OF MIXED IONIC-ELECTRONIC CONDUCTING (MIEC) CERAMIC MEMBRANE MATERIALS

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Cubic perovskite Ba<sub>x</sub>Sr<sub>1-x</sub>Co<sub>y</sub>Fe<sub>1-y</sub>O<sub>3- $\delta$ </sub> (BSCF), with x = 0.5 and y = 0.8, is one of the oxygen permeable mixed ionic-electronic conducting (MIEC) membrane materials having the highest oxygen permeation flux reported. The material has potential for hightemperature electrochemical applications such as oxygen separation membrane and cathode for Solid Oxide Fuel Cells (SOFCs). However, its thermochemical instability is of great concern. BSCF exhibits a non-linear expansion with temperature and oxygen partial pressure,  $pO_2$  due to oxidation, spin transition of cobalt, thermal reduction and precipitation of hexagonal phase. While its cubic perovskite structure is thermodynamically stable above 850 °C, the material is susceptible to degradation by high-temperature creep and kinetic demixing/decomposition. At intermediate temperatures below 650 °C, oxygen permeation flux is relatively low. At temperatures above 650 °C, precipitation of the hexagonal phase affects the oxygen permeation flux exhibited by the cubic perovskite phase. The need for stabilization of the cubic perovskite phase by doping/co-doping can therefore not be overstated. Cation interdiffusion and interface reactions are also of great concern for application in solid oxide fuel cells. Studies indicate that cation interdiffusion and interface reactions occur both during the high temperature sintering and during SOFC operations. Therefore, its occurrence cannot be completed ruled out even for intermediate temperature SOFCs. Clear evidence on the occurrence of cation interdiffusion and interface reactions was demonstrated using cobalt oxide (CoO) and La<sub>2</sub>NiO<sub>4+ $\delta}$ </sub> as diffusion couples. Methods that can be used to control the occurrence of cation interdiffusion and subsequent interface reactions are discussed.

DFT WORKSHOP

# Workshop on atomistic calculations in materials science Chair: Stevan Armaković

We are thrilled to announce that this year's the Conference CYSC-2023 (The 15<sup>th</sup> ECerS Conference for Young Scientists in Ceramics) will host a captivating workshop dedicated to exploring the versatile applications of atomistic calculations in materials science. This satellite event of CYSC-2023 is thoughtfully designed to introduce fundamental computational methods that are accessible to beginners in this field. The main goal of this event is to introduce beginners to fundamental concepts of materials modeling with atomistic calculations and to present examples that effectively showcase what can be done with DFT calculations.

In the dynamic landscape of materials science, computational methods have emerged as powerful tools, revolutionizing the way we approach materials design and innovation. Through techniques like quantum mechanical calculations and molecular dynamics simulations, researchers can gain a deeper understanding at the atomic level, predicting vital properties of structures that have yet to be synthesized in the lab.

Computational modeling with atomistic calculations empowers scientists to make informed decisions and optimize their experimental endeavors. Researchers can strategically allocate their resources by sifting through countless possibilities and identifying the most promising candidate structures, sparing both time and expenses associated with exhaustive laboratory experiments.

Join us at CYSC-2023 for this enlightening workshop, where you can meet with atomistic calculations. Whether you are just beginning your exploration or seeking to deepen your understanding, our speakers, who are experts in the field, will guide you through the exciting world of atomistic calculations and their transformative impact on materials research.

	PL-5 Plenary lecture
14.45 – 15.25 h	Ivano E. Castelli, Denmark, Computational workflows for an
	accelerated design of novel materials and interfaces
	IL-11 Invited lecture
15.25 – 15.45 h	Dejan Zagorac, Serbia, Advanced ceramic materials:
	Bridging the gap between theory and experiment
15.45 – 16.00 h	Discussion
16.00 – 16.15 h	Coffe Break
	IL-12 Invited lecture
16.15 – 16.35 h	Stevan Armaković, Serbia, Introducing fundamental
10.15 – 10.55 h	concepts of atomistic calculations with tools of
	atomistica.online - online molecular modeling platform
16.35 – 16.55 h	Hands-on molecular modeling with atomistica.online
16.55 – 17.30 h	Open discussion – participants' questions
$10.55 - 17.50 \mathrm{II}$	Open discussion – participants questions

**ORAL PRESENTATIONS** 

# COMPATARIVE ANALYSIS OF Batio<sub>3</sub> NANOCERAMICS DERIVED FROM CUBOIDAL AND EQUIAXED NANOPARTICLES

<u>Vlad-Alexandru Lukacs</u><sup>1</sup>, Radu Stirbu<sup>1</sup>, Lavinia Curecheriu<sup>1</sup>, Oana Condurache<sup>2</sup>, Maria Teresa Buscaglia<sup>3</sup>, Gabriel Caruntu<sup>4</sup>, Liliana Mitoseriu<sup>1</sup>

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 $BaTiO_3$  ceramics produced from nanopowders with similar particle size, having two different morphologies (cubic-like and spherical) have been sintered in two steps and their functional properties have been comparatively analysed. In terms of the structural composition and nanoscale characteristics of both the starting powders and the sintered ceramics, there were no discernible variations between the two types of ceramics.

The initial morphology of the starting powders is lost in the final ceramics, higher densities being reached in the ceramics originating from cuboidal nanoparticles. Thus, the assembly of nanoparticles during the pressing process is used to describe the main distinction between the two types of ceramics. The simulation of the cubic and spherical nanoparticles compacting led to the derivation and comparison of the resulting green bodies' densities and porosity distribution. In cuboid-originated ceramics, higher permittivity, polarization, and tunability are observed as a result of improved densification following sintering. In contrast, the spherical particle-derived ceramics have less defects than the cuboid-originated counterparts, due to a more uniform and finer porosity distribution within the ceramic volume and as a result, despite having lower material constants, these ceramics are more stable and can withstand the application of larger fields.

Acknowledgment: This work was supported by the Romanian Research Grant PN-III-P1-1.1-PD-2021-0531.

OC-2

# INVESTIGATION OF CNT/OXIDE COMPOSITES IN THE APPLICATION OF NANOFLUIDS

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Nowadays efficient energy utilization is an urgent problem. During heat transfer operations (e.g., heat exchangers or solar collectors) it is a major concern to reduce heat loss and maximize efficiency. One way to do so is the utilization of the so called "nanofluids".

Nanofluids are engineered work fluids with a continuous phase (base fluid) of a conventional work fluid e.g., water, ethylene-glycol or mineral oils and a dispersed phase of some kind of nanoparticle. These nanofluids bear the property of increased thermal conductivity compared to the base fluid as the nanoparticles have several orders of magnitude higher thermal conductivity than any base fluid [1].

Nanofluids can be grouped by the nanoparticles used to create them: metal-, metal-oxide- and carbon-based nanofluids are the most common ones. Carbon has lots of different modifications with different properties, which can be exploited for different roles. For example, carbon nanotubes have an astonishing longitudinal thermal conductivity with 3000 W/mK. On the contrary carbon nanotubes are extremely hydrophobic, which creates quite a challenge to make stable water-based nanofluids out of them. The use of water is critical, as it has two-three times higher thermal conductivity than ethylene glycol or mineral oils and a lot cheaper in an economical point of view [2].

Our research aims to study the effect of oxide coating on carbon nanotubes to create stable water based nanofluids. We created stable nanofluids with and without surfactants in 150, 500 and 1000 ppm(V) concentrations. The nanofluids zeta potential and particle size were analysed to determine stability. We also measured the nanofluids thermal conductivity and viscosity. The composite nanoparticles were characterized using Fourier-transformation infrared spectroscopy, X-ray powder diffraction, electron microscopy and thermogravimetry/differential thermos analysis coupled with mass spectroscopy.

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# DEVELOPMENT OF CERAMIC SUSPENSIONS FOR LITHOGRAPHY BASED CERAMICS MANUFACTURING (LCM)

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This abstract focuses on developing photosensitive ceramic-based suspensions for one of the additive manufacturing methods – lithography-based ceramics manufacturing (LCM) called Digital Light Processing (DLP). In our case, the photosensitive suspension is irradiated by a blue light source with a wavelength of 465 nm. It involves selective polymerization of a photopolymer matrix with incorporated ceramics particles.

The present phase of development is focused on aluminium oxide-based suspension. The polymeric matrix of suspension is a mixture of several types of bi- and multifunctional monomers. The photoinitiator system comprised camphorquinone (CQ – photoiniciator) and 2-dimethylamino-ethyl methacrylate (DMAB – co-inciator). This composition was loaded with aluminium oxide ( $Al_2O_3$  – ceramic powder with a specific surface area of 11,85 m<sup>2</sup>/g, partice size = 200 nm) up to solid loading 81 wt.%. The relative proportions of ceramic powder, dispersant and prepolymer mixture were optimized with regard to the final defect-free dense ceramic structure.

Prepared photosensitive suspensions were printed and sintered. The microstructure of the sintered objects were characterized and evaluated, focusing on the presence of defects specific to the debinding process. Prepared suspensions are suitable for 3D printing using the LCM method, and the knowledge gained from developing this photosensitive suspension may pave the way for the future development of suspensions based on different materials or it's combination. That is promising for future advancements and innovations in various applications including bone tissue engineering.

# HIGH-ENTROPY SPINEL OXIDES: FUNDAMENTALS, SYNTHESIS AND CHARACTERIZATION

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High-entropy spinel oxides (HESOs) are oxides with 5 or more cations with the general formula AB<sub>2</sub>O<sub>4</sub> and the spinel ( $Fd\overline{3}m$ ) structure. Due to their unique structure and properties, HESOs have shown great potential in various technological applications, i.e. they can be used as catalysts, adsorbents and photocatalysts. Since the first successful synthesis in 2018, researchers have been experimenting with different precursors and synthesis methods. However, further research is still needed in order to fully understand their capabilities and exploit their properties. The aim of this research is to synthesize novel HESOs using the self-propagating room temperature (SPRT) method, which is time and cost-effective and has not been utilized so far. Our results indicate that chlorides are not good precursors, since the formation of spinel structure has not been achieved. On the other hand, the use of nitrates has successfully led to a primary spinel ( $Fd\overline{3}m$ ) phase. Still, certain issues persist, as secondary phases are commonly formed, especially in the presence of Mg or Cu. A potential solution that could ensure the formation of a single phase is the use of quenching from high temperatures (1000 °C), instead of gradual cooling.

# DEVELOPMENT OF POROUS HYPER-STOICHIOMETRIC LITHIUM TITANATE (Li<sub>2</sub>TiO<sub>3</sub>) FOR TRITIUN BREEDER APPLICATION

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Lithium titanate  $(Li_2TiO_3)$  is one of the prominent materials for the tritium breeding application in nuclear fusion reactors because of its optimum properties like lithium atom density, tritium release rate at low temperatures range, low activation characteristics, low thermal expansion coefficient and high thermal conductivity.

In a fusion reactor, solid breeder material is envisioned to be utilized in the pebble bed configuration. The major merits of pebble bed configuration include ease of filling breeder material and optimisation of packing density of the bed with controlled size fraction of breeder pebbles. However, this configuration poses several limitations like pebble movement, fracture or fragmentation during operation, sintering, and low thermal conductance at breeder wall interface. Engineered porous breeder configuration with interconnected porosity can offer better temperature distribution, tritium release, mechanical strength and lifetime of the breeder.

In the present investigation, ceramic foams of lithium titanate material were synthesized for the breeder application through sponge replication methodology that exhibits high porosity with interconnected pores. Compared to stoichiometric lithium titanate ( $Li_2TiO_3$ ), hyper-stoichiometric lithium titanate is reported to perform better due to excess lithium in the structure. To evaluate the effect of excess lithium, hyper stochiometric porous breeder was synthesised and characterized. The improvement of functional properties of hyper-stoichiometric lithium titanate foam as compared to stoichiometric composition shall be discussed in the presentation.

Keywords: lithium titanate, breeder, porous ceramic, hyper-stoichiometry, Li<sub>2</sub>TiO<sub>3</sub>

# CRITICAL PARAMETERS FOR GROWTH OF ORIENTED ZnO NANOWIRE ARRAYS DURING HYDROTHERMAL SYNTHESIS

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Traditional batteries used to meet the energy needs of wearable devices, which have become an integral part of our lives as a result of technological developments in recent years, pose a problem both in terms of user comfort and environmental effects. For this reason, the interest in renewable and environmentally friendly energy harvesting systems is increasing. Lead-free piezoelectric nanogenerators (PENGs) offer an innovative approach to converting mechanical energy into electrical energy at the nanoscale level for meet the demand in the wearable devices. Zinc oxide (ZnO) with wurtzite crystal structure is widely preferred in lead-free systems due to its low cost, chemical stability, wide band gap semiconductor and piezoelectric properties.

In previous studies, it has been observed that ZnO is widely used in the piezoelectric layer in plate and thin film morphologies. Flexibility and comfort are very important factors in wearable systems. The design of innovative materials is needed to achieve higher piezoelectric potential with lower forces and improved flexibility. Nanowire morphology is seen as an innovative candidate to meet this need. Many methods are used to produce ZnO with nanowire morphology. However, since the hydrothermal synthesis method is cost-effective, easy to manufacture and environmentally friendly, it is much more preferred.

The research objective of this study was to investigate the effects of hydrothermal synthesis parameters on ZnO nanowire structures, grown on flexible ITO coated PET substrates. The effects of seed layer, reaction time and solution concentration were investigated as synthesis parameters. The morphology of ZnO nanowires produced under different synthesis conditions was analyzed by X-ray diffraction (XRD) and scanning electron microscopy (SEM). It was determined that the seed layer, reaction time and solution concentration have a critical role on the production of oriented ZnO nanowires (in the c-axis).

# TUNING THE PROPERTIES OF THE MAGNETOSTRICTIVE COBALT FERRITE – A PROMISSING CANDIDATE FOR WIRELESS NEURAL STIMULATION APPLICATION

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Neural stimulators can be used for the management of neurological disorders such as Parkinson's disease via electrical stimulation of neurons. However, implanted neural devices are large, complex, and invasive. Instead, neural devices that are nanoscale, injectable, and wireless can provide a less invasive approach to neurostimulation, but wireless power transfer at the nanoscale is a challenge.

Magnetoelectric nanoparticles (MENPs) enable wireless power transfer to the brain via conversion of an applied magnetic field into an electric field. These ferromagnetic MENPs constitute a piezoelectric component and magnetostrictive component which are coupled by a common interface to generate magnetic-toelectric coupling via strain translation.

Tuning the properties of the magnetostrictive component enables tuning of the translated strain to the piezoelectric, and therefore controls the output signal of these nanoelectrodes.

Here we show the synthesis of the magnetostrictive core via a coprecipitation method, where the properties have been tuned by varying three synthesis parameters: the concentration of the coprecipitation agent, and the time and temperature.

XRD measurements confirmed that cobalt ferrite crystallized in the cubic spinel structure. Higher calcination time gave rise to bigger grains as expected. VSM measurements showed that the coercivity and magnetic saturation could be tuned by varying the synthesis parameters. Additionally, the change in the magnetic properties leads to a change in the magnetoelectric properties.

Wirelessly powered and injectable MENPs are a promising technology that could one day provide a less invasive approach to the treatment of neurological disorders. This work shows that we can use basic chemistry to control the magnetostrictive, and thereby magnetoelectric, properties of these materials. The methods discussed herein demonstrate a facile approach to tune the signal-response of wireless nanoelectrodes.

# OPTIMIZATION AND BIOACTIVITY EVALUATION OF SILICA-DOPED HYDROXYAPATITE SCAFFOLDS FOR BONE TISSUE ENGINEERING – A DIRECT FOAMING APPROASH

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Bone tissue engineering aims to develop biocompatible scaffolds for efficient bone regeneration. Scaffolds made from hydroxyapatite (HA), a biocompatible and bioactive ceramic resembling natural bone, provide a favorable environment for cell attachment, proliferation, and differentiation. Incorporating silica into these scaffolds enhances their bioactivity, mechanical properties, degradation control, cell response and antibacterial properties. In our study, we utilized the direct foaming method to fabricate scaffolds using HA powder, including HA powder doped with silica. Our objective was to optimize the method's parameters (pressure, foaming agents, concentrations) to achieve well-defined scaffolds with pore sizes larger than 100  $\mu$ m, porosity exceeding 70% and open interconnectivity. To evaluate the scaffolds' characteristics, we employed techniques such as X-ray diffraction, scanning electron microscopy, Raman spectroscopy and confocal laser scanning microscopy for structural and physical characterization. Mercury intrusion porosimetry and water intrusion based on the Archimedes principle were utilized to assess pore size distribution, open interconne-ctivity and porosity. By optimizing the direct foaming method, we achieved significant improvements in pore size and porosity. Silica-doped scaffolds displayed larger pore sizes, up to 300 µm, compared to HA scaffolds. This increased pore size facilitates cell infiltration, migration and attachment, creating a favorable environment for tissue regeneration. Moreover, the optimized scaffolds exhibited porosity of up to 80%, indicating a high volume fraction of interconnected pores. The wettability of HA and HA-silica doped scaffolds was evaluated by measuring the contact angle of liquid droplets on the scaffold surface. Silicadoped scaffolds demonstrated improved wettability compared to undoped scaffolds in both water and diiodomethane, indicating enhanced interaction with liquids and potential benefits for cell adhesion and proliferation. We also used simulated body fluid (SBF) and degradation testing to assess the bioactivity of the scaffolds. SBF testing determined scaffold ability to induce hydroxyapatite layer formation, a crucial indicator of bioactivity. Degradation testing monitored changes in scaffold mass and structural integrity provides insights into degradation behavior and biocompatibility. The results revealed that the silica-doped scaffolds exhibited superior bioactivity compared to the undoped scaffolds. This highlights the positive impact of silica incorporation on the scaffolds' bioactive properties and their potential for bone tissue engineering applications.

# COMBINING FREEZE CASTING WITH PRESSURE-LESS SPARK PLASMA SINTERING FOR THE MANUFACTURING OF BULK ULTRA-HIGH TEMPERATURE CERAMICS

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This work explores the manufacturing by freeze casting of ultra-high-temperature ceramic (UHTC) materials. The method is used in combination with pressureless Spark Plasma Sintering (SPS) as a means for producing complex UHTC 3D parts containing internal channels to be used as heat exchangers. Ceramic powders were mixed and stably suspended in water and, subsequently, used to produce the desired complex green parts by casting the suspension into suitable moulds.

A mixture with the right proportions of the selected UHTC powder,  $ZrB_2$ , and  $MoSi_2$ , which was used as a sintering additive, was first prepared. The homogeneously mixed powders were then used to make an aqueous suspension with the aid of an appropriate dispersant. The solid content in the suspension was maximized while keeping a low enough viscosity for a successful casting and avoiding trapping air bubbles. However, the high density of the  $ZrB_2+MoSi_2$  mixture caused difficulties in achieving a very stable aqueous suspension and produced undesired inhomogeneities if a conventional drying process was used to consolidate the green parts. This limitation was overcome by resorting to a fast freeze-drying of the parts using liquid nitrogen. An unidirectional freezing front was created throufg the use of the cold finger freezing technique.

Thus, the moulds consisted of a metallic cylinder at the bottom serving as cold finger method, an external cylindrical silicone mould and finally a plastic mold to create the channels inside of the part. This last piece was made from a light-curing resin using Digital Light Processing (DLP) technique.

The debinding thermal treatment of the resulting casts was optimized to ensure a complete and clean burn-out of the resin. Consolidation of the ceramic parts was subsequently carried out using pressureless Spark Plasma Sintering (SPS), a fast and energy-efficient sintering technology, in order to maximize densification and reduce grain growth in order to improve their thermo-mechanical performance.

# CERAMIC MICROBEADS FABICATED VIA UV CURING ASSISTED DROP-CASTING METHOD

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One of the challenges in production of ceramic materials is the shaping of microbeads characterized by uniform size distribution and high circularity. These materials are widely used primarily as grinding media in the ultrafine grinding of non-metallic minerals, printing ink, pharmacy etc. The ceramic microbeads are usually fabricated by dropping a suspension with alginates (as a gelling agent) into solutions containing calcium ions. However, such methods often result in the presence of  $Ca^{2+}$  ions in the sintered bodies which might negatively affect their mechanical properties.

The aim of the presented studies was to obtain alumina and zirconia microbeads via UV curing assisted drop-casting – a novel shaping method proposed by Falkowski and Zurowski [1]. In this technique, the drops of photocurable, hydrophilic ceramic suspensions are exposed to UV irradiation while they fall in a hydrophobic non-solvent (silicone oil). This method allows the shaping of ceramic microbeads in a controlled, repeatable, automated and cost-effective way. It is also worth noting that UV curable suspensions used in the process are based on water as a solvent, which is important from the ecological point of view.

The authors present the results on adjusting the composition of suspensions to obtain appropriate rheological properties. The influence of the solid loading, the ratio of water to monomers and the content of photoinitiator were investigated. In addition, the basic mechanical properties of sintered materials were determined.

Keywords: ceramic microbeads, UV curing, drop-casting, alumina, zirconia

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# FEATURES OF SINTERING NANOPOWDERS OF METASTABLE ALUMINUM OXIDE DOPED WITH STABILIZED ZIRCONIUM DIOXIDE

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One of the most promising composite materials is ceramics of  $Al_2O_3$ –YSZ system. The matrix of such a system is  $Al_2O_3$ , and the filler is zirconium dioxide stabilized by three mole percent of yttrium (ZrO<sub>2</sub> – 3 mol% Y<sub>2</sub>O<sub>3</sub>) (YSZ). The sintering temperature of pure  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> powders is 1700–1800 °C [1], which requires a lot of energy and expensive equipment. Authors already showed [2] that powders based on metastable phases  $Al_2O_3$  ( $\gamma + \theta$  modifications) can be used to obtain dense composite ceramics of the composition  $Al_2O_3$ –YSZ at lower sintering temperatures compared to powders based on the stable phase  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>.

This work is aimed at the investigation of high hydrostatic pressure effect on sintering process of  $Al_2O_3$  ( $\gamma + \theta$  modification) + *n*YSZ ceramics, where *n* = 0, 1, 5, 10, 15 wt%. An X-ray diffraction study of the  $\theta+\gamma-Al_2O_3+n\%$  YSZ powder system annealed at 1000 °C for 2 hours showed that the process of mutual protection against recrystallization occurs in the  $\theta+\gamma-Al_2O_3+n\%$  YSZ system. In the  $\theta+\gamma-Al_2O_3+5\%$  YSZ system, the maximum inhibition of the crystallization process was found, and zirconium dioxide, as a separate phase, was not detected at 1000 °C.

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# PHOTOCURABLE, AQUEOUS CERAMIC DISPERSIONS FOR 3D PRINTING TECHNIQUES

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Technological progress contributing to miniaturization increases the interest in the processes of obtaining elements of complex shapes and small sizes. A promising group of techniques which enable the production of these types of products are rapidly developed additive manufacturing techniques. 3D printing methods that use radical photopolymerization as a curing mechanism for a single printed layer gain great interest.

The main stage in the manufacturing of ceramic objects using this type of 3D printing technique is the preparation of time-stable, homogeneous, photocurable ceramic dispersion. These systems are mostly fully organic and at the sintering stage of the ceramics, the organic phase is completely thermally removed. Therefore, reducing the quantity of organic additives in 3D printing dispersions is important causing the release of lower amounts of gaseous substances into the atmosphere.

The purpose of the research was the utilization of aqueous, photocurable ceramic dispersions based on submicron ceramic powders as well as acrylate and dimethacrylate monomers for the 3D printing of ceramic elements. Ceramic dispersions of this type can compete with the currently used ceramic dispersions based solely on organic dispersing media. The influence of particular components on the rheological properties of dispersions, cure depth and selected properties of green and sintered bodies were investigated.

Keywords: 3D printing, complex-shaped ceramics, photocurable dispersion

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# DLP PRINTING METHOD IN OBTAINING OF CERAMIC-METAL COMPOSITES

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Additive manufacturing methods allow to obtain complex and precise elements with smooth surfaces and high accuracy of dimensional mapping without the use of expensive casting moulds and cutting tools. One of such methods is DLP (*Digital Light Processing*) in which the surface of the slurry is selectively cured layer by layer with UV light.

Ceramic-metal composites, thanks to their properties, find numerous applications in various industries, for example as anti-corrosion or thermal coatings. The aim of the research was to obtain alumina ceramics and composites with the addition of nickel and molybdenum by using DLP printing method. Even a small addition of the metallic particles can significantly improve the mechanical properties of the single-phase ceramic materials. The rheological properties of the slurries were determined and the cure depth of the suspensions with the addition of various photoinitiators was measured. The printing parameters were matched and samples were obtained with the use of DLP 3D printer. Printed objects were sintered and the properties of the obtained composites (such as Vickers hardness and fracture toughness) were examined. The microstructure of sintered bodies was analyzed by using SEM followed by EDS and XRD.

Keywords: ceramic-metal composites, digital light processing, 3D printing, photocurable suspensions

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OC-14

# FEATURES OF THE PHASE AND STRUCTURE FORMATION OF POWDER COMPOSITES OF THE AI-TI-C SYSTEM REINFORCED WITH FINELY DISPERSED AI<sub>2</sub>O<sub>3</sub> OBTAINED BY THE METHOD OF THERMAL SYNTHESIS

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The phase and structure formation of sintered alumomatric samples reinforced with titanim carbide and aluminum oxide during their synthesis and consolidation were studied. Composite alumomatric materials with different contents of aluminum and dispersion-strengthened additives were investigated: 30Al-40Ti-10C-20Al<sub>2</sub>O<sub>3</sub> (wt.%), 30Al-32Ti-8C-30Al<sub>2</sub>O<sub>3</sub> (wt.%). Sintered alumomatric samples were obtained by thermal synthesis, which was carried out in a vacuum induction furnace at a temperature of 950 °C and held for 1 hour with a sintering rate of 5–10 °/min. The initial mixtures were previously pressed at 200 MPa. As a result of the synthesis, the original samples (Fig.



Figure 1. Appearance of the initial pressing (left), sample after reaction synthesis (right)

1a) turned into sufficiently strong samples (Fig. 1b). At the same time, after sintering, a volumetric there is growth of compaction and the formation of macropores due to the fact that one component has a high solubility in the solid phase (the main component) and a low solubility in the liquid phase (additive) in the systems. There is a significant change in shape of the sample occurs after synthesis.

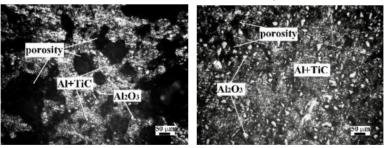


Figure 2. The microstructure of alumomatric samples of the Al-Ti-C-Al<sub>2</sub>O<sub>3</sub> system after thermal synthesis: a -30Al-40Ti-10C-20Al<sub>2</sub>O<sub>3</sub>; b - 30Al-32Ti-8C-30Al<sub>2</sub>O<sub>3</sub>(wt. %)

A new approach to the creation of such powder materials boils down to the fact that dispersing additives (in particular, TiC) are formed by thermal synthesis from

elementary powders. As a result, it is possible to obtain the necessary dispersed phases during thermal synthesis, which will allow to purpose fully affect the growth rate of grains and artificially create heterogeneity of the composite and thus form a fine-grained microheterogeneous structure with high physical and mechanical properties. The results of the microstructural analysis showed that the samples have mainly a two-phase structure - an aluminum matrix with small particles of TiC,  $Al_2O_3$  and a certain number of porosity are evenly distributed (Fig. 2). Figure 2 shows that after thermal synthesis the number of porosity in the composite with 20 wt.%  $Al_2O_3$  is significantly greater than in the composite with 30 wt.%  $Al_2O_3$ . The microhardness of the matrix phase in both samples is 2.4 GPa, and that of the oxide phase is 10.9 GPa. The results of the X-ray analysis showed a high intensity of titanium carbide, aluminum and aluminum oxide lines in both composites.

OA-15

# SOLUTION-BASED DEPOSITION OF CERAMICS ON GLASS SUBSTRATES FOR ALKALI-METAL VAPOR CELLS

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Atomic polarization lifetime of alkali-metal vapor, such as rubidium, is limited by physico-chemical interactions between confined atoms and glass structure of gas-filled cells [1], including hollow-core fibers. These interactions can be inhibited by the addition of a protective ceramic coating between the gas and the glass. A careful control of the ceramic deposition glass substrates enables the modulation of the physico-chemical interactions between the alkali-metal vapor and the glass [2]. In our study, we have focused on the deposition of ceramic thin films, such as aluminosilicates, on glass substrates [3] with different concentrations, various cleaning treatments [4] prior to the deposition of thin films, and by modifying annealing temperature and coating deposition methods. AFM investigations (see an example in Fig. 1) revealed that the microstructure of the thin films depend on the cleaning method of the glass substrates, which ensures a regular wetting of the glass surface, the concentration employed for the coating depositions and the annealing temperature. The different parameters have a large influence on the microstructure and roughness of the films, when transparent and very

homogenous coatings, as well as and smooth surfaces are desirable for optical applications.

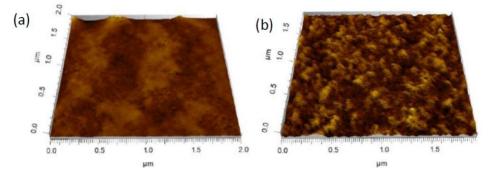


Figure 1. AFM images of aluminosilicate thin film deposited on glass substrates by dip-coating, pre-immersed in hydrogen peroxide (a) and (b) on a substrate solely rinsed with ethanol

Keywords: alkali-metal vapor cells, ceramic, coating, glass surface, chemical solution deposition

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OC-16

# MICROSTRUCTURE AND SELECTED PROPERTIES OF THE METAL-CERAMIC NANOCOMPOSITES FOR THE AEROSPACE APPLICATIONS

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For many years, the aviation industry has observed the development of composite materials and their production technologies based on conventional and advanced solutions. They are an attractive group of materials, hence there is a clear trend towards composite materials at the expense of traditional monolithic alloys. In the aerospace industry, superalloys Alloy 625 and Alloy 718 are currently used for the structural reconstruction of castings. Therefore, the selection of ceramic nanoparticles becomes a research issue, depending, among others, on reactivity with superalloy, wettability, coefficient of thermal expansion and density. Composites consisted of the Alloy 625 or 718, and ceramic nanoparticles (TiC, WC,  $TiB_2$ ) has been produced via mechanical alloying and then synthesized via suction casting. The amount of nanoparticles was in the range of 0-5.0 wt.% with a step of 1.25 wt.%. The microstructure, chemical composition, phase constitution, phase transformation temperatures, and microhardness of the ex-situ composites were investigated by X-ray diffraction, scanning and transmission electron microscopy, energy-dispersive X-ray spectroscopy, thermodynamic simulation and Vickers microhardness measurements. The investigation showed that the Inconel alloys and selected nanoparticles composite without oxide inclusions, cracks or other defects can be prepared.

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OC-17

# FEATURES OF STRUCTURE FORMATION OF ZTA CERAMICS DOPED WITH ZIRCONIA

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One of the most promising ways to obtain a dense ZTA material at relatively low sintering temperatures can be the use of metastable  $\gamma$ + $\theta$ -Al<sub>2</sub>O<sub>3</sub> phases as a basis for composite ceramics. The work [1] shows the possibility of obtaining dense composite ceramics of the Al<sub>2</sub>O<sub>3</sub>–YSZ system based on metastable powders  $\gamma$ + $\theta$ -Al<sub>2</sub>O<sub>3</sub> at lower sintering temperatures compared to powders of the stable phase  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, however, aggregation of the YSZ impurity in the intergranular space of Al<sub>2</sub>O<sub>3</sub> leads to a decrease in the level physical and mechanical characteristics of ceramics due to the pronounced polydispersity of its structural elements. This problem can be solved by varying the conditions for obtaining composite ceramics, in particular, by varying the value of high hydrostatic pressure during the processing of compacts.

As a basis for ceramics, nanopowders of the  $\gamma+\theta$ -Al<sub>2</sub>O<sub>3</sub> + n% (ZrO<sub>2</sub> + 3mol% Y<sub>2</sub>O<sub>3</sub>) (YSZ) system were used, where n = 0, 1, 5, 10, 15 wt.%, annealed in air at a temperature of 1000 °C. The powder compacts were processed under high hydrostatic pressure (HHP) conditions ranging from 300 MPa to 700 MPa. The compacts were sintered in air at a temperature of 1550 °C. The obtained ceramic composite had a two-phase structure  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>+n% YSZ.

The study of the surface structure by SEM showed the distribution of YSZ grains in ceramics depending on the treatment of compacts with HHP. At pressures of 300–500 MPa, YSZ grains are concentrated in the intergranular space of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, and at 600–700 MPa, YSZ grains are distributed over the entire volume of the ceramic matrix. In the first case, the structure of the ZTA composite corresponds to an aggregate-strengthened structure, and in the second case, to a dispersion-strengthened structure.

X-ray diffraction analysis and SEM revealed that the process of primary recrystallization and normal growth of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> grains occurs during sintering of compacts, which were processed under HHP conditions at 300–500 MPa. In compacts processed at 600–700 MPa, the process of secondary recrystallization occurs, which leads to a bimodal size distribution of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> grains.

The study of physical and mechanical characteristics showed that the maximum values of physical and mechanical characteristics are achieved in ceramic compositions Al<sub>2</sub>O<sub>3</sub> + 10% YSZ ( $\rho = 4.1 \text{ g/cm}^3$ , Hv = 20.16 GPa,  $\sigma = 338 \text{ MPa}$ ) and Al<sub>2</sub>O<sub>3</sub> + 15% YSZ ( $\rho = 4.09 \text{ g/cm}^3$ , Hv = 18.5 GPa,  $\sigma = 396 \text{ MPa}$ ) based on metastable  $\theta + \gamma - \text{Al}_2\text{O}_3$  powder annealed at 1000 °C and treated with high hydrostatic pressure at 700 MPa.

Keywords: zirconia-based ceramic materials, direct ink writing, yttrium content

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OC-18

# EFFECT OF METAL-ORGANIC FRAMEWORK (MOF) INCORPORATION ON THE PROPERTIES OF α/β-SIAION MATRIX CERAMIC COMPOSITES

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SiAlONs have emerged as a promising class of advanced materials with multifaceted applications owing to their remarkable combination of mechanical strength. thermal stability, and chemical resistance. Despite these superior features, their electrical conductivity and fracture toughness has remained a challenge, limiting their potential applications in various fields, such as aerospace, automotive, shaping processes, and electrical. To overcome these limitations, researchers have explored innovative strategies to enhance the properties of SiAlONs. One of the promising practices is the incorporation of metal-organic frameworks (MOFs). MOFs are porous materials known for their exceptional surface area, making them promising candidates for a wide range of applications. MOFs have drawn significant interest in fields like electrochemical application, gas storage, separation, and catalysis due to their high porosity and large internal surface areas. To our best knowledge, there is no report on zeolitic imidazolate framework (ZIF-8) incorporated  $\alpha/\beta$ -SiAlON matrix ceramic composites. Therefore, in this work, the effect of zeolitic imidazolate framework addition on the properties of  $\alpha/\beta$ -SiAlON composites were investigated. The composites were fabricated by Spark Plasma Sintering at 1850 °C for 10 min under 50 MPa using two different starting powder mixtures. Their compositions were kept the same (Si<sub>3</sub>N<sub>4</sub> (76.22 wt.%), AlN (13.45 wt.%), Al<sub>2</sub>O<sub>3</sub> (3.83 wt.%) and  $Y_2O_3$  (6.50 wt.%)), however, their particle sizes were monitored by ball-milling process to explore the particle size effect on the densification behavior and properties of ceramic composites. The obtained results showed that while the coarser particles of conventional starting powder were restricted to densification, full densification was achieved using nano-sized starting powder. On the other hand, 5 wt.%

ZIF-8 incorporated  $\alpha/\beta$ -SiAlON composites exhibited a dense microstructure, a lower electrical resistivity, and a higher fracture toughness.

#### OA-19

# SYNTHESIS AND STRUCTURAL ANALYSIS OF POLYSILAZANE-DERIVED SICN WITH TUNEABLE CARBON CONTENT

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Silicon carbonitride (SiCN) is an example of a material from Polymer Derived Ceramics (PDCs) class. Polysilazane-derived SiCN contains strong, covalent Si–N and Si–C bonds as well as nanodomains built from  $sp^2$ -coordinated carbon, the so-called free carbon phase. The presence of former provides high mechanical strength and excellent resistance to high-temperature oxidation, and the presence of the latter ensures high electrical conductivity [1].

The aim of this study was to obtain polysilazane-derived SiCN with tuneable carbon content. Polysilazanes were synthesized by hydrosilylation of silazane monomers and vinyl-group-containing crosslinkers, catalyzed by Karstedt's catalyst. Two silazane monomers were considered: 1,1,3,3-tetramethyldisilazane (TMDS), and 1,2,3,4,5,6,7,8-octamethyltetracyclosilazane (OMTCS); and three crosslinkers, each containing a different amount of carbon: 1,7-octadiene (OD), divinylbenzene (DVB), and 1,3,5-trivinyl-1,3,5-trimethylcyclotrisilazane (TVTMCTS). In total, 6 polysilazanes, each with a different amount of carbon was obtained. The polysilazanes were pyrolyzed in argon at two different temperatures: 800 and 1000 °C, and as a result polysilazane-derived SiCN with different carbon content was obtained. The obtained polysilazanes were characterized using: ATR-FTIR spectroscopy, Raman spectroscopy, thermogravimetric analysis (TGA). The ceramic materials were characterized using: transmission-FTIR spectroscopy, Raman spectroscopy, X-ray diffraction (XRD), SEM microscopy with EDS and combustion elemental analysis.

Acknowledgements: Authors gratefully acknowledge Prof. Magdalena Szumera from Faculty of Mater. Sci. and Ceram., AGH University of Krakow for TGA measurements, Ph.D. Zbigniew Olejniczak from Institute of Nuclear Physics, Polish Academy of Sciences for NMR measurements, and MSc. Eng. Patryk Szymczak from Faculty of Materials Science and Ceramics, AGH University of Krakow for XRD measurements.

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# TAILORING THE PROPERTIES OF ZIRCONIUM OXYCARBIDES AND OXYCARBONITRIDES BY ADJUSTING THEIR CHEMICAL COMPOSITIONS

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Investigation of the Zr-C-O-N system has been continued for decades. Promising mechanical and thermoelectric properties of zirconium oxycarbides and oxycarbonitrides as well as their good refractoriness offer great potential for various applications such as in nuclear reactors, refractories and fuel-cells. The number of reported compounds showed a considerable scattering of distinguishing characteristics for these materials. Thus, the relation between the stoichiometry of ZrCO and ZrCON phases and their properties became the focus of this research. For this purpose, common methods of chemical composition determination for these materials were extensively discussed. A new approach to the investigation of Zr-C-O-N compounds is suggested. First, it attempted to obtain single phases of these materials by optimizing the synthesis procedures. As a result, two different solid solutions with respect to oxygen content were formed. By adjusting the carbon content in the recipe of the initial blend, the final composition of obtained solid solutions can be tailored. Secondly, the characterization routine for stoichiometry determination was introduced including X-ray diffraction combined with microscopical investigations with chemical microanalysis. Lastly, the correlation between the properties of zirconium oxycarbides and oxycarbonitrides and their corresponding phase diagrams were revealed. The presented findings offer new perspectives on tailoring the properties of materials containing ZrCO and ZrCON compounds for different applications by adjusting their chemical compositions.

# UHTCs COMPOSITES BASED ON THE BORON CARBIDE WITH INTERMETALLIC ADDITIVES FROM TI-SI SYSTEM

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Composites based on boron carbide ( $B_4C$ ) have attracted considerable attention because of their outstanding physical and chemical properties. As part of this research, by using appropriate reagents, including boron carbide, carbon, and selected intermetallic compounds mixed in appropriate molar proportions, it was possible to obtain a composite consisting of 99% reacted starting materials. TiB<sub>2</sub>-TiC-SiC-Intermetallic (TiSi, TiSi<sub>2</sub> or Ti<sub>5</sub>Si<sub>3</sub>) composites were prepared by three different methods: pressureless sintering, hot pressing, and spark plasma sintering (SPS).

Different sintering temperatures are used for each consolidation process. For free sintering, the synthesis was conducted at 1650–1750 °C, for hot-pressing and SPS it was possible to lower the synthesis temperature to 1500–1550°C and 1400–1450°C, respectively.

The mechanism of potential chemical reactions is related to the type of intermetallic phase used; surprisingly, the simple and lower-temperature method according to the invention [1] is possible only because of the use of a phase in the form of intermetallic (TiSi, TiSi<sub>2</sub>, Ti<sub>5</sub>Si<sub>3</sub>) [2] in combination with the appropriate molar proportions of the reactants used. This process eliminated the carbon from the final composition of the material. This was an unexpected effect, particularly when using a commercial  $B_4C$  substrate, which typically contains carbon in the form of graphite. The obtained high-temperature refractory TiB<sub>2</sub>-TiC-SiC composite showed high mechanical strength and fracture toughness [2]. The chemical reactions that occur during sintering are highly effective. Almost 99% of the initial phases decomposed and allowed the formation of new TiB<sub>2</sub> and SiC phases, which were well-densified at relatively low temperatures. The TiC phase was formed only when no boron was present in the system during sintering. Hence, with the addition of  $Ti_5Si_3$ , when there is a significant amount of Ti in the system and a lack of boron, a small amount of TiC is formed [3]. When assessing the sinterability of materials in the case of the free sintering method, it can be concluded that the amount and type of additive added have an impact on lowering the sintering temperature.

Keywords: B<sub>4</sub>C, intermetallics, SPS, UHTCs, high pressure high temperature

Acknowledgments: This work was carried out within the statutory activities of AGH University of Science and Technology in Cracow, Faculty of Materials Science and Ceramics (16.16.160.557). This research was supported by a grant from the National Center for Research and Development (LIDER XIII; Grant No. 0024/L-13/2022 - Principal Investigator: D. Kozień)

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#### OC-22

# PRODUCTION OF FUNCTIONALLY GRADIENT CERAMIC-METAL INTERPENETRATING COMPOSITES VIA PRESURELESS INFILTRATION FOR BALLISIC APPLICATIONS

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Developing lightweight armour is vital for improving personnel and vehicle protection, comfort, mobility, speed, and efficiency. Due to their high abrasion resistance, ceramics are ideal candidates for ballistic applications. However, they demonstrate a poor multi-hit potential. This is due to the ceramic strike faces and ductile backing materials used in traditional armour having mechanical impedance mismatches. Because of this, the incoming compressive stress waves will be reflected as tensile stress waves at the interface of the ceramic strike face and can cause catastrophic failure.

3-3 interpenetrating composites (IPCs) are composites with 3-dimensional interpenetrating matrices. Since both phases are interconnecting in all 3 dimensions, they can yield superior properties for stress wave attenuation, and therefore provide resistance to high-energy projectile impacts. Using these as an intermediary layer between the strike face and backing material to reduce the impedance mismatch is an attractive solution.

In the current work, alumina foams infilled with molten aluminium-magnesium (Al-10Mg) alloy were assessed and ballistic targets produced by bonding the IPC to a monolithic alumina strike face during infiltration in a single process step. The thickness of the strike faces and IPCs were varied to determine their effect. The infiltrations were carried out at 915  $^{\circ}$ C in a nitrogen atmosphere over a range of durations. Subsequent

characterisation was done to determine the extent of infiltration and possible mechanisms. The ballistic performance of the samples were tested against a 7.62 APM2 penetrator by means of depth of penetration measurements. The presence of the IPC was observed to improve the ballistic performance of the front face tiles.

#### OA-23

# PREDICTIONS OF THE GRAIN SIZE DEPENDENCE OF THERMAL CONDUCTIVITY FOR La<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> AND OTHER PYROCHLORE CERAMICS

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Lanthanum zirconate ( $La_2Zr_2O_7$ ) is a major candidate for thermal barrier coatings (TBCs), but in doped form also as a photoluminescent material, similar to other doped pyrochlores. For both applications thermal conductivity is an important basic material property. In this contribution, the grain size dependence of the thermal conductivity was predicted using two different analytical modeling approaches (phase mixture model / PMM and sharp interface model / SIM) for  $La_2Zr_2O_7$  and other pyrochlore ceramics. Two different types of grain shapes were assumed (cubes and tetrakaidecahedra) and input parameters (thickness of grain boundary, grain boundary conductivities corresponding to the so-called amorphous limit estimated according to Clark's relation on the basis of Young's modulus, interfacial thermal resistance) were estimated based on typical data from the literature. These analytical model predictions were compared to numerical PMM-based calculations on computer-generated random grain microstructures (i.e. dense three-dimensional arrays with randomly arranged flat-faced isometric grains), using the commercial software package GeoDict<sup>®</sup>. The results of parametric studies indicate that the SIM predictions are very sensitive to the value of the interfacial thermal resistance used as an input information, whereas the PMM predictions are relatively robust and rather insensitive with respect to changes in the grain boundary thickness and conductivity as well as the grain shape. Although tetrakaidecahedral grain shapes are slightly more realistic than cubic grain shapes, an overall comparison of analytical and numerical PMM-based predictions shows that the model assumptions concerning grain shape are of secondary importance compared to those concerning grain boundary thickness. The comparison of SIM and PMM predictions show that the correct order of magnitude of interfacial thermal resistances is  $10^{-9}$  m<sup>2</sup>K/W rather than  $10^{-8}$  $m^2K/W$ .

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# COMPOSITION TUNABLE Ni<sub>1-x</sub>Mg<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> ( $0 \le x \le 1$ ) CERAMIC NANOCATALYSTS FOR ENHANCED HYDROGEN EVOLUTION AND OXYGEN EVOLUTION REACTIONS

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The current technological advancement has created a room to existence of diverse functional ceramic materials for energy applications. However, there is still an unmet demand for electrocatalysts with structural controllability and electrochemical stability. Fortunately, mixed metal ferrites can rationally be tailored to meet these requirements, and deliver enhanced electrochemical activity. Herein, a series of Ni<sub>1-x</sub>Mg<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> ( $0 \le x \le$ 1) ceramic solid solutions were synthesized via solventless thermolysis at mild temperature. The physicochemical properties of nanostructures were acertained via p-XRD, EDX, SEM, TEM, HRTEM, and SAED techniques. Electrochemical investigation of the ceramic nanoferrites revealed that by tuning the local chemical environment of Ni in NiFe<sub>2</sub>O<sub>4</sub> via Mg substitution, the intrinsic catalytic activity was enhanced. Clearly, the optimized Ni<sub>0.4</sub>Mg<sub>0.6</sub>Fe<sub>2</sub>O<sub>4</sub> ceramic displayed enhanced HER activity with much lower overpotential of 121 mV compared to the pristine NiFe<sub>2</sub>O<sub>4</sub> catalyst. Moreover, Ni<sub>0.2</sub>Mg<sub>0.8</sub>Fe<sub>2</sub>O<sub>4</sub> catalyst demonstarted the best OER performance with a low overpotential of 284 mV at 10 mA/cm<sup>2</sup> in 1 M KOH. Our results suggest a feasible route for developing earth-abundant metal oxide-based ceramic electrocatalysts for future water electrolysis applications.

## OPTIMIZATION OF PHENYL LADDED-LIKE SYSTEMS BY SOL-GEL SYNTHSIS

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The aim of this work was to select optimal conditions for the sol-gel synthesis of ladder-like silsesquioxanes in order to accelerate the gelation process while maintaining their ladder-like structure and storage stability.

Parameters such as pH, amount of aqueous hydrochloric acid solution, and thermal conditions, as key synthesis factors, were changed during the project. The whole project was divided into three steps involving subsequent changes to the abovementioned parameters. In the first step, the effect of the pH of the hydrochloric acid aqueous solution on the reaction to obtain ladder-like organosilicon compounds was studied. In the second one, the amount of aqueous hydrochloric acid solution was changed and lastly, the effect of temperature on the kinetics of the reaction was studied.

Structural studies of all three optimization steps were carried out using FT-IR spectroscopy. The sols were studied using the attenuated total reflectance method. The xerogels were studied using the standard KBr pellet method.

During this experiment, it was indicated that the fastest gelation had sol obtained at pH 3, using 1.5 chemical equivalents of aqueous hydrochloric acid solution at room temperature. At the same time, the presence of the silsesquioxanes ladder structure was confirmed for each of the analysed cases.

# MAGNESIUM SUBSTITUTION WITH NICKEL AND ITS INFLUENCE ON THE SENSING PROPERTIES OF MgFe<sub>2</sub>O<sub>4</sub>

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Mixed spinel ferrites  $Mg_xNi_{1-x}Fe_2O_4$  were synthesized via sol-gel combustion synthesis with citric acid as fuel, followed by calcination at 700 °C for 3 hours. Obtained powders were characterized via X-ray diffraction analysis (XRD), X-ray photoelectron (XPS), FTIR and Raman spectroscopy and FESEM microscopy. Elemental composition was examined via energy dispersive spectroscopy (EDS). Humidity sensing properties were tested by measuring AC impedance in a climactic chamber at 25 °C and in the relative humidity range of 40–90%. Temperature sensing properties were tested by measuring DC resistance at 40% RH in the temperature range 40–90 °C.

Synthesized powders were proven to be pure spinel  $Fd\bar{3}m$  phase with spherical, slightly agglomerated particles. Substitution of Mg with Ni results in structural changes such as a change in inversion parameter and particle agglomeration, which influences sensing properties of the material. Results show that the sensing properties of magnesium ferrite, which is already a well-established NTC sensor, can be improved by incorporating 10% of nickel in the spinel lattice structure. Mg<sub>0.9</sub>Ni<sub>0.1</sub>Fe<sub>2</sub>O<sub>4</sub> exhibited higher temperature sensitivity and higher sensitivity towards humidity compared to MgFe<sub>2</sub>O<sub>4</sub>, while further substitution of Mg with Ni resulted in the decline of sensing properties, increase in particle size and agglomeration degree.

# SYNTHESIS OF TERNARY BORIDE Cr<sub>3</sub>AlB<sub>4</sub> BY SOLID-STATE REACTION

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The MAB phases (M-transition metal, A-aluminium or zinc, B-boron) are promising candidates, to overcome the problems of traditional ceramics due to their combination of properties attributed to both metals and boride ceramics. The MAB phases are analogous to the widely known MAX phases (M-transition metal, A-elements from A group (mostly group 13 and 14) X-carbon or nitrogen), with the nanolaminate orthorhombic crystal structures, consisted of M-B layers (M-transition metal: B-boron). interspersed with the layers of aluminium or zinc, or double layers of aluminium atoms. This particular work is focused on the MAB phase from the Cr-Al-B system - Cr<sub>3</sub>AlB<sub>4</sub>.  $Cr_3AlB_4$  is one of the attractive compounds for high-temperature constrictive ceramics due to its combination of properties typical for ceramics or metals. In this study, the formation mechanism of Cr<sub>3</sub>AlB<sub>4</sub>, presented in the literature, was confirmed by means of the following analyses: XRD, DSC and SEM. According to them the synthesis of  $Cr_3AlB_4$  occurs is multi-step process with  $CrB_2$  being formed first, followed by  $Cr_3B_4$ , which is intercalated by aluminium to form Cr<sub>3</sub>AlB<sub>4</sub>. Furthermore, most scientific papers do not consider the possibility of synthesising MAB phase from the powders containing amorphous phase. In presented work, possibility of synthesis of Cr<sub>3</sub>AlB<sub>4</sub> from the powders containing the amorphous phase and influence of this kind of raw powders on synthesis process is shown.

#### AION POWDER VIA DYNAMIC CHEMICAL METHOD

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Being a major candidate for armor and infrared windows, Aluminum Oxynitride (AlON) is a ceramic with elite mechanical properties and excellent transparency. AlON has been for long, a subject of interest to determine an inexpensive method allowing the synthesis of its powder. The conventional process to synthesize AlON requires the use of highly pure alumina (Al<sub>2</sub>O<sub>3</sub>) and aluminum nitride (AlN) as well as excessive energy since AlON starts forming at 1640 °C and is considered instable below 1750 °C. In this study, the synthesis of aluminum oxynitride has been investigated via dynamic thermochemical method (DTM). DTM is a patented novel approach that has been deemed effective in the synthesis of technical ceramics. DTM is based on the carbothermal reduction nitridation method (CRN) with the difference of the synthesis taking place in a rotating kiln allowing the synthesis of homogenous spherical powders in a relatively shorter time. A conversion to AlON was obtained from aluminum hydroxide Al(OH)<sub>3</sub> after a continuous heating and two stages holding at 1100 °C for 1.25 h and 1500 °C for 2 h under flowing ammonia (NH<sub>3</sub>) and propene (C<sub>3</sub>H<sub>8</sub>). The obtained powders were investigated via XRD and FESEM.

Keywords: aluminum oxynitride, dynamic thermochemical method, CRN, advanced ceramics

# PREPARATION, SYNTHESIS AND CHARACTERIZATION OF NANOMETRIC Ca<sub>0.9</sub>Er<sub>0.1</sub>MnO<sub>3</sub>

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The present research demonstrates the synthesis and characterization of  $Ca_{0.9}Er_{0.1}MnO_3$  perovskite powder using the sucrose nitrate procedure (SNP) technique. The following substances were used to obtain this complex perovskite: sucrose  $C_{12}H_{22}O_{11}$ , which has a dual role (complexant and fuel), and metal nitrates were used as oxidants - calcium nitrate tetrahydrate  $Ca(NO_3)_2 \times 4H_2O$ , manganese(II) nitrate hydrate  $Mn(NO_3)_2 \times H_2O$ , erbium(III) nitrate pentahydrate  $Er(NO_3)_3 \times 5H_2O$ . Nanopowder was prepared by combining metal nitrates in their respective stoichiometric ratios. The synthesized  $Ca_{0.9}Er_{0.1}MnO_3$  powder was calcined in a temperature range of 800–1000 °C for a period of 15 min. The effects of calcination were characterized through different experimental techniques (differential thermal analysis (DTA), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Field emission scanning electron microscopy (FESEM), and inductively coupled plasma (ICP)).

# SYNTHESIS AND CHARACTERIZATION OF VANADIUM CARBIDE FOR SERS SENSORS

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MXenes as two-dimensional inorganic materials with tailorable surface chemistry have been attracting huge attention to the scientific community because of the variety in their functional properties, such as high electrical and thermal conductivity, large surface area, biocompatibility, etc. [1–4]. In this work, V<sub>2</sub>CT<sub>x</sub> was obtained by etching of V<sub>2</sub>AlC precursor by HF and mixture of HF and HCl too. XRD, Raman and SEM analyses were performed to evaluate the structure and morphology of the synthesized powder, which indicated micron-sized and almost phase-pure nature of the material. Surface-enhanced Raman scattering (SERS) is a rapid, highly sensitive, non-destructive technique with high signal amplification possibilities. MXenes are good candidates for SERS substrates due to their highly metallic characteristics such as good and unique electronic and optical properties. Also, they are flexible and hydrophilic and which makes them suitable for tagging with Raman reporters [5]. However, since V<sub>2</sub>CT<sub>x</sub> hasn't been much in focus for this application, it motivated us to develop such a sensor.

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# DRUG CONJUGATES FOR TUMOR-SPECIFIC LOCALIZATION AND SUPERIOR THERAPEUTIC ACTION

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Cancers or some subtypes of cancer show heterogenous tumor biology with a high risk of reoccurrence and metastasis and, therefore, therapeutic options for patients are limited. In addition, low solubility of the molecular drugs fetches poor efficacy and systemic toxicity. Wrapping of therapeutic molecules in nanocarriers such as mesoporous silica and liposomes can alter their solubility and bioavailability in the blood stream via various mechanisms. Fusion of ligand molecules on the surface of drug nanocarriers molecules facilitate their bio-distribution overcoming the intrinsic physiological barriers of clearance through innate immune system and ensure safe delivery of therapeutics to tumor sites. Our lab focused on the development of nanocarriers for tumor targeting and dual therapeutic responses by integrating complementary functionalities and payloads (anticancer drugs and/or radionuclides) into one system through smart covalent functionalization strategies. In our study surface grafted ligands and stimuli responsive polymers assist improved drug storage and controlled drug release. The cellular targeting and site-selective drug-delivery can be further enhanced by co-conjugating anticancer drugs on chemically engineered nanocarriers, which is an important determinant of the therapeutic index. The data on therapeutic study showed enhanced higher cell death after the incorporation of radionuclides and drugs together into these carriers. The minimization of off-target effects and effective ligand-driven delivery of nanocarriers to the mice tumors illustrate the therapeutic potential of our site-specific drug delivery approach.

# FABRICATION OF BIOINSPIRED STRUCTURES FOR DENTAL APPLICATIONS BY INDIRECT DLP

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Nature is a logical source of inspiration for the development of new materials to cover the needs of multiple industrial applications. Today's engineering materials struggle to meet simultaneously strength and toughness requirements in many of those applications. In Nature, the strength-toughness problem is solved through hierarchy and a combination of microstructure related mechanisms. Teeth are a good example of this, and the difficulties to find appropriate dental materials for replacing them when they fail are a good illustration of the aforementioned struggle and current industrial limitations.

The objective of this study is to fabricate dental materials by using a bioinspired design, i.e., engineering their microstructure to be similar to that of natural enamel. Microstructure of natural enamel consists of tightly packed, highly mineralized rods, running from the dentin-enamel junction to the occlusal surface, separated by softer sheaths with a lower mineral content.

To emulate the microstructural features of natural enamel, an indirect digital light Processing (DLP) route was used to fabricate intricate columnar ceramic structures with interstitial gaps. More concretely, a commercial photocurable resin appropriate for DLP was used to fabricate a polymeric mold mimicking the interstitial organic phase that will eventually separate the bioceramic columns. After cleaning and elimination of uncured resin remnants, concentrated  $Al_2O_3$  slurries were cast into those molds, making sure no trapped bubbles are left during the process. After drying the ceramic cast, the sacrificial molds were burn-out without leaving residues. Later, a ductile polymer was infiltrated into the interstitial gaps of the sintered  $Al_2O_3$  columnar structure. These new bioinspired composites are expected to have better mechanical performance – in terms of strength and toughness, as well as wear resistance – than current dental composites.

## COLD SINTERING PROCESS FOR DEVELOPING HYDROXYAPATITE CERAMIC AND POLYMER COMPOSITE

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Cold sintering process (CSP) is a non-conventional, low-energy sintering technique that promotes the densification of ceramics in the presence of transient liquids under low temperatures ( $\leq$ 300 °C) and pressures ( $\leq$ 500 MPa). Additionally, it provides a new strategy for the co-sintering of ceramic and polymers into a single system which is not feasible through conventional methods. Exploiting the advantages of cold sintering, this investigation has aimed to densify the hydroxyapatite (HA) at nanoscale as well as the co-sintering of HA/polylactic acid (PLA) based composite for bone regeneration applications. The importance of liquid phase chemistry in cold sintering of HA was assessed using water, acetic acid, and phosphoric acid as liquids. A linear variation in the relative density was observed with respect to the nature of liquid/ionic concentrations (0.5M, 1.0M & 2M). In the case of composites, the influence of different compatibilizers on the homogeneous integration of HA/PLA composite was examined. Eventually, this study demonstrates the importance of liquid phase chemistry in the cold sintering of HA as well as the influence of compatibilizers in co-sintering of HA/PLA composites.

# SYNTHESIS AND CHARACTERIZATION OF CELLULOSE-HYDROXYAPATITE COMPOSITE MATERIAL WITH PROPER ANTIMICROBIAL PROPERTIES

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Hydroxyapatite  $Ca_{10}(PO_4)_6(OH)_2$  - HAp presents natural construction material for bones and teeth and therefore is considered biocompatible, and has various applications in fields such as biomedicine, drug delivery, and as a biomarker. Considering nanoceramics' high specific surface area and antimicrobial activity, they can be used as potential antimicrobial materials. Bacterial cellulose (BC) is a biopolymer that stands out in its biodegradability, biocompatibility, and high water retention capacity, but also lacks functional properties. The main goal of this study is to create a newly designed composite material with the functionalization of cellulose by hydroxyapatite with antimicrobial properties. Hydroxyapatite material was incorporated in wet cellulose during the precipitation synthesis reaction. Obtained BC-HAp material was structurally and phase investigated by the XRD method. The morphology of obtained material was done by SEM, and appropriate functional groups were determined by the FTIR method. According to antimicrobial results investigated composite is shown proper antimicrobial activity.

# COMPOSITE Ni/SiO<sub>2</sub> SCAFFOLDS OBTAINED BY DIW 3D PRINTING

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Fossil fuels are now the dominant source of energy for humanity, and their combustion produces a large amount of carbon dioxide. Excessive  $CO_2$  emissions in recent decades caused a number of environmental problems, such as global warming, sea acidification and extreme climate change. Capture and catalytic conversion of  $CO_2$  into fuels or chemical compounds useful for industry (e.g. methane, methanol, ethanol, formic acid) are currently arousing great interest among scientists.

Currently designing an efficient heterogeneous catalyst with a high degree of conversion in the  $CO_2$  methanation reaction that will operate at relatively low temperatures remains a big challenge. Transition metals such as Ru, Rh, Pd and Ni are proved to be active in this reaction. Among them, nickel catalysts attract special attention because of their good catalytic properties and low price of the material.

The research involved the development of new procedure for the preparation of 3D silica-nickel heterogenous catalyst using Direct Ink Writing 3D printing. This included the following steps:

- Optimalization of the starting material including selection of appropriate proportions of the components and additives influencing rheology.
- Optimization of the 3D printing process using F-NIS 23151 (Sygnis, Poland), especially the selection of appropriate pressure and printing speed.
- Thermal processing of the printed samples to obtain the 3D structures with given microstructural properties such as specific surface area.
- Characterization of the microstructure (BET analysis, scanning electron microscopy) and structure (FT-IR and Raman spectroscopy).

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# POST-LITHIATION: A WAY TO CONTROL THE IONIC CONDUCTIVITY OF SOLID-STATE THIN FILM ELECTROLYTE

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An enormous amount of effort has been made to develop the next generation of batteries for the transition towards renewable energy. To meet the specification required for safer batteries with a higher energy density (namely about 35% increases in the gravimetric energy density), [1] we need to move away from the current intercalation mechanism to a Li metal-based structure. An all-solid-state architecture with lithium conducting ceramic electrolyte is the key to utilising this lithium metal anode.

In our project (Powering Internet of Things with Ambient Solutions – PIIoT), we are trying to develop a new family of miniaturized and sustainable devices, able to harvest and store energy from the ambient environment in a single device that we call "Micro-Harvest/Storage" ( $\mu$ -HS).

The first step of fabricating the next-generation thin-film batteries (TFB) is to obtain a thin, high-performance solid-state electrolyte, which enables fast Li-ion transfer. Lithium lanthanum zirconium oxide (LLZO) is one of the most promising materials due to its high ionic conductivity and wide electrochemical stability windows. [2] In order to manufacture this nanometre-thick electrolyte film, pulsed laser deposition (PLD) was used. Although many attempts have been made to deposit LLZO using PLD, the effects of deposition/post-deposition conditions on the performance of the films were not fully investigated. There is still a large gap between the bulk material and pulsed laser-deposited films in terms of ionic conductivity. [3]

In this presentation, we are going to outline the strategies we used to increase the qualities of the films deposited by PLD and highlight the results we obtained with different deposition/post-deposition parameters.

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# THERMALLY STABLE SILICONE ELASTOMER COMPOSITES BASED ON M<sub>0</sub>S<sub>2</sub>@BIOMASS-DERIVED CARBON WITH HIGH DIELECTRIC CONSTANT AND ULTRALOW LOSS FOR FLEXIBLE MICROWAVE ELECTRONICS

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The demand for low-cost flexible composites with temperature-stable high dielectric constant and low loss has increased due to the miniaturization and integration of electronic components in wireless communication and wearable devices. However, it is challenging to combine such comprehensive properties in conventional conductive and ceramic composites. In this study, we have developed silicone elastomer (SE) composites using hydrothermally grown MoS<sub>2</sub> on cellulose carbon (CC) derived from biomass-tissue paper. This design facilitated the formation of microcapacitors, multiple interfaces, and defects, which enhanced interfacial and defect polarizations. As a result, the composites exhibited a high dielectric constant of 9.83 at 10 GHz, with a low filler loading of 15 wt.%. Unlike highly conductive fillers, MoS2@CC had low conductivity, ensuring a very low loss tangent of  $7.6 \times 10^{-3}$ . The filler dispersion and adhesion to the matrix also influenced the loss tangent. In addition to breaking the traditional conflict between high dielectric constant and low losses in conductive composites, MoS<sub>2</sub>@CC/SE composites were highly flexible and stretchable and had temperaturestable dielectric properties. This makes them suitable for applications as flexible substrates in microstrip antennas and extreme environment electronics while preserving composite integrity. Furthermore, the use of biomass-tissue paper for cellulose carbon makes these composites potential candidates for low-cost and sustainable dielectric materials.

Keywords: biomass carbon, composite material, dielectric constant, loss tangent, microwave applications

# ELECTRICAL PROPERTIES OF NANOSTRUCTURED SYSTEMS FeSe-CuInSe<sub>2</sub> AND MnSe-CuInSe<sub>2</sub> UNDER CONDITIONS OF VARYING MOISTURE AND LIGHTING

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At the present time, the problem of renewable energy sources is relevant due to the depletion of traditional energy resources. In this aspect, a lot of work has been done in the field of developing converters into an electric form of solar energy. New devices are being developed, which are based on new physical principles. In particular, encouraging results have been obtained in the field of adsorption electric power. Development of devices capable of converting the chemical energy of adsorption of atmospheric moisture molecules into an electrical form, including by electrostatic charge capture of microscopic water droplets using electrostriction caused by water adsorption and etc.

It is relevant to study the electrical properties of nanostructured crystals FeSe-CuInSe<sub>2</sub> and MnSe-CuInSe<sub>2</sub> at direct current, which was the purpose of this work.

Crystals of two compositions were used as the studied objects: 10 mol% MnSe - 90 mol% CuInSe<sub>2</sub> and 5 mol% FuSe - 95mol% CuInSe<sub>2</sub> obtained using chemical technology. voltammograms (VAC) were obtained in the linear scan mode on the R-20 device ("Elinns") in the moisture saturation mode at three points (75, 35 and 26%  $\pm$ 5%). The sample chamber was a closed 350 ml container with controlled atmospheric humidity using MgCl<sub>2</sub>×6H<sub>2</sub>O (35%) and NaCl (75%) salts.

A new dimensional effect of switching an electrical parameter has been established. It is assumed that the observed features of the behavior of the studied

systems in humidification and lighting conditions are due to the bimodal structure of the sample. Large single crystals are effective photo adsorbers, and nanopores are physicochemical proton reactors. It was found that an increase in humidity over 35% leads to the appearance of nonlinear sections of the VAC. The established effects can find application to nanoelectronics in the development of new devices microsystem technology, etc.

#### OC-39

## DESIGN OF BaTiO<sub>3</sub> WITH THE SHAPES OF EQUIAXED, PLATELET AND NANOWIRE BASED FLEXIBLE NANO GENERATORS

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With the growing global population and escalating non-renewable energy consumption per capita, the integration of renewable energy sources into our daily lives has become paramount. To power micro/nano devices that demand low operating energy and prolonged battery life, the development of renewable energy harvesting technologies capable of generating green energy is imperative. Among the numerous renewable sources available, kinetic energy harvested from routine daily activities such as walking, running, and dancing give a great promise.

While electrochemical batteries have traditionally served as the primary power source for micro/nano devices, their drawbacks, including weight, inflexibility, the need for regular charging, and associated risks of explosion and recycling, have triggered a surge of interest in nanogenerators. Nanogenerators can convert environmental mechanical energy into electrical energy, eliminating the need for supplementary power sources. In recent years, nanogenerators have exhibited significant enhancements in output performance, making them a potential solution for powering small electronic devices.

The research objective of this study was to design a nano generator, based on lead-free piezoelectric materials which come with the benefits on eliminating the harmful effects of materials on human health and the environment with the higher electrical output. Among lead free piezoelectric materials,  $BaTiO_3$  is a strong candidate due to its exceptional biocompatibility, high dielectric constant, and remarkably high piezoelectric constant. To the best of our knowledge, the piezoelectrical performance analysis of

 $BaTiO_3$  structures with different dimensions and shapes, equiaxed powders, nanowires (NWs), and platelets is missing in the existing literature. Addressing this research gap, the study aims to compare the electrical output properties of these different  $BaTiO_3$  structures and contribute to the body of knowledge surrounding piezoelectric materials.

In summary, with the findings about the effect of different shapes of  $BaTiO_3$  including equiaxed, nanowire and platelet on piezoelectrical performance, the optimum design parameters of nano generators can be determined. This will facilitate the development of more efficient energy harvesting devices while ensuring a safer and more sustainable approach using lead-free materials.

#### OC-40

# SCREEN PRINTED GRAPHITE-GLASS COMPOSITE ELECTRODES FOR DETECTION OF NEONICOTINOID PESTICIDES

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This study addresses the fabrication and evaluation of screen-printed electrodes (SPEs) for electrochemical (EC) detection of neonicotinoid pesticides (NNI) in water based on the reduction of nitro functional group. Apart from their low cost and the possibility of mass production, SPEs also have the advantage of providing a reasonable level of reproducibility, miniaturization, and on-site analysis [1,2]. The developed SPEs consist of a silver reference electrode (RE) and electrical connections, a platinum counter electrode (CE) and a novel graphite-glass composite working electrode (WE).

Commercially available Pt and Ag pastes were screen printed on alumina substrate and fired at 1100 and 900 °C, respectively. The paste for WE was prepared by dispersing 15 vol% of a graphite-glass powder in a terpineol-based vehicle. The paste was screen printed on the same alumina substrate and annealed at 850 °C in an argon atmosphere. The resulting graphite-glass layers were well adhered to the alumina, and exhibited uniform surface, consistent thickness of  $37\pm1.5 \ \mu\text{m}$  and surface roughness of 1.1  $\mu\text{m}$ . However, their sheet resistance ( $R_s$ ) was affected by trace amount of oxygen in the furnace during annealing. When we maintained an oxygen concentration below 100 ppm, we were able to achieve a reproducible  $R_s$  of 7  $\Omega$ /sq.

The effect of different  $R_s$  of WE on their EC behavior was investigated using three batches with  $R_s$  of 68  $\Omega$ /sq, 17  $\Omega$ /sq and 7  $\Omega$ /sq. Cyclic voltammetry (CV) measurements

of these SPEs were performed in a model solution containing 5 mM Fe(CN)<sub>6</sub><sup>3-/4-</sup> in a 0.1 M phosphate buffer solution (PBS, pH = 7). The results showed that the WE with the lowest R<sub>s</sub> exhibited high reversibility with a cathodic-to-anodic peak current ratio of 0.98 and the smallest peak-to-peak separation ( $\Delta Ep$ ) of 80 mV, confirming its superior conductivity. We also confirmed EC response of these SPEs in a model solution containing 1 mM of the selected NNI imidacloprid. We will discuss the differences in the voltammograms obtained and demonstrate the potential of these SPEs for neonicotinoid detection.

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OA-41

# IMPACT OF THE STEEL PRE-OXIDATION ON THE QUALITY OF MC11- AND CMF-BASED PROTECTIVE LAYERS FOR SOC INTERCONNETS

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Solid Oxide Cell (SOC) stacks are intricate, multi-layered systems constructed of i.e. metallic components such as interconnects. The interconnects are commonly made from ferritic steel with chromium content of over 20%. However, even employing these extremely sophisticated steel types, a protective coating is needed to maintain high durability of SOC stack. The role of protective layers applied on interconnects is to minimize the occurrence of chromium volatilization and poisoning of the cell's air electrode. Value of thermal expansion coefficient (TEC) of the protective material should be compatible with the TEC of interconnect steel, as well as, the coating layer should possess a low area specific resistance (ASR), be uniform and well-adhered at the whole coated area. Typically  $Mn_{1.5}Co_{1.5}O_4$  spinel is used, however, cobalt's toxicity, scarcity, and supply uncertainty make it challenging to apply this material in SOC technology on an industrial scale.

In presented studies, two spinel oxides:  $Mn_{1.5}Co_{1.5}O_4$  (MC11) and  $CuMn_{1.9}Fe_{0.1}O_4$  (CMF) were investigated and compared in terms of their application as protective coatings for SOC interconnects. In order to reduce the oxidation rate of the steel substrate, the samples were additionally examined through different conditions of steel pre-oxidation before the coating deposition. Protective layers were applied using the electrophoretic deposition (EPD) method.

The microstructure of the coatings, as well as, the contact between the coatings and steel were assessed by the SEM method. Moreover, the ASR measurements of the prepared samples were carried out in air and at SOC stack operation temperature in range of 650-700 °C.

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Keywords: interconnect, protective coating, pre-oxidation, spinel oxides, EPD.

#### OA-42

# EXPLORING PrVOy-CaVOy OXIDES: CHARACTERIZATION AND PERFORMANCE IN SOLID OXIDE FUEL CELLS

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Rare-earth and alkaline-earth vanadates have garnered significant attention as promising materials for electrochemical applications, especially as redox-reversible components for fuel electrodes in solid oxide fuel cells (SOFC). An indispensable advantage of (Ln,A)VOy-derived components (A - alkaline-earth metal cation) in SOFC anodes is their expected resistance to carbon deposition and sulfur-containing impurities, making them crucial for the efficient operation of hydrocarbon- and biogas-fueled solid oxide fuel cells (SOFCs). The current study primarily investigated the oxides within the PrVO<sub>4</sub>-Ca<sub>2</sub>V<sub>2</sub>O<sub>7</sub> system as precursors for fuel electrodes, with a specific emphasis on phase formation, redox and thermomechanical behaviour, as well as electrical properties. PrVO<sub>4</sub>, Ca<sub>2</sub>V<sub>2</sub>O<sub>7</sub>, and ceramics with the nominal composition Pr<sub>1-x</sub>Ca<sub>x</sub>VO<sub>4-δ</sub> (x = 0.02-0.20) were synthesized using the conventional solid-state route. Ceramics samples were sintered at 1000 °C for Ca<sub>2</sub>V<sub>2</sub>O<sub>7</sub> and 1300 °C for other materials. XRD demonstrated the formation of phase-pure Pr<sub>1-x</sub>Ca<sub>x</sub>VO<sub>4-δ</sub> solid solutions with the tetragonal zircon-type structure for up to 5 at.% of calcium in the Pr sublattice. At the same time, SEM/EDS suggest a lower solubility indicated by the presence of Ca-V-O phase impurities. The

introduction of calcium through doping enhances the ionic-electronic conductivity of  $Pr(Ca)VO_4$  ceramics, particularly under oxidizing conditions.

The electronic contribution is p-type and decreases with reducing  $p(O_2)$ . The reduction of  $Pr_{1-x}Ca_xVO_{4-\delta}$  ceramics in a  $10\%H_2$ -N<sub>2</sub> atmosphere at 800 °C induces phase separation, forming perovskite-like PrVO<sub>3</sub> and CaVO<sub>3</sub> phases. The redox behaviour of PrVO<sub>4</sub>-Ca<sub>2</sub>V<sub>2</sub>O<sub>7</sub> ceramics was investigated through isothermal cycling between air and  $10\%H_2$ -N<sub>2</sub>, employing techniques such as impedance spectroscopy, thermogravimetry, dilatometry, and post-mortem XRD analysis.

OA-43

#### STRUCTURAL AND ELECTRICAL STUDIES OF SILICON-DOPED Bi<sub>4</sub>V<sub>2</sub>O<sub>11</sub>

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The partial substitution of vanadium by silicon in the compound  $\text{Bi}_4\text{V}_2\text{O}_{11}$ , which belongs to the Aurivillius family, results in the creation of a solid solution  $\text{Bi}_4\text{V}_{2-x}\text{Si}_x\text{O}_{11-\delta}$  $(0.0 \le x \le 0.4)$ . The structural characterization of those materials was performed using Xray powder diffraction, SEM-EDS, Raman, FT-IR, and Electrochemical Impedance Spectroscopy. Compounds with x = 0.10 correspond to  $\alpha$ -monoclinic form of  $\text{Bi}_4\text{V}_2\text{O}_{11}$ , whereas the  $\beta$ -orthorhombic phase is obtained in the compositional range of  $0.20 \le x \le$ 0.30, and the compound with x = 0.35 is of  $\gamma$ -tetrahedral polymorph. The stability of these phases was confirmed by Raman spectroscopy, infrared spectroscopy and DTA. The ceramic sample with x = 0.1 has the highest ionic conductivity values ( $10^{-5}$  S.cm<sup>-1</sup> at 300 °C and  $10^{-2}$  S/cm at 600 °C).

# PROCESSING AND CHARACTERIZATION OF ULTRATHIN EPITAXIAL LaMnO<sub>3</sub> BASED FILMS BY CHEMICAL SOULTION DEPOSITION

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Development of faster, smaller and less energy consuming devices led to new concept of electronics called spintronics. Spintronics are based on properties of electrons and associated magnetic moments. Promising materials which can follow this concept are materials based on lanthanum manganite ( $LaMnO_3$ ).  $LaMnO_3$  is specific due its magnetoelectric properties such as giant magnetoresistance (GMR) and multiferroic behaviour. Also, perovskite structure of LaMnO<sub>3</sub> is very sensitive to external stimuli and ions substitution which gives possibility of phase control and fast manipulation of electric and magnetic properties. Ultrathin epitaxial films are conventionally obtained from gas phase, utilizing sophisticated equipment and a lot of energy. Following the trend of devices miniaturization, this form of material is very desirable. The aim of this work was obtaining epitaxial thins film based on LaMnO<sub>3</sub> / La<sub>1-x</sub>Sr<sub>x</sub>MnO<sub>3</sub> from liquid phase. Polymer assisted deposition (PAD) technique was used as synthesis method.  $LaMnO_3 / La_{1,y}Sr_yMnO_3$  solutions were prepared by dissolving of  $La(NO_3)_3 \cdot 6H_2O$  and  $Mn(NO_3)_2 \cdot 4H_2O$  in the distilled water, while strontium nitrate was used as a source of  $Sr^{2+}$  ions. Polyethileneimine and EDTA were added to stabilize aqueous solution of precursors Prepared solutions of polymer and precursors were deposited on single crystal  $SrTiO_3$  (001) substrate in order to achieve highly oriented growth. Deposited films were thermally treated up to 900 °C. After the annealing process, structural characterization was performed by XRD and HR-TEM, while functional properties were investigated by magnetic measurements.

Keywords: lanthanum-manganite, epitaxial film, chemical solution deposition

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# SYNTHESIS AND INVESTIGATING THE PROPERTIES OF PURE AND DOPED ZnO THIN FILMS OBTAINED BY SPRAY PYROLYSIS

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Transparent and conductive ZnO films – pure and doped were prepared by spray pyrolysis technique on glass and silicon substrates. Transparent and conductive ZnO films have been extensively studied in recent years, because of their low cost precursors materials and relatively low deposition temperature. Due to these properties the films have been used in varities of applications such as high temperature thermoelectric device fabrication, active emitter in LED and laser diodes, TFT for real-time sensing of gas molecules, acoustic sensors, etc.

Our main task consisted of obtaining thin films of ZnO using  $Zn(CH_3COO)_2 \cdot 2H_2O$  undoped and doped with different additives. The specific amount of dopant (Na, In (NaOH, InCl<sub>3</sub>)) influences the conductivity, the transparency and the electrical resistivity of the films.

The microstructure and morphology were studied by XRD, SEM and AFM techniques. The spectrophotometer UV-VIS in the region of 300–1200 nm was used to study the transmission spectra of the films.

Key words: doped ZnO, thin film, spray pyrolysis

# EFFECT OF RE<sup>3+</sup> DOPING ON THE MAGNETO-OPTICAL AND LUMINESCENT PROPERTIES OF Y<sub>2</sub>O<sub>3</sub>

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Submicron-sized Eu<sup>3+</sup>:Y<sub>2</sub>O<sub>3</sub> particles were successfully prepared via an sol gel method using EDTA acid, followed by a calcination at 800 °C. Crystal structure, morphological and luminescent properties were analyzed by X-ray diffraction (XRD). scanning electron microscope (SEM), photoluminescence (PL) and luminescence decay curves. XRD analysis confirmed the presence of single phase. Incorporation of europium oxide ions was observed to increase the crystallite size of the synthesized materials. SEM observations confirmed the existence of uni-modal grain size distribution. The highly crystallinity transparent ceramics have been prepared by an arc plasma melting process [1]. The samples demonstrated the highest in-line transmittance, of 60–80% from 400 to 1110 nm wavelength range. For the  $Eu^{3+}$ : Y<sub>2</sub>O<sub>3</sub> powder and bulk samples, the luminescence emission was observed in the 200-650 nm range. Sharp and intensive peaks in polycrystalline materials were found around 366, 470, 535 and 610 nm. The excitation spectrum was found at 255 nm. The magneto-optical properties of samples were studies using the special setup described in details elsewhere [2]. Studied materials exhibited large values of Verdet constant (V=122-33 rad/Tm in VIS spectrum) and optical band gap energy (Eg = 4.5-5.2 eV). These results indicate that the prepared samples can be used in magneto-optics devices.

Keywords:  $Y_2O_3$ , luminescence, transparent ceramics, magneto-optical properties, arc plasma melting

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# SOLUBILITY OF ZrO<sub>2</sub> IN YTTRIUM OXIDE AND ITS INFLUENCE ON TRANSPARENT CERAMICS PROPERTIES

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Defect-free  $Y_2O_3$  transparent ceramics is an interesting material which is used for IR windows, nozzles or laser host, etc, due to its mechanical and optical properties, high thermal and chemical stability, and high melting point. To achieve full densification and the absence of pores, sintering aids are used. For yttria ceramics, the optimal choice is  $ZrO_2$ . It has a significant influence not only on transmittance, but on the other characteristics of the material as well. For high transparency, ceramics has to remain single-phase, thus forming a solid solution of  $Y_2O_3$  and  $ZrO_2$ , but the solubility limit isn't studied in detail. Therefore, the presented work aims to investigate the solubility limit of  $ZrO_2$  in yttria ceramics and the dependence of properties on the concentration of sintering aid.

Ceramics samples with a concentration of  $ZrO_2$  of 0–11 mol% were obtained by uniaxial and cold isostatic pressing (CIP) followed by vacuum sintering at 1735 °C for 22 h. The SEM and XRD analysis, refractive index and transmittance measurements were performed. No secondary phases were observed in analysed samples by SEM, which indicates the formation of a solid solution and full solubility of  $ZrO_2$  in the analysed range of concentration. Also, it was found that even 3 mol% of ZrO<sub>2</sub> significantly improve the microstructure of samples. It allows for the elimination of residual porosity due to full densification during sintering. Also, the grain size decreased from 14  $\mu$ m for pure Y<sub>2</sub>O<sub>3</sub> to 3.8–6.1  $\mu$ m for samples with 3–11 mol% of ZrO<sub>2</sub>. XRD analysis showed that the phase composition did not change between samples with 0 and 11 mol% of sintering aid. Decreasing of lattice parameters with increasing of ZrO<sub>2</sub> amount was observed due to the replacement of  $Y^{3+}$  ions in the lattice by the smaller Zr<sup>4+</sup>. A steady increase of refractive index was observed with increasing Zr content. Obtained results allow us to conclude, that the solubility limit of ZrO2 in Y2O3 was not reached within the studied range and that the presence of ZrO<sub>2</sub> improves ceramics properties. In the next step, optical transmittance was analysed. Among all obtained  $Y_2O_3$ :Zr<sup>4+</sup> ceramics the highest transmittance (78.60% at 1100 nm at a thickness of 2.11 mm) was achieved for the sample with 7 mol.% of ZrO<sub>2</sub>.

#### STRUCTURAL POSSIBILITIES OF INVERSE OPALS

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Naturally occuring opals are hydratated amorphous silica and precious opals are iridescence, this play-of-color is caused by ordered silica spheres. Inverse opals are the negative of this; a repeating pattern of void spheres are in a bulk material. The synthesis of such nanostructures uses the bottom-up preparation method; building an artificial opal using nanospheres as a sacrificial template, coating the opal, then removing it. The structure of inverse opal's cause some interesting optical properties, like the photonic band gap and the "slow" photon effect, where the latter can enhance light-matter interactions, this can be utilized for photocatalysis.

The optical properties of such an inverse opal can be adjusted by changing the initial nanospheres size and the coating materials refractive index. In this work we explore other possibilities for inrease its effectiveness or to apply it for industrial applications. Alternative lower quality (higher diameter distribution) carbon nanospheres were tested alongside with not full, but partial filling of the opal template. Combining the inverse opal with other nanostructures, such as nanorods could change the optical properties or increase the surface area. Composite inverse opals of two photocatalytic materials (ZnO and TiO<sub>2</sub>) can increase the effectiveness of the structure, also the tempalte removal heat treaments effect was investigated. Inverse opals can be stacked on top of each other, by stacking these structures and changing the top layer thickness, I investigated its effect on the optical properties.

#### ADVANCED OPTICAL ZnS AND MgF<sub>2</sub> CERAMICS: MODIFICATION OF THE SURFACE BY CARBON NANOTUBES

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At present, research in the field of the materials development using nanotechnology approach is being actively carried out. One of the promising areas is the creation of the advanced optical ceramics. It is known that oxygen-free ceramic materials, in particular ZnS and MgF<sub>2</sub>, have more stable physical and chemical properties, which permit to create the optically transparent ceramics. The zinc sulfide structures have wide area of application in the solar energy, modulation technique, and in the creation of semiconductor lasers as well. Magnesium fluoride is used to create the output window of the UV lamps for medical facilities. Nanotechnology has attracted the attention of researchers due to the significant modification of the properties of the optical materials in the nanometer scale: spectral, mechanical and refractive. In this paper, the changes of the spectral, mechanical, wetting and hygroscopic properties of the ZnS and MgF<sub>2</sub> ceramics after using an innovative method of the laser-oriented deposition (LOD) of the carbon nanotubes (CNT) on its surface were studied.

For the surface modification, a  $CO_2$  laser at a wavelength of 10.6 µm with a power of 30 W was used. Single-walled carbon nanotubes (SWCNTs type #704121 Aldrich Co.) were used as oriented nanoobjects and were deposited on the zinc sulfide and magnesium fluoride surface at the electric field strengths from 100 to 600 V/cm.

The following changes are observed as a result of the experiments. The ZnS structure modified with CNT has a slight increase in transmittance in the mid-IR region and a slight spectral shift. The results for ZnS also show a 5% increase in the mechanical strength and an increase in the wetting angle from  $102^{\circ}$  to  $106^{\circ}$ . The MgF<sub>2</sub> modified with the CNT has significant spectral shift in the UV region. Moreover, there are a 6% increase in mechanical strength and an increase in the wetting angle from  $56^{\circ}$  to  $81^{\circ}$ . In addition, quantum-chemical simulations were presented, which show that the depth of the penetration of the CNT into the surface atomic layers of the material depends on their diameter and the rate of dispersion during the laser deposition. The obtained results can be used for further application of structuring ceramics with the CNT for the liquid crystal molecules orienting in the display elements and light modulators, for optimization of the medical equipment (UV and quartz lamps) and optimization of the solar cell layers.

# PREPARATION OF ZnO/Bi<sub>2</sub>WO<sub>6</sub> HETEROSTRUCTURES VIA SURFACTANT-ASSISTED HYDROTHERMAL METHOD: CHARACTERIZATION AND PHOTOCATALYTIC ACTIVITY

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I hereby present the recent experimental findings and the key concepts of our laboratory's research on photocatalytic degradation. We synthesized 3D Bi<sub>2</sub>WO<sub>6</sub> via hydrothermal method using two different surfactants, i.e. hexadecyltrimethylammonium bromide (CTAB) and polyvinylpyrrolidone (PVP), in order to control various properties of the resulting material (particle size, morphology, crystallinity, surface area, band gap etc.). Here we explored the formation of a  $ZnO/Bi_2WO_6$  heterojunction photocatalyst for the efficient photodegradation of pollutants. A comprehensive characterization of the prepared catalysts was done by X-ray diffractometry (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), and diffuse reflectance UV-vis spectroscopy (UV-DRS). The photocatalytic activity was evaluated using phenol as the target pollutant, and the decomposition products were analyzed by using high-performance liquid chromatography (HPLC). The results revealed the distinct effects of PVP and CTAB on Bi<sub>2</sub>WO<sub>6</sub> morphology, but with a somewhat lower phenol decomposition activity. The  $ZnO/Bi_2WO_6$  heterojunction, however, exhibited tunable properties and demonstrated highly efficient phenol degradation with a remarkable degradation rate compared to that of the as-synthesized Bi<sub>2</sub>WO<sub>6</sub>.

# PHOTOCATALYTIC DEGRADATION OF REACTIVE ORANGE 16 DYE USING TiO<sub>2</sub>/PPy NANOCOMPOSITES UNDER SIMULATED SOLAR LIGHT

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It is well known that titanium dioxide is usually used as a photocatalyst due to its nontoxicity, low cost, and stability. Conductive polymer, polypyrrole (PPy) is also appropriate for photocatalytic application being stable and easy to synthesize. The aim of this study was to obtain  $TiO_2/PPy$  composites reaching higher photocatalytic efficiency compared to pure  $TiO_2$ . Therefore,  $TiO_2$  was synthesized by the hydrothermal route, while PPy was obtained by the chemical oxidative polymerization method.  $TiO_2/x\%$  PPy nanocomposites (x = 0, 0.5, 1, 1.5, 3, 5 wt.%) were prepared by hand-mixing of powders in agate mortar for 30 min in order to find the optimal PPy content. Obtained materials were characterized by XRD, FTIR, TG/DSC, FESEM, and UV-Vis methods while their photocatalytic activity was estimated towards degradation of Reactive Orange 16 dye (RO16). A kinetic study was performed and a detailed mechanism of RO16 photocatalytic degradation in the presence of  $TiO_{2}/PPy$  composites was proposed based on scavenger tests. The results showed that, despite PPy addition,  $TiO_2$  was present in anatase form in all samples with sufficiently small crystallites (around 26 nm) and preserved structure with no significant deviations in unit cell parameters. The band gap energy decreased with increasing of PPy content, from 3.14 eV for pure TiO<sub>2</sub> to 2.94 eV for TiO<sub>2</sub>/5% PPy. All the obtained nanocomposites demonstrated higher photocatalytic activity than pure TiO<sub>2</sub>, whereby TiO<sub>2</sub>/1% PPy nanocomposite was the most efficient by degrading 99.6% of the dye for 105 min under simulated solar light. Therefore, the amount of 1 wt.% should be consider as optimal amount of PPy in a composite. It is established that the photodegradation of RO16 using TiO<sub>2</sub>/PPy nanocomposites follows pseudo-first kinetic order. RO16 photocatalytic degradation mechanism in the presence of TiO<sub>2</sub>/PPy nanocomposites can be well described by direct Z-scheme heterojunction which has never been reported for TiO<sub>2</sub>/PPv system.

# OBTAINING A HYBRID ELECTRODE BASED ON IMIDAZONIUM IONTERMINATED AND METALLIC NANO-CLUSTERS AND ITS CATALYTIC ACTIVITY TOWARD HER

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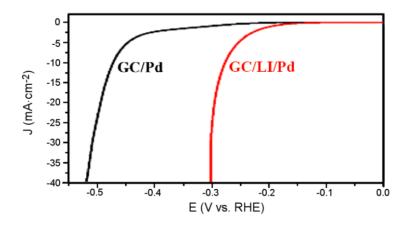
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Surface modification with thin organic layers is crucial for designing materials with specific properties to develop practical applications such as supercapacitors, metalair batteries; fuel cells, corrosion resistance.

In the present work, we focused on the grafting of thin film cationic layer (LI) on a glassy carbon electrode (GC), having labile anions. The obtained electrochemical results confirm the ionic layer grafting success as well as the metallic nanoparticles (Pt or Pd) formation. The as-prepared modified electrodes catalytic performances of towards the hydrogen evolution reaction (HER) were evaluated. Interestingly, the ionic layer presence displays an increase catalytic performances, for both investigated metallic nanoparticles, especially with palladium. This behavior is probably due to the synergistic effect existence between the grafted ionic layer and the metallic nanoparticles.



## ACID TREATED g-C<sub>3</sub>N<sub>4</sub> PHOTOCATALYSTS FOR THE PHOTOCATALYTIC REDUCTION OF Cr(VI)

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Great attention is directed towards potential visible light photocatalyst,  $g-C_3N_4$ , due to its simple preparation, high stability, low cost and medium band gap ( $\sim 2.7 \text{ eV}$ ) [1]. However, its practical application is limited, mostly because of the high recombination rate of electrons and holes. One of the methods for optimizing  $g-C_3N_4$ properties is the chemical exfoliation with concentrated acid. It was reported that treating  $g-C_3N_4$  with sulfuric acid leads to certain surface and structure modifications, which would further enhance the surface functionality and provide better dispersion of metal/semiconductor nanoparticles when forming heterojunction [1]. Different researches showed that chemical etching, i.e. exfoliating bulk  $g-C_3N_4$  to obtain nanosheets with concentrated  $H_2SO_4$  could increase the specific surface area, obtain more active sites and convert surface charge. Furthermore, introducing the electronwithdrawing groups at the edge of  $g-C_3N_4$  nanosheets by using oxidizing agent can boost electron migration and enhance the charge separation, but also expand its band gap [2]. In this work, concentrated  $H_2SO_4$  was used to exfoliate bulk g-C<sub>3</sub>N<sub>4</sub> and produce g-C<sub>3</sub>N<sub>4</sub> nanosheets (sample  $E-C_3N_4$ ) and the improved Hummer's method [2] was used for synthesis of oxygen functionalized  $g-C_3N_4$  (sample EO-C<sub>3</sub>N<sub>4</sub>).

The properties of the photocatalysts were studied by FESEM, FTIR, XRD, BET, PL and DRS analysis, as well as by determination of the point of zero charge ( $pH_{PZC}$ ) and the number of acidic and basic surface functional groups. Photocatalytic reduction of Cr(VI) was tested at a constant solution concentration (10 mg/l), at pH = 3, under the simulated visible irradiation and in the presence of citric acid as a hole scavenger. It was determited that acid treatment led to the decreasing in the specific surface area, lowering the  $pH_{PZC}$ , slightly increased bandgap energy but also enhanced photocatalytic efficiency and the number of surface functional groups.

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# PREPARATION OF Pt BASED HYDROTALCITE DERIVED Mg(AI)O SHAPED CATALYSTS VIA WET IMPREGNATION FOR PROPANE DEHYDROGENATION REACTION

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Propane dehydrogenation (PDH) is an endothermic reaction that requires relatively high operating temperatures with a high-performance catalyst for obtaining high propylene yield. Generally, the supported bimetallic Pt-based catalysts are extensively used in dehydrogenation processes. Catalyst supports such as pure and alkali promoted gamma alumina, MgO, mixed oxide with a spinel structure and hydrotalcite derived Mg(Al)O play also an important role in the propane dehydrogenation reaction. Among them, hydrotalcite derived Mg(Al)O structure seems to be much more attractive because of its basic nature, having a layered morphology with a defect-rich  $A^{3+}$  doped MgO surface and a high thermal stability up to a temperature of 800 °C. For pilot and industrial plants, the catalysts that are used in a reactor should have structured forms such as pellets, tablets, spheres and etc. Therefore, the final catalysts consisted of support that has a shaped form and active metal phases deposited onto the shaped structure. In this study, the catalysts were produced by wet impregnation of active metal precursors on commercial hydrotalcite based shaped supports. After that, the PtSn supported Mg(Al)O shaped catalysts were loaded into a fixed bed reactor. The propane dehydrogenation reaction was carried out under atmospheric pressure and at a temperature of 550 °C. The effects of Sn/Pt ratio, total Pt loading, impregnation time were evaluated in terms of propane conversion, selectivity to propylene and stability. In addition, the effects of these parameters on the structural, microstructural and chemical properties of the final catalyst were also investigated. Moreover, the reaction performances of commercial and homemade shaped hydrotalcite-based catalyst that was modified by PtSn nanoparticles were also compared. The best performing catalyst composition was determined with the Sn/Pt ratio in the range of 1.40 to 1.50 by wt/wt. which is attributed to between 2.30 and 2.50 by mole/mole for the highest PDH reaction performance.

# EVALUATING THE MECHANICAL INTEGRITY AND RELIABILITY OF MULTI-CHANNELLED FLAT-SHEET CERAMIC MEMBRANES FOR FILTRATION APPLICATIONS

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Ceramic membranes are an emergent filtration technology that is receiving increasing research attention, particularly concerning their filterability/selectivity and industrial scalability. Given the inherent brittleness and proneness to failure of ceramic materials, mechanical properties, especially those related to fracture, of ceramic membrane pieces are also important information which is nonetheless rarely evaluated. Therefore, herein a methodology for assessing the mechanical integrity and reliability of a multi-channelled flat-sheet ceramic membrane is presented. Specifically, a flat-sheet SiC ceramic membrane piece designed for submerged outside-inside vacuum-driven filtration was subjected to (i) four-point bending, (ii) tensile, and (iii) compressive tests, and the experimentally measured failure loads were converted to failure stresses and analysed by Weibull diagrams. It was found that the ceramic membrane piece is  $\sim 1.4$ times stronger and  $\sim 2.9$  times less reliable under compression than in bending, and in turn  $\sim 3.3$  times stronger and  $\sim 5.4$  times more reliable in bending than in uniaxial tension. These differences in mechanical integrity and reliability are rationalised considering the fracture modes (i.e. mode I for both bending and uniaxial tension but mode II for compression) and the critical flaw populations responsible for failure (i.e. membrane layer flaws for bending but support flaws for both uniaxial tension and compression). Finally, relevant implications for ceramic "membranologists" interested in mechanical characterization are discussed.

# HYDROTHERMALLY ACTIVATED CERAMIC MEMBRANES FOR OXYGEN SEPARATION

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Oxygen is one of the most essential chemicals and is indispensable in several industrial processes. The most common and widespread processes to separate oxygen from air are not flawless particularly in terms of energy efficiency. A promising alternative is based on dense ceramic membranes with mixed ionic-electronic conductivity (MIEC) that can separate high-purity oxygen from air at high temperatures. In these membranes, the oxygen is selectively transported through the crystal lattice of the membrane material under a driving force of oxygen chemical potential gradient.

The main objective of the proposed work was to find an alternative strategy for the surface activation of MIEC membranes for oxygen separation involving hydrothermal processing. For the first time, the hydrothermal processing was directly applied to bulk MIEC ceramic membranes to activate their surface towards oxygen exchange while avoiding their mechanical and chemical disintegration under hydrothermal conditions. The composite  $(SrFe)_{0.7}(SrAl_2)_{0.3}Oz$ , consisting of two phases, the strontium-deficient perovskite  $Sr(Fe,AI)O_{3-\delta}$  responsible for the ionic transport properties and the monoclinic  $SrAl_2O_4$  phase responsible for adequate thermomechanical properties, was considered as a model membrane material.

The hydrothermal treatment of the composite samples, included several top-down and bottom-up type approaches, leading to controlled modification of the membrane surface. These approaches were based on selective etching under alkaline conditions, followed by additional acid or mechanical treatments, and deposition of a layer of PrO<sub>x</sub>, possessing high catalytic activity in the processes involving oxygen. Detailed microstructural studies of the ceramic membranes after hydrothermal modification have confirmed the presence of desired effects, namely, the formation of etched porous SrFeO<sub>3- $\delta$ </sub> layer at the surface and deposition of PrO<sub>x</sub>. The comparative studies of oxygen permeation through non-modified and modified membranes have provided a convincing proof-of-concept for proposed strategies of hydrothermal activation. In all cases, the modified membranes showed superior oxygen fluxes compared to the non-modified membrane, making the proposed hydrothermal activation strategies very promising for increasing the performance of MIEC membranes.

# SIMULATION OF PROPERTIES OF ANISOTROPIC POROUS CERAMICS BASED ON 3D RECONSTRUCTED MICROSTRUCTURES

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The functional properties of electroceramics are strongly dependent on their porosity level, as well as on the microstructural characteristics of the existing pores (size, shape, orientation, distribution, connectivity). Specific porosity distribution and anisotropy [1] may determine peculiar strain-stress distributions, thus providing superior piezo/ferro/pyroelectric properties. In the present work, the dielectric, ferroelectric and piezoelectric properties of porous BaTiO<sub>3</sub> ceramics with anisotropic porosity are numerically estimated by using Finite Element Models [2]. Real ceramic microstructures have been reconstructed from experimentally determined microstructures based on X-ray tomography data [2,3] and used as input to determine the local potential and electric field and the strain-stress fields inside the ceramic body. By this method, peculiar features as structural defects, cracks, percolated pores can be locally investigated and their role on the material property failure can be better understood. Further, the dielectric, ferroelectric and piezoelectric properties are estimated and compared to the experimental ones. The employed approach provides a bridge from meso- to macroscale in understanding the relationship between the microstructure and material properties.

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# SINTHESIZING SiO<sub>2</sub>-ZnO COMPOSITE NANOPARTICLES FOR APPLICATION OF NANOFLUIDS

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Nanofluids are colloids in which particles added to the liquid to improve the thermophysical properties of the fluid. One of the most studied applications is to increase the thermal conductivity of the fluid to produce better heat transfer media.

The fact that the solid materials have higher thermal conductivity than conventional heat transfer fluids has been well known for a long time, but it was only three decades ago, that Choi used nano-sized particles to increase the thermal conductivity of a fluid and coined the term of "nanofluid". From this point on more and more research groups have been working on this field. Nowadays, more complex systems, such as hybrid and composite nanofluids are becoming the focus of interest.

In my study, ZnO covering was synthesized on SiO<sub>2</sub> nanoparticles (average diameter 10–20 nm) by the sol-gel method. The deposition of ZnO was carried out in ethanolic media, and base catalysis was used to control the reaction. After deposition, the particles were washed by water, and 100 ml of suspension was made. Three times 5 ml of the suspension were dried to determine the total mass of the prepared particles. The efficiency of the deposition was checked by TEM-EDX. The density of the particles was measured by a pycnometer. Water was chosen as basefluid due to its high thermal conductivity, low viscosity, and low cost. The stability was checked by visual observation, and aggregation was detected. Different surfactants (at 10w/w% of particles) were tried to prevent aggregation. The sodium carboxymethyl cellulose (CMC) caused the higher increase in stability. Zeta-potential was measured to quantify the aggregative stability of nanofluids, and it was -55.5 mV for the CMC stabilized nanofluid. The size of the aggregates was measured by DLS, and it was 195 nm. Nanofluids were prepared with particle content of 0.5 and 1.0 v/v%. The viscosity was measured by a rotation viscosimeter. 0.5% of particles increased the viscosity of the water by 80%. The thermal conductivity of the composite nanofluids was higher than nanofluids with simple SiO<sub>2</sub> nanoparticles.

# RELATIONSHIP BETWEEN BENTONITE INTERNAL AND EXTERNAL SURFACE AREA AND ITS PERFORMANCE IN WINE CLARIFICATION

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Developed protein haze in bottled white wine is considered a serious quality defect and such wines are not marketable. It originates mainly from denaturation and aggregation of the so-called pathogenesis-related (PR) grape proteins, consisting mainly of thaumatin-like proteins and chitinases. The practice of choice universally employed to prevent protein haze in wine is their removal before bottling by adsorption onto bentonite, a natural clay-based mineral belonging to the group of montmorillonites (aluminum hydrate silicates) with a lamellar structure alternated with exchangeable cations and hydration water. Although effective, the use of bentonite in winemaking has several drawbacks, such as loss of wine volume and stripping of aromas, so it is of interest for the winemaking industry to identify bentonite types with reduced negative impacts.

To investigate the relationship between bentonite internal and external surface area and its performance in wine clarification, eight Na-based natural/activated bentonites and a Ca/Na-based bentonite were applied for protein stabilization of Malvazija istarska wine at laboratory scale. Their internal surface area was determined by OIV methylene blue test, while external specific surface area was calculated based on nitrogen adsorption-desorption isotherms using the BET method. Bentonite doses were determined by the standard heating test. Clarified wines were subjected to the analysis of phenols (RP-HPLC-DAD), volatile aromas (HS-SPME-GC/MS), and elements (ICP-OES). The majority of applied bentonite doses significantly reduced the concentration of most volatile esters, while the levels of *trans*-caftaric and *trans*-coutaric acids were preserved. All the bentonites increased the concentration of Na, while particular ones released additional amounts of Ca, Mg, and Mn into wine. Negative correlation was determined between internal surface area and bentonite dose, especially when only Nabased bentonites were considered. When expressed per gram of bentonite dose, the reduction of many important volatile esters (%) showed medium to strong positive correlation (r > 0.5) with internal surface area. Somewhat the opposite was observed for external surface area: after excluding a particular Na-based bentonite sample as an outlier, strong negative correlation with volatile aroma reduction rate (%) was observed. It was concluded that physical properties of bentonite, such as internal and external surface area are possibly among the factors that determine bentonite performance in wine clarification, including its efficacy in protein removal and side-effects.

# VISIBLE LIGHT DRIVEN PHOTOCATALYTIC CERAMIC BASED NANO-COMPOSITES WITH ANTIBACTERIAL ACTIVITY

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Intending to invent an efficient self-cleaning solution for inner spaces, the aim of this research was to develop a pure visible light driven photocatalytic suspension which can be applied onto interior walls and wall paints. Ceramic based nano-composite layered double hydroxides (LDH were used to carry metal-doped TiO<sub>2</sub>. As titania is a very good photocatalyst, although active only in the UV region, doping with transition metals was used for the reduction of the activation (band-gap) energy. The series of molybdenum doped and tungsten doped TiO<sub>2</sub>-ZnAl-LDH suspensions were synthesized using a modified low supersaturation co-precipitation route at various pH values and with different dopant to titanium ratio.

Detailed material characterization studies by means of scanning and transmission electron microscopy (SEM/TEM), thermogravimetry (TG), dynamic scanning calorimetry (DSC), as well as microstructure analysis by X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR) were performed. The photocatalytic selfcleaning properties of the optimally synthesized  $TiO_2$  doped LDH suspension were assessed when applied on stone, brick, wall paint and glass substrates after having been illuminated only with LED visible light. Finally, the antibacterial activity of newly developed sole visible light driven photocatalytic ceramic based nano-composites was proven.

**Acknowledgements:** The authors would like to acknowledge the support from Ministry of Science, Technological Development and Innovations (Republic of Serbia), project No. 451-03-47/2023-01/200134 and from the EUREKA project E!13085 CAPTAN.

# PHOTOCATALYTIC PERFORMANCE OF TiO<sub>2</sub>-COATED ALUMINUM FOIL FOR DEGRADATION OF RHODAMINE B IN WATER

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Water contamination from organic pollutants, particularly synthetic organic dyes, significantly threatens water quality and human health. These dyes, widely utilized in textiles, printing, and photography industries, are characterized by their large-scale production, toxicity, and slow biodegradation. Rhodamine B is particularly concerning among these dyes due to its detrimental effects on aquatic life and its carcinogenic properties in humans and animals. Consequently, the efficient degradation of Rhodamine B is crucial for water purification and conservation efforts.

In this study, we aimed to develop TiO<sub>2</sub>-coated aluminum foils using different methods to achieve efficient and environmentally friendly self-cleaning surfaces. Such surfaces have the potential to revolutionize water purification systems. We employed two approaches for coating the aluminum foils with TiO<sub>2</sub>. The first method involved using the spray method to deposit TiO<sub>2</sub> monolayer films onto the foil surfaces. In contrast, the second method utilized a mixture of TiO<sub>2</sub> and white acrylic paint, which was coated onto the foils. The prepared foils were then heated in an oven at 200 °C for 15 min.

We analyzed the kinetic curves of Rhodamine B degradation to evaluate the efficiency of the photodegradation processes. The degradation procedure was conducted under UV-LED irradiation. To further enhance the photocatalytic activity of the foils, plasma treatment was applied after the heating process to activate the  $TiO_2$  layer better (plasma treatment aimed to optimize the performance of the photocatalytic system).

The efficient degradation of Rhodamine B using  $TiO_2$ -coated aluminum foils holds promise for addressing water contamination challenges and promoting sustainable water treatment methods.

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# LEVERAGING DEFECTS TO PROMOTE DUAL EXSOLUTION ON $(BaLa)_{1-x}Ag_xCoFeO_{6-\delta}$

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Slow surface reaction kinetics are a limiting factor in the electrochemical performance of air-electrodes and increases the concentration polarization which results in increased cathodic losses [1]. Electrochemical processes occur at the double-phase boundaries of triple-conducting oxides. Thus, it is essential to increase the number of reaction sites many-fold. To improve the rate of oxygen reduction reaction on the cathode's surface, several approaches such as infiltration and physical vapor deposition of noble metallic catalysts have historically been explored with less desirable outcomes. Infiltration and deposition techniques have a tendency to produce agglomerated and unevenly distributed nanoparticles [2]. The results can also be non-reproducible. As such, this has created the need for 'intelligent' catalysts using a novel approach known as exsolution [1,2]. Exsolution of nanoparticles refers to the spontaneous expulsion of catalytically active cations from either the A-site or B-site of the host perovskite. They co-segregate with oxygen vacancies, diffuse through the lattice and nucleate to form partially submerged metallic or oxide nanoparticles on the surface [2]. The exsolution method is deemed to be an efficient way to produce resilient and evenly distributed catalytically active nanoparticles. Exsolution can be achieved by reducing conditions, lattice strain or via voltage induction [1,2].

In this work, we present the results of dual exsolution achieved from both the Asite and B-site of silver doped  $(BaLa)_{1,x}Ag_xCoFeO_{6-\delta}$  (x = 0.04, 0.10 and 0.20) perovskite. Notably, the exsolution of nanoparticles is often achieved from either the Asite or B-site of the perovskite structure [2]. The concept of dual exsolution is enabled by the ease in reduction of Ag cations to their metallic state. At the right temperatures in reducing conditions, this leads to exsolution of metallic Ag from the A-site, creating a non-stoichiometric compound. Due to the resultant A-site deficiency, subsequent exsolution of B-site transition metals follows which results in a mixture of catalytically active metal and oxide nanoparticles at the surface. Given that not all exsolved nanoparticles will produce electrochemically active sites, it is crucial to have a high density of nanoparticles. Moreover, silver nanoparticles exhibit good catalytic effects even at room temperature [3]. Cobalt oxide nanoparticles equally have high catalytic activity that could improve electrochemical activity on the cathode [4]. The aliovalent substitution of  $Ag^+$  and  $Ba^{2+}$  in the A-site of the host perovskite not only increases hydration properties but also increases the formation of oxygen vacancies which are essential for exsolution [4]. The presence of multi-valent Co and Fe cations enable electronic conductivity. Hence, the host perovskite has desirable ionic-electronic properties [4]. The results to be presented include scanning electron microscopy

micrographs, x-ray diffraction patterns, thermogravimetric data, hydration, iodometric titration, total ionic-electronic conductivity in dry and wet air. In addition, the chemical diffusion and reaction co-efficients from conductivity relaxation measurements in oxidizing and reducing atmospheres will also be presented.

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# CARBON NANOTUBES-BASED THIN FILMS PREPARED BY LOW-COST TECHNIQUE FOR BIOSENSING APPLICATIONS

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The detection of bacteria is crucial when it comes to areas, such as food safety, environmental monitoring, public health, etc. Hence, developing a simple, affordable, highly sensitive device for recognizing specific bacteria could have a serious impact on mankind. In that sense, novel solutions for bacteria detection are based on biosensors, novel devices that use a biorecognition element for specific target detection to enhance the sensitivity of biosensors through a large surface area suitable for the immobilization of biorecognition elements.

It is well known that electrical and optical properties of CNTs are very sensitive on exposure to biomolecules which is why these materials are also suitable as sensing element in the biosensors. In this work, the properties of CNTs-based thin films will be investigated with the potential application as an electrochemical immunosensor for *E. coli* detection.

For the fabrication of an inexpensive CNTs-based immunosensor, we used a simple, low-cost layer-by-layer (LbL) assembly technique. LbL deposition method implies alternating deposition of positively and negatively charged layers in order to form electrostatic bond between them. Custom-made gold leaf-based electrodes were used as a substrate and deposition method was alternate deposition of polyethyleneimine and carboxylic functionalized single-walled carbon nanotubes.

Horiba XploRA Plus Raman spectrometry and Thermofisher Scientific Apreo C High Resolution Scanning Electron Microscope (HRSEM) were used for examination of structural properties of fabricated CNT-based thin films on gold electrodes. Electrical

properties were investigated with Ecopia HMS-3000 Hall device and Hewlett Packard multimeter. Electrochemical properties were evaluated using cyclic voltammetry and electrochemical impedance spectroscopy and the potential of the PEI-SWCNT film was examined for biosensing applications by immobilizing antibodies for *E. coli* detection.

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## SYNTHESIS OF Ti<sub>3</sub>C<sub>2</sub>T<sub>x</sub> AND ITS POTENTIAL USE IN WATER PURIFICATION PROCESSES

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A group of drugs such as  $\beta$ -blockers are among the most commonly prescribed drugs worldwide. Their excessive use leads to a shortage of drinkable water resources. In addition to stability in water, the problem is also their conjugates, which often return to the original compounds, so it is necessary to remove  $\beta$ -blockers from water. Photocatalytic degradation compared to conventional methods of water purification has been shown to be an effective method for the removal of drugs from the aqueous medium [1].

MXenes are carbides, nitrides and carbonitrides of transition metals, and represent a rapidly developing group of 2D materials, where  $Ti_3C_2T_x$  is the most studied material [2].  $Ti_3C_2T_x$  could be suitable for photocatalytic water purification because of the large and aboundant surface area covered by the large number of hydrophilic terminal groups (-F, -O and -OH) that enable drug adsorption [3]. In this work, the synthesis of singlephase  $Ti_3C_2T_x$  from  $Ti_3AlC_2$  was performed using HF obtained *in situ* in a mixture of LiF and HCl. The synthesized material was characterized by X-ray diffraction (XRD), Raman spectroscopy and BET analysis. In our research, the photolysis and photocatalysis of the  $\beta$ -blocker PIN were carried out in  $Ti_3AlC_2$  and  $Ti_3C_2T_x$  aqueous suspensions under the influence of UV radiation. Also, under the same experimental conditions, without UV radiation, the adsorption of PIN on the  $Ti_3C_2T_x$  surface was examined. The photolysis kinetics of photocatalysis and adsorption were monitored by HPLC analysis. The results show that PIN is stable during photolysis and that pure  $Ti_3C_2T_x$  is not photocatalytically active due to the challenge of band gap tailoring.

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# ENERGY STORAGE PROPERTIES OF (1-x)Pb(Fe<sub>0.5</sub>Nb<sub>0.5</sub>)O<sub>3</sub>-xBiFeO<sub>3</sub> BULK CERAMICS AND CERAMIC THICK FILMS

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Energy storage is the ability to collect and store energy for use at another time. Dielectric capacitors have higher power density compared to batteries and are good candidates for energy storage in pulsed systems. Among different dielectric materials, the most promising are the materials with slim and pinched polarization versus electric field (*P*–*E*) hysteresis loops [1]. Such properties are obtained in  $(1-x)Pb(Fe_{0.5}Nb_{0.5})O_{3}-xBiFeO_{3}$  (PFN–100*x*BFO) solid solutions [2]. Compared to bulk ceramics, thick films withstand higher electric fields, which makes them even more promising for energy storage applications [3].

In this work, we investigated the energy storage properties of PFN–100*x*BFO bulk ceramics and thick films. PFN–100*x*BFO (x = 0–0.5) powders were prepared by mechanochemical synthesis. The isostatically pressed pellets were sintered in an oxygen atmosphere at 950 °C (x = 0–0.1) and at 900 °C (x = 0.2–0.5) for 2 hours. The recoverable energy storage density ( $U_{rec}$ ) and efficiency ( $\eta$ ) were calculated for all compositions. The best properties were determined for the composition x = 0.3, which exhibits slim and pinched *P*–*E* hysteresis loop. The  $U_{rec}$  and  $\eta$  values at 70 kV·cm<sup>-1</sup> are 1.0 J·cm<sup>-3</sup> and 80 %, respectively. Furthermore, PFN–30BFO thick films were prepared by screen-printing the thick-film paste on golden Al<sub>2</sub>O<sub>3</sub> substrates. The samples were sintered in an oxygen atmosphere at 800 °C for 2 hours. The  $U_{rec}$  value of the samples is 2.2 J·cm<sup>-3</sup> at 350 kV·cm<sup>-1</sup>, which is higher compared to the  $U_{rec}$  value of the bulk ceramic due to the higher electric field. On the other hand, the  $\eta$  value for the thick films at 350 kV·cm<sup>-1</sup> is 54 %, which is lower than that of the bulk ceramic due to the wider *P*–*E* hysteresis loop.

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# IMPACT OF MAGNETIC FIELD STRENGTH ON THE CATALYTIC ACTIVITY OF CHEMICAL VAPOR DEPOSITION (CVD) SYNTHESIZED CoFe<sub>2</sub>O<sub>4</sub> THIN FILMS FOR ELECTROCHEMICAL OXIDATION OF NITROGEN

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Nitrates have extensive applications as both fertilizers and oxidizing agents. Traditional commercial nitrate production methods from nitrogen entail complex multistep processes conducted at high temperatures and pressures. Hence, the significance of an alternative approach for nitrate production under ambient conditions is paramount [1–3]. Herein, a  $CoFe_2(O'Bu)_8$  complex was synthesized and employed as a precursor for the production of  $CoFe_2O_4$  thin films via magnetic field-assisted Chemical Vapor Deposition (mf-CVD). The influence of external magnetic fields on the microstructure and anisotropy of the  $CoFe_2O_4$  thin films was investigated, along with a detailed characterization through XRD, SEM, EDX, and XPS. Moreover, the electrochemical nitrogen oxidation reaction (NOR) approach was developed with these thin films and revealed their excellent catalytic performance. The most striking aspect of this research lies in the discovery that the application of an external magnetic field led to changes in specific planes within the crystalline cobalt ferrite film, resulting in altered electrocatalytic activity during nitrogen oxidation.

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# EFFECT OF POST-ANNEALING ON MICROSTRUCTURE AND FERROELECTRIC PROPERTIES OF LEAD-FREE BaTiO<sub>3</sub> THICK FILMS ELABORATED BY AEROSOL DEPOSITION METHOD

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The field of piezoelectric materials has experienced significant growth in recent decades. These materials are highly valuable due to their ability to convert mechanical energy into electrical energy and vice versa, enabling a wide range of applications including energy harvesters, pressure sensors, and micro-actuators. In compliance with the European directive on hazardous substances (RoHS), there is a strong drive to develop lead-free alternatives to high-performance PZT materials for certain applications. To realize piezoelectric energy harvesting devices, the fabrication of thick films with thicknesses ranging from 1 to 100  $\mu$ m is technically required. Aerosol Deposition (AD), an emerging deposition technique, offers the capability to deposit thick films within this desired thickness range.

In this study, BaTiO<sub>3</sub> thick films were successfully deposited onto glass and kovar substrates using the AD method at room temperature. The as-deposited films exhibited a linear dielectric-like behavior, with high resistivity values indicative of the excellent densification of the deposited BaTiO<sub>3</sub> films. The absence of ferroelectricity in the as-deposited films can be attributed to the size effect. To recover the desired piezoelectric and ferroelectric properties, post-deposition annealing trials were conducted at different temperatures in air and under an argon flux. The annealing process successfully restored ferroelectricity above 800 °C, where grain growth became possible and residual stress was further released compared to lower temperatures (< 800 °C). The influence of the annealing atmosphere became particularly significant at 900 °C, as the low oxygen atmosphere promoted greater grain growth. However, the presence of residual oxygen vacancies in BT film annealed under an argon flux led to reduced fatigue resistance. Thus, a compromise must be made between enhanced ferroelectricity and the dynamic performance of post-annealed BT films.

# POROSITY EFFECTS ON THE FUNCTIONAL PROPERTIES AND PIEZOELECTRIC HARVESTING PERFORMANCES OF BCTZ CERAMICS

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In this work, the effect of porosity on the structural and functional (dielectric, ferroelectric, non-linear, and piezoelectric) properties in Ba<sub>0.85</sub>Ca<sub>0.15</sub>Ti<sub>0.90</sub>Zr<sub>0.10</sub>O<sub>3</sub> (BCTZ) ceramics was investigated. Various levels of microporosity in the range from 3% to 31% have been produced using poly(methyl-methacrylate) (PMMA) microspheres as sacrificial templates. The structural investigation indicates a phase coexistence at room temperature; the amounts of polymorph change with porosity level. The maximum permittivity decreases with increasing porosity, from around 7000 (ceramic with 3% porosity) down to 3500 (ceramic with 31% porosity), and Curie temperature shifts from 47 °C to 67 °C when increasing porosity, related to the possible porosity-induced structural and internal stress modifications. An enhanced piezoelectric response was found in the BCTZ ceramic with intermediate porosity around 18 vol.%, with the highest value of piezoelectric response of 470 pC/N and corresponding figure of merit of 7.3  $pm^2/N$ . The optimum piezoelectric properties at the intermediate porosity level are related to the microstructural changes (pore shape and connectivity) and to possible field-induced structural modifications. The piezoelectric energy harvesting results have shown the possibility of using Pb-free porous ferroelectric materials in devices for piezoelectric energy harvesting applications.

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# HYDROTHERMAL SYNTHESIS OF SBN AURIVILLIUS CERAMICS FOR FERROELECTRIC APPLICATION

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 $SrBi_{2-x}RE_xNb_2O_9$  (RE = Y, Nd, Gd) ceramics were prepared by Hydrothetrmal process treatment. Crystal structure of bismuth layer-structured ferroelectrics (BLSFs) was studied by powder X-ray diffraction technique. Plot patterns well matched with reported data and the lattice parameters of the samples were calculated using two method. Pellets of 10 mm diameter and thickness about 1 mm~2 mm were prepared and sintered. The surface morphology of the samples was studied from scanning electron microscope pictures. The properties of all the compositions were investigated and found to be greatly affected, Partial substitution of bismuth by RE changed the ferroelectric–paraelectric transition temperature, once the ferroelectric–paraelectric phase transition diffuse, the Curie temperature shifts toward lower temperatures typically from 395 to 280 °C.

Keywords: ferroelectric, ceramics, SBN, rare earth.

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## MAGNETO-ORIENTATION OF BULK MgB<sub>2</sub> SUPERCONDUCTOR

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Magneto-processing of ceramics can be a useful method to obtain textured bulk materials with improved properties. The ceramic subjected to the orientation with magnetic field must present anisotropy of magnetic susceptibility and/or of shape. MgB<sub>2</sub> is a practical superconducting material. It has a hexagonal crystal structure being composed of boron and magnesium layers stacked in the *c*-axis direction. In early studies, it was claimed that powders of MgB<sub>2</sub> cannot be properly oriented in a high magnetic field. In this work, a colloidal solution of MgB<sub>2</sub> powder in ethyl alcohol is ultrasonicated. After that the colloidal solution is poured into plastic containers under high magnetic field of  $H_{\text{texturing}} = 12$  T and the as obtained greenbody has according to X-

ray diffraction a partial *c*-axis texture. The orientation of the sample attains values between 21 - 40 %, significantly higher than in the dried greenbodies. Results suggest a template growth mechanism. Samples were characterized from the view point of critical current density and irreversibility field by magnetic measurements with the applied field  $H_{appl.}$  in parallel ( $H_{appl.}$  //  $H_{texturing}$ ) and perpendicular ( $H_{appl.} \perp H_{texturing}$ ) directions relative to the surface of the disc samples.

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## NOVEL BORON CARBIDE COMPOSITES SINTERED AT LOW TEMPERATURE

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The attainability of fabricating ground-breaking boron carbide-silicon carbide composites by spark-plasma sintering (SPS) of boron carbide + silicon powder mixtures at only 1400 °C was examined. To start with, it is exhibited that boron carbide can be fully densified at 1400 °C if ~20 vol.% silicon aids are used, leading to bi-particulate composites constituted by boron carbide (major phase) and silicon carbide (minor phase). The formation of these composites is due to the fact that silicon acts as a reactive sintering additive during SPS. Lower and higher proportions of silicon aids are not optimal, the former leading to porous bi-particulate composites and the latter to dense triplex-particulate composites with some residual free silicon. Importantly, it is also shown that these ground-breaking boron carbide-silicon carbide composites are finegrained, nearly-ultrahard, moderately tough, and more affordable to fabricate, a combination that makes them very appealing for many engineering applications. Also, it is demonstrated that during the heating ramp of the SPS cycles a eutectic melt is formed that promotes full low-temperature densification by transient liquid-phase sintering if sufficient silicon aids are used. Otherwise, a subsequent stage of solid-state sintering is required at higher temperatures once the eutectic liquid has been consumed in the *in-situ* formation of silicon carbide. And finally, it is demonstrated that during SPS the original boron carbide goes through a gradual isostructural crystallographic transition towards a silicon-doped carbon-deficient boron carbide that is more relevant with increasing proportion of silicon aids, and it is determined that the carbon source for the formation of silicon carbide is almost exclusively the carbon exsoluted from the boron carbide crystals themselves during their isostructural transition.

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#### COMPOSITES FROM THE TiB<sub>2</sub> - MoSi<sub>2</sub> - C SYSTEM

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Titanium diboride  $(TiB_2)$  with the AlB<sub>2</sub>-type structure is one of materials classified as high-temperature ceramics. Due to strong covalent bonds present in the  $TiB_2$  structure, titanium diboride has a number of unique properties, but it is also a poorly sinterable material. Obtaining dense  $TiB_2$  polycrystals requires the use of chemical and physical sintering activation.

The paper presents research on sintering of TiB<sub>2</sub>. Sinterability of composites based on TiB<sub>2</sub> with different amounts of carbon (0-4 mass%), different amounts of MoSi<sub>2</sub> (2.5, 5.0 and 10 mass%) and both carbon and MoSi<sub>2</sub> (2.0 mass% C+2.5; 5.0 and 10 mass% of MoSi<sub>2</sub>) additives were studied. The composites were manufactured using a hot-pressing technique (HP). It was found that, when the carbon addition is larger than 1% and sintering temperature is 2100°C it is possible to obtain high-density polycrystals, with densities greater than 95%. In contrast, the composites with densities close to 100% can be produced at 1800 °C when the addition of MoSi<sub>2</sub> is not less than 5%. By combining both additives, i.e. carbon and MoSi<sub>2</sub>, fine-grained, dense composites can be produced at 1800 °C (Fig. 1). Vickers hardness of all dense composites, irrespective of the amount of carbon, MoSi<sub>2</sub> and both additives, is high and in the range of 23–27 GPa.

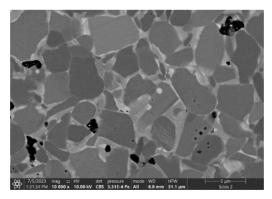


Figure 1. Microstructure (SEM) of the  $TiB_2+2\% C+5\% MoSi_2$  composite

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# RAPID PRESSURELESS SINTERING OF CELLULOSE NANOFIBRE BASED CERAMIC MATRIX COMPOSITES

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The Discovery of novel materials is necessary for implementations in futuristic sustainable applications. Ceramic matrix composites open up a wide range of applications where the beneficial refractory properties of ceramics can be combined with additional functionalities like electrical conductance and high mechanical strength. Complex-shaped composite structures are needed to miniaturize devices. decentralize/domesticate technologies, and improve static structures. Carbon nanotubes and graphene oxide are widely used to develop ceramic composites to take advantage of their nano size, good electrical properties, and structural reinforcement properties. However, their origin as a petroleum by-product makes the process unsustainable. In a sustainable approach to developing similar structures of ceramic-carbon composites, a recent study [1] demonstrated the in-situ graphitization of naturally derived cellulose nanofibres in a ceramic matrix to create a percolating electrical network across the ceramic composite. It was established that the in-situ graphitization was possible via sintering in a pressurized environment (spark plasma sintering - SPS).

The current study aims to mimic this graphitization in additively manufactured (thermoplastic 3D printed – T3DP) complex structures in a pressureless environment to retain their geometry. This is done by the sole effect of radiation-based rapid heating. For this, a modified version of the SPS is employed where the heat transfer to the sample is solely through radiation from the chamber walls. Very high heating rates (33 °C/min to 900 °C/min) to the sintering temperature (1550 °C) are employed for this process. The success of the graphitization during the rapid sintering processes at different heating rates are measured via characterization techniques such as scanning electron microscopy (SEM) imaging, x-ray diffraction spectroscopy (XRDS), and Raman spectroscopy. The products prepared from this technology will find their applications as self-heatable catalyst frameworks in reaction chambers to produce hydrogen.

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OC-74

# OXIDATION AND THERMAL PROPERTIES OF INCONEL 625 – NIOBIUM CARBIDE SYSTEM

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The recently developed Inconel 625 – niobium carbide system is a promising metal matrix composite (MMC) for high-temperature application if obtaining technology was to be improved [1,2]. In this talk, the influence of the weight percent of niobium carbide addition on oxidation performance and thermal properties is discussed. Additionally, the positive influence (i. e. mechanical property improvement) of such addition is added for the context of potential applicability. Phase and microstructural analyses, oxidation kinetics study, and thermal properties of Inconel 625-NbC MMC and pure Inconel 625 obtained by Spark Plasma Sintering are shown in this work. Then, the authors discuss their plans regarding narrowing and increasing the peak addition of niobium carbide in Inconel 625, creating superalloys instead of composite within the proposed system.

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# ELECTROLESS Ni-P FILM DEPOSITION – SURFACE TREATMENT ON ALUMINA AND POLYAMIDE

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The study examined the morphology and adhesion strength of an autocatalytic electroless nickel phosphorus (Ni-P) film. This film was deposited at a temperature of 40 °C, 50 °C and 60 °C onto both polished alumina (Al<sub>2</sub>O<sub>3</sub> 99.6%) and polyamide film (Kapton) substrates. The substrates underwent a series of treatments, including sulfur/nitric solution pretreatment, sensitization with acid stannous chloride, and activation using palladium chloride solutions. The investigation employed various techniques such as contact angle (CA) measurements, scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), a mechanical profilometer (DekTak), and direct laser writer ("maskless lithography"). The results of this study revealed that the treated polished Al<sub>2</sub>O<sub>3</sub> and polyamide (Kapton) substrates exhibited remarkable performance in facilitating the electroless deposition of a thin Ni-P film at a low temperature (40 °C, 50 °C and 60 °C). Consequently, this process resulted in more compact and continuous films characterized by small grain size and strong adhesion.

Keywords: alumina, kapton, electroless nickel-phosphorus, wet surface treatment; thinfilm, sensors

# AN ALL-CARBIDE TRIPLEX PARTICULATE CERAMIC COMPOSITE FOR TRIBOLOGICAL APPLICATIONS

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An all-ceramic triplex particulate ceramic composite with equal volume fractions of  $B_4C$ -SiC-TiC was fabricated by first time by solid-state spark plasma sintering from commercially available powders, optimizing their densification temperature, measuring their mechanical properties, and investigating the unlubricated sliding wear of the optimally SPS-ed composite. It was found that the  $B_4C$ -SiC-TiC triplex-particulate composite is hard and wear resistant, and therefore a candidate material for use in tribological applications.

OA-77

# TAILORING SILICA NANOCARRIERS TO OVERCOME HYDROPHOBIC DRUG CHALLENGES - AN INVESTIGATION INTO ENHANCED MOLECULAR WEIGHT AND HYDROPHOBICITY MODULATION

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Hydrophobic drugs play a critical role in targeting specific receptors or enzymes within lipidrich cell membranes, but their limited bioavailability and circulation time due to insolubility in aqueous media present significant challenges. To enhance the blood circulation time of hydrophobic drugs, they can be loaded into biocompatible and hydrophilic carriers, thereby improving drug uptake. In this study, we synthesized mesoporous silica (mSiO<sub>2</sub>) nanocarriers with different morphologies (cubes, capsules, and spheres of sizes 60 and 300 nm) and functionalized their surfaces with long alkyl chain hydrocarbons, such as octadecyltrimethoxysilane, to convert them into hydrophobic carriers ((OCH<sub>3</sub>)<sub>3</sub>Si(CH<sub>2</sub>)<sub>18</sub>)@mSiO<sub>2</sub>). These hydrophobic nanocarriers demonstrated up to 80% uptake of hydrophobic drugs; however, a significant proportion of the drugs were found to release uncontrollably within 6 hours. To address this issue, we successfully prolonged the drug release control by encapsulating the drug-loaded nanocarriers with a pH-sensitive lipid overlayer. The lipid-coated nanocarriers exhibited

prolonged drug release for up to 72 hours (compared to 6 hours for uncoated nanocarriers), making them suitable for achieving longer blood circulation times. Furthermore, we observed that the shape and size of the nanocarriers (i.e., cubes, capsules, spheres) influenced the drug entrapment capacity and release behaviour. Moreover, the molecular weight and chemical structure of the drug molecules were found to significantly impact drug uptake and release patterns. Overall, our findings highlight the promising potential of silica nanoparticle-based hydrophobic carriers, and their tailoring with lipid overlayers, to overcome the challenges associated with hydrophobic drug delivery and improve drug release kinetics for enhanced therapeutic efficacy.

**Keyword:** mesoporous silica nanocarriers, hydrophobic drugs, long chain hydrocarbon, lipid overlayer, drug molecular weight

OA-78

# FLUOROMETRIC DETERMINATION OF LYSOZYMES WITH APTAMER MODIFIED SILICA NANOPARTICLES

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Aptamers are short DNA or RNA sequences that can bind specifically to proteins or other biomolecules. They can be used as inhibitors to influence the activity of proteins or to visualize certain cells. However, aptamers have some challenges due to their poor cell penetration, unstable structure and fast degradation by nucleases. Silica nanoparticles (SiNPs) can act as a gatekeeper for aptamer because they can improve cell penetration, release, and stability of the aptamer. In this paper we functionalized alkyne modified SiNPs with fluorescein isothiocyanate (FITC) labeled aptamer by a click reaction. A fluorometric assay was developed for the detection of the enzyme lysozyme. It is based on the hybridization of FITC labeled aptamer with its complimentary DNA strand containing a black hole quencher (BHQ), resulting in fluorescence quenching of the FITC due to the close proximity of the fluorophore FITC and BHO. After addition of the lysozyme, the BHQ labeled aptamer was displaced by lysozyme, causing fluorescence restoration of FITC. Fluorescence measurements ( $\lambda ex/em = 495/519$  nm) showed that the emission of FITC could be suppressed by the hybridization with the BHQ labeled aptamer. After the addition of the enzyme lysozyme, the fluorescence could be restored. This aptamer based fluorescent assay can be extended on other proteins and enables the identification of biomarkers for specific diseases, where SiNPs serve as gatekeeper for the aptamer.

Key words: aptamer, silica nanoparticles, fluorescence, hybridization, FITC, black hole quencher

#### OC-79

# BIOSILICA DERIVED FROM AGRICULTURAL AND INDUSTRIAL WASTE FOR DEVELOPMENT OF NANO-SILICA/POLYMER COMPOSITES FOR APPLICATION IN VARIOUS FIELDS

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Agricultural waste biomass (wheat straw ash) and industrial wastes (fly ash) were used as natural sources of silicon precursor using alkaline treatment to form a Na<sub>2</sub>SiO<sub>3</sub> solution. It was shown that different sources of silicon precursors influence on characterristics of derived silica nanoparticles. Silica particles obtained from wheat straw ash (SWS) are spontaneously nucleated and precipitated from sodium silicate solution and were monodispersed with uniform size of ~600 nm, having total pore volume of 0.164 cm<sup>3</sup>/g and average surface area of 23.13 m<sup>2</sup>/g. On the other hand, silica particles obtained from fly (SFA) ash were generated by acidic neutralization of sodium silicate solution and have irregular particle size with average pore size of 10.2 nm, total pore volume of 0.513 cm<sup>3</sup>/g and surface area of 201.0 m<sup>2</sup>/g. SWS were used as filler for polyvinilacetate adhesive and tune the adhesives' different properties like rheology and bonding strength. On the other hand, SFA were used for preparation of alginate-silica-microbial cell composite materials which were used as biosorbents for efficient removal of heavy metals from water.

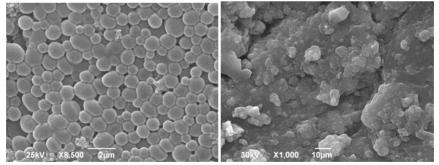


Figure 1. Micrograph of silica particles derived from wheat straw and dispersed in polyvinilacetate adhesives (left) and micrograph of silica particles derived from fly ash (right)

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OC-80

# A MODIFIED PHOTO-ACTIVATION PROTOCOL OF A HIGHLY-FILLED DENTAL COMPOSITE USING OPTICAL FIBERS

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The study aimed to introduce a novel photo-activation protocol of dental composites using optical fibers inserted into the filling, for reducing polymerization shrinkage stress (PSS) and guiding light directly into deeper layers of the restoration.

Standardized tooth cavity models were filled with a commercial dental composite (dimethacrylate-based organic phase; F-B-Al-Si inorganic phase: 87 wt.%), and the proposed curing was designed as follows: in the 1<sup>st</sup> step, two optical fibers ( $\phi$  1 mm) connected with a dental LED curing unit were inserted into the filling to cure the composite from within; in the 2<sup>nd</sup> step, fibers were extracted, remaining voids were filled and final conventional curing was performed with the commercial light guide positioned above the sample. As a control, conventional curing was performed on a separate group of samples. Tooth model deformation as a secondary manifestation of PSS was measured in real-time using digital holographic interferometry. Simultaneous monitoring of composite temperature change was conducted with an infrared thermal camera. The degree of monomer-to-polymer conversion (DC) was measured immediately after curing and after 24 h of dark storage, using Raman spectroscopy.

The results showed a 17% reduction of model deformation using the proposed curing compared with the control. This was associated a lower temperature change (20%), leading to a lower thermal shrinkage during the cooling process. Moreover, the time to reach the maximum exothermal temperature was prolonged compared with the control, allowing polymer chains to re-arrange and accommodate the volume reduction by viscous flow. Meanwhile, significantly higher immediate DC was measured (41.8 % vs. 32.7%) for the experimental group of samples. After 24 h of dark storage, the DC increased in both groups as expected, and no significant difference in DC was noticed. In conclusion, the proposed photo-activation using optical fibers was presented as beneficial for PSS reduction, with a potential of improving DC. It should be noted that the protocol can be easily implemented into dental clinical practice by coupling the fibers with standardized commercial light guides.

# BIO-STABILIZATION OF EARTH ELEMENTS BASED ON INDUSTRIAL WASTES

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The demands of innovative, cost effective and sustainable construction practices require the development of energy-efficient and environmental friendly building techniques. Rammed earth is an ancient building technique where soil constrained within formworks is pressed together in lifts. Rammed earth possesses low embodied energy, high recyclability, and low toxicity while having little impact on biodiversity and virtually no depletion of biological nutrients. The goal of this work was to significantly reduce the atmospheric  $CO_2$  emissions and waste associated with the production and application of Portland cement by exploring bio-stabilization of earth elements to enhance the material properties. Building materials that do not require thermal treatment were designed based on low grade clay and waste materials (cuticle, shell of river crabs) and industrial waste products (saturation sludge from the sugar industry, the so-called "calcite sludge"). The designed products were artificially aged, and examined in terms of microstructural, chemical-mineralogical and physical-mechanical characteristics. The obtained results indicate the fact that the designed elements of earthen architecture could be used as building materials, which opens up the possibility of reducing carbon dioxide emissions and the consumption of natural mineral resources in the production of building materials.

OA-82

# CALCIUM PHOSPHATE CERAMICS WITH MAX PHASE ADDITIVES FOR MEDICAL APPLICATIONS

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Calcium phosphate ceramics is widely used in medical practice as implants. Hydroxyapatite (HA) is the mineral component of hard tissues of mammals. The implants on the basis of HA are bioactive and have high biointegration with bones. They

usually produced in the form of granules. In spite of high hardness and compressive strength, HA ceramic is very brittle. Its fracture toughness is about 1 MPa $\cdot$ m<sup>1/2</sup> which is in several times low that the corresponding values for hard tissues [1]. The reinforcement of HA ceramics is performed by incorporation of reinforced particles such as ZrO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and HA whiskers [2] into ceramic matrix.

We have developed new kind of HA bioactive ceramics with additives of MAX phases by combination of traditional sintering and the sintering in direct current impulse technology [3]. The reinforcement of HA ceramics was made by  $Ti_3AlC_2$  in interval of 0–10 wt.%. HA was synthesized by the precipitation from the aqueous solutions [4]. The HA pressing with different content of  $Ti_3AlC_2$  particles were obtained by pressing of the samples at 100 MPa in stainless die. The pressing were sintered from room temperature to 1200 °C in the muffle furnace for 1 hour. The second part of the samples was sintered in DC impulse up to the temperature 1300 °C for 20 min. The obtained HA ceramics were analyzed by XRD, SEM, hardness, compressive strength, fracture toughness.

The results have shown that the HA ceramics obtained both technology has one phase HA. All samples have high microhardness and compressive strength. However the samples produced by traditional sintering in the furnace have lot of cracks and their fracture toughness was low than 1 MPa·m<sup>1/2</sup>. In the same time, the HA samples sintered in DC impulse technology have uniform microstructure. Their fracture toughness raised the values 2.4 MPa·m<sup>1/2</sup> what in two and half times high than the values for traditional samples. Moreover, this technology of HA sintering gives possibility to save time and power for production of new advanced biomaterials. SA – ASA

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#### OA-83

## ALUMINUM OXIDE LAYERS DEPOSITED USING PULSED DIRECT CURRENT ELECTROPHORETIC DEPOSITION

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Electrophoretic deposition is one of the innovative methods for coating deposition on conductive uneven substrates/elements. Despite many advantages of the technique, deposition from aqueous media is still challenging. During the process, water electrolysis may occur, which leads to gas formation and desorption from the substrate surface while

leaving cavities in the coating. Pulsed Direct Current Electrophoretic Deposition (PDC EPD) is successfully used in coating deposition from aqueous media to limit cavities formation. The research aims to establish the best combination of parameters to obtain cavity-free aluminum oxide coating on a steel substrate. There is a complex microstructural and topographical analysis presented. Spectroscopic studies confirmed obtaining  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> coating after thermal treatment. Aluminum oxide coatings obtained using PCD EPD are compared among each other and with the reference samples. Cavities formation phenomena is explained based on textural studies. Mechanical studies combined with contact angle tests enable to choose of the best parameters for  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> coatings deposition using PDC EPD.

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OA-84

# SOL-GEL SYNTHESIS OF METAL-ION MODIFIED PRECERAMIC POLYMERS FOR DLP 3D PRINTING

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Silicon oxycarbide materials, so-called black-glasses, are characterised by an amorphous silica structure in which carbon ions are introduced by substitution of some of oxygen atoms. This results in a local increase in bond density thereby strengthening the structure of the glass network. By the phase separation, carbon can form a free phase, which gives characteristic colour of these materials. Its total content and the ratio between the amounts introduced into the structure and as a separate phase, determines the properties of black glasses. The amount of carbon can be controlled by using specific organosilicon compounds as substrates in the synthesis.

In this study, sol-gel synthesis of ladder-like polysilsesquioxanes was performed using 3-(trimethoxysilyl)propyl methacrylate (TMSPMA) and dimethyldiethoxysilane (DMDES) as substrates. The precursors have been appropriately selected to enable the processing of the material by Digital Light Processing (DLP) 3D printing. Modifications with specific ions (e.g. copper(II) cations) were introduced by adding an appropriate amount of hydrated nitrate(V) of a given metal into the reaction mixture in order to provide desired properties of the final product (e.g. catalytic). The obtained materials were subjected to detailed microstructural (SEM analysis) and structural (FT-IR and Raman spectroscopy) characterisation.

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OA-85

## INVESTIGATING THE REACTIVITY OF SURFACE FUNCTIONALIZATION WITH IONTERMINATED USING SCANNING ELECTROCHEMICAL MICROSCOPY (SECM)

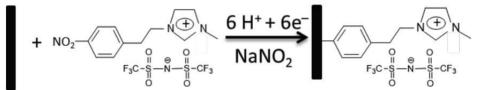
Hocine Moussouni<sup>1</sup>, Mourad Mechouet<sup>1</sup>, Jalal Ghilane<sup>2</sup>, Selma Bencherif<sup>1</sup>

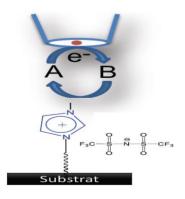
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The SECM technique is used for the first time for study the local electrochemical reactivity of electrografted ionic liquid without and with metallic nanoparticules (Pt or Pd). It is carried out by the microelectrode approach on the modified surface in a solution





containing redox couples. The obtained results highlight the change in the SECM response depending on the used redox mediator. In deed a positive feedback is observed for negatively charged mediator suggesting the anion exchange within the layer and the presence of 2D mediation within the layer, while a negative feedback is observed with an outersphere charge transfer mediator In addition, the ability to make anion exchange has been exploited to form nanoparticles (with Pt or Pd) inside the organic layer matrix. It seems that, both modified electrode the SECM responses

are reversed to those observed without nanoparticles, indicating charge transfer mechanism change.

# OA-86 SYNTHESIS AND STRUCTURAL ANALYSIS OF SOL-GEL DERIVED SiFeOC LAYERS

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Sol-gel process allows the formation of solid materials from solutions of precursors. A network of solid material is formed by hydrolysis of precursors and subsequent condensation of the reaction products to form macromolecules and networks. The properties of materials obtained by sol-gel process depend on the type and concentration of precursors, type and concentration of catalysts, amount of added water, order in which the reactants are added, time of mixing, temperature, and humidity.

One group of the very interesting materials, produced frequently by means of solgel method are Polymer Derived Ceramics. Their intriguing properties are strictly dependent on the very each level of the material's formation process. One of them include so-called cationic substitution of basic Si<sup>4+</sup> cations present in the glassy network with different ones i.e.  $Al^{3+}$ ,  $Ni^{2+}$ .

The aim of this study was to obtain SiOC layers glasses doped with Fe<sup>3+</sup> cations and analyse the obtained materials structurally. Fe<sup>3+</sup> cations were introduced by dissolving iron acetylacetonate [Fe(C<sub>5</sub>H<sub>7</sub>O<sub>2</sub>)<sub>3</sub>] in the synthesised sol. After synthesis and deposition by dip coating method, the material was subjected to a two-step heat treatment process: drying at 70 °C for 7 days and annealing in argon atmosphere at 800 °C.

For materials after both heat-treatment processes, structural analysis was carried out, which included the following methods: X-ray diffraction (XRD), Middle Infrared spectroscopy (MIR), Raman spectroscopy, and SEM microscopy with EDS.

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# PHOTOCURABLE CERAMIC DISPERSIONS USED IN THE PREPARATION OF COMPOSITE MATERILAS

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The aim of this research was to develop composite materials that can operate at high frequencies based on ferroelectric ceramic powder dispersed in a polymeric matrix. Composites were obtained by the novel method combining photopolymerization of monomers with traditional tape casting. The compositions of ceramic slurries containing different types and amounts of photocurable monomers, photoinitiators and dispersing agents were optimized as well as the conditions of photopolymerization process. The influence of the compositions of the suspensions on their rheological properties was examined. The effect of the time of the photopolymerization process and the type and amounts of selected monomers on the cure-depth of the polymerization was investigated. After the formation of composite tape on a special substrate, ceramic-polymer composites were obtained by photopolymerization. The physicochemical and electrical properties of the obtained composite materials were examined, including the BDS analysis which allowed to measure the electric permittivity of ceramic-polymer composites as a function of temperature and frequency of the applied electric field.

Ferroelectric ceramic-polymer composites are characterized by low values of dielectric permittivity and dielectric losses, the possibility of changing the relative value of electric permittivity in the external electric field and flexibility. These composites can be used in tunable antenna systems, satellite telecommunications, materials imaging and chemical analysis.

Keywords: ceramic-polymer composites, photopolymerization, tape casting, colloidal processing

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# PRECERAMIC POLYMERS FOR THE PREPARATION OF 3D SILICON OXYCARBIDE STRUCTURES BY DIGITAL LIGHT PROCESSING

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Preceramic polymers used to prepare Polymer-derived Ceramics (PDCs) are new materials that have been widely developed in recent years. Their main advantage is that they can be processed using methods typical of polymers. Through their application, samples with complex geometry which are subsequently converted to ceramics may be obtained. This provides more opportunities to prepare materials with a specific microstructure compared to direct processing of ceramics. Therefore, this concept is very attractive in the preparation of new materials.

Silicon oxycarbide (SiOC) is one of the most interesting silicon-based ceramic material due to its physicochemical properties, which originate from the characteristic structure. The typical amorphous structure of silica is modified by the introduction of carbon as a substituent for some oxygen as well as a separate phase. The total content of carbon and the ratio of its two forms determine the final properties of the product.

The research involved the development of a new procedure for the preparation of 3D SiOC structures using Digital Light Processing 3D printing. This included the following three steps. The first was sol-gel synthesis of photocurable polysilsesquioxanes with the introduction of methacrylate groups designed to obtain a specific amount of carbon in the final material. The second was optimization of the 3D printing process using Cellink Lumen X+, including the selection of the photoinitiator system to provide the efficient photopolymerization process, and the preparation of the 3D samples which fully reproduce the predesigned virtual models. The final step was the thermal processing of the printed samples to obtain the final 3D SiOC structures.

The products were characterized in terms of their microstructure (scanning electron microscopy) and structure (FT-IR and Raman spectroscopy).

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# ECO-FRIENDLY GLASS FERTILIZERS – CONTROLLED NUTRIENT RELEASE FOR WHEAT PLANTS

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This study delves into the sustainable application of materials for the efficient valorization of plant yield and quality, focusing on a novel ecological approach using phosphate glass-matrix as controlled-release fertilizers. The research investigates the physico-chemical properties, structural changes, and dissolution kinetics of four distinct phosphate glasses enhanced with different microelements. These modified glasses serve as eco-friendly fertilizers, designed to release essential macro and micronutrients in synchronization with the specific demands of wheat plants.

The characterization of these innovative glass formulations was executed through a comprehensive analysis involving differential thermal analysis, density measurements, X-ray diffraction, FTIR spectroscopy, and Raman spectroscopy. Dissolution behaviors were probed using both weight and pH measurements, with leachate solutions subjected to ICP-OES for ionic concentration assessment. The study unequivocally demonstrates the pivotal role of chemical composition and lattice structure of the glasses in governing nutrient release kinetics. The nuanced variations observed among the developed glass compositions are attributed to the interplay between the strength of bonds among latticeforming and modifying components, along with the ionic field strength of the incorporated elements.

Furthermore, an agronomic assessment was conducted to evaluate the efficacy of these glass-based fertilizers on wheat crops. The results unveiled a constructive influence on multiple aspects of wheat growth, including yield, photosynthetic parameters, and grain mineral content. Remarkably, the enhancements achieved ranged from 4 to 89% compared to conventional fertilizers, signaling the promising potential of these eco-friendly alternatives. These compelling outcomes lay the groundwork for large-scale experimentation, affirming the viability of these phosphate glass-matrix fertilizers for sustainable and eco-conscious crop production.

Keywords: phosphate, glass, chemical durability, wheat, growth; yield, Triticum durum

# HIGH-TEMPERATURE ALKALINE CORROSION BEHAVIOUR OF [CaO, SrO, BaO]–Na<sub>2</sub>O–B<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> ENAMEL COATINGS ON METALLIC SUBSTRATES

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Enamel coatings are widely used in appliances, cookware, chemical, and water storage tank applications due to their chemical resistance, wear resistance, and thermal shock resistance properties. This study investigates the effect of the earth's alkaline oxides, such as calcium oxide (CaO), strontium oxide (SrO), and barium oxide (BaO), on the alkaline resistance of enamel coatings. The enamel formulas were modified by incorporating different types of earth alkaline oxides, and the resulting coatings were characterized for their chemical composition, microstructure, and alkaline resistance performance. The presence of equal molar of CaO, SrO, and BaO in each enamel formula leads to the formation of stable oxide phases such as calcium-silicate, and barium-silicate hydrates that act as barriers against alkaline attack. Furthermore, the earth-alkaline oxides contribute to the formation of a dense and protective surface layer, which further enhances the resistance of enamel coatings to alkaline environments. The alkaline resistance of the enamel coatings is evaluated through various tests, including XRF for determinate oxide distribution of frit formulas, XRD for the determinate crystalline formation of coatings, immersion in NaOH alkaline solutions according to ISO 28706-4 standard, pH measurements, and surface characterization techniques such as SEM and surface topography. The results of the test to determine the alkaline resistance show that SrO in the formulas provides the highest alkali resistance to coatings compared to CaO and BaO. Also, when comparing the gloss change of coatings, formulas consisting of BaO and SrO have gloss values of approximately 97%, whereas formulas consisting of CaO present a 70% gloss change.

Keywords: enamel coatings, alkaline resistance, earth alkaline oxides, CaO, SrO, BaO

# MULTI-ANALYTICAL NON-INVASIVE METHODS AS A TOOL FOR PIGMENT CHARACTERIZATION

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Historical paintings typically have very complex chemical compositions that come from a variety of sources, including pigments, binders, ground layers, varnishes, fillers, extenders, and more. Based on the mentioned fact, it would be very useful for scientists and conservators to have a reference database of pigments already mixed with other relevant compounds. The goal of this study was investigation of the possibility of utilizing non - invasive techniques in the identification of pigment, binder and pigmentbinder interactions on egg tempera paintings. In total, over than 35 samples of pigments with egg as binder were examined and cross-compared. All samples were characterized by using analytical techniques: Vis spectrophotometry - colorimetry, X-ray fluorescence spectrometry (XRF), Infrared spectroscopy with Fourier transformation (FTIR) and Raman spectroscopy. Further, the formed database was used as a guideline for the examination of the real samples -icons from the iconostasis of the Church of St. Archangel Gabriel in Molovin - the icon of St. John the Theologian and the icon of the Blessed Virgin Mary. By using formed database it was possible to identified pigments and binders in painted layers and technology of icons painting, as well as to reveal previous interventions and non-original paintings layers. This research also provides a resource for further development of multi-analytical non-invasive methods, by comparing the weaknesses and strengths of the particular characterization techniques in regard to the identification of an individual pigment.

Keywords: multi-analytical, non-invasive methods, pigment identification

# THE INFLUENCE OF COAL FLY ASH ADDITIVES ON EVOLUTION OF CLAY-CEMENT MORTARS

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This study investigates the influence of fly ash from coal combustion on the evolution of clay-cement mortars. Coal fly ash, a byproduct of coal combustion, is a common substitute in construction materials due to its pozzolanic properties. Experimental testing was conducted on clay-cement mortars with different fly ash fractions and percentages of fly ash content. The evolution of the mortars was analyzed in terms of compressive strength, porosity, hydration kinetics, and microstructure. Results showed that the addition of fly ash influenced the early-age and long-term properties of the mortars. The findings reveal that the incorporation of fly ash tree led to improved compressive strange and decrease porosity of the mortars. The pozzolanic reactions between the fly ash and cement resulted in the shaping of additional building material compounds, enhancing the strength and durability of the mortars. Furthermore, the microstructural analysis demonstrated changes in the morphology and distribution of hydration products.

Overall, this contemplate highlights the positive effect of coal fly ash additive in clay-cement mortars. The use of fly ash fractions can raise the physical properties and durability of the mortars. Different fractions can find qualification suitability in various construction applications. These findings contribute to a better understanding of the influence of fly ash fractions on clay-cement mortars which can help in optimizing their application parameters.

**Keywords:** fly ash, clay-cement mortars and suspensions, pozzolanic reactions, compressive strength, microstructure, durability

# NANO-COPPER OXIDE-INDUCED SURFACE IMPROVEMENTS IN (Na,Li)<sub>2</sub>O-ZnO-P<sub>2</sub>O<sub>5</sub>-B<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> GLASS-CERAMIC COATINGS

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This study investigates the impact of nano-copper oxide addition on the microstructure and surface characteristics of glass-ceramic coatings. The coatings were synthesized using a conventional melt-quench method and subsequently subjected to heat treatment at 750, 800 and 850 °C. Comprehensive characterization techniques, including X-ray fluorescence (XRF), X-ray diffraction (XRD), differential thermal analysis (DTA), optical microscopy, 3D surface topography imaging, and surface roughness analysis, were employed. XRD patterns revealed no significant alteration in the crystalline phases of quartz and lithium phosphate, indicating that the addition of nano-copper oxide did not affect the overall phase composition of the coatings. Optical microscopy observations showed controlled grain growth in the coatings with nanocopper oxide addition, resulting in smaller and more uniformly distributed grains. The presence of dark areas (voids or porosity) and bright areas (impurities or nonuniformities) was reduced, indicating improved microstructural homogeneity. 3D surface topography imaging demonstrated distinct variations in surface topography with different nano-copper oxide concentrations. LP 50 CuO (5 wt.% nano-CuO addition) exhibited the most uniform and featureless surface, while LP\_05\_CuO (0.5 wt.% nano-CuO addition) and LP 10 CuO (1.0 wt.% nano-CuO addition) displayed scattered irregularities and small protrusions. Surface roughness analysis, based on Ra (Arithmetic Average Roughness), Rz (Maximum Height of Roughness Profile), and Rq (Root Mean Square Roughness), revealed that nano-copper oxide addition effectively reduced surface roughness, leading to smoother coatings. LP\_50\_CuO (5 wt.% nano-CuO addition) consistently exhibited the lowest Ra and Rq values among the samples with nano-copper oxide, indicating the highest surface smoothness. The successful incorporation of nanocopper oxide, minimal alteration in crystalline phases, controlled grain growth, improved microstructural homogeneity, and reduced surface roughness contribute to the understanding of how nano-copper oxide affects these coatings.

#### OT-94

## THE INFLUENCE OF BRUSHITE-METAKAOLIN GEOPOLYMER MATERIALS ON PHYTOSTABILIZATION OF LEAD IONS BY *FESTUCA RUBRA*

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Inorganic materials such as lime, calcium phosphates, and numerous aluminosilicates (red mud, fly ash, synthetic zeolites, clay) are used to immobilize metal/metalloid ions and improve soil quality, contributing to easier revegetation of contaminated soil. Using aided phytoremediation as a technique of choice for soil purification on metal-rich soils reduces the spread of pollutants by wind, soil erosion, and surface runoff. Studies have shown that Festuca rubra can tolerate high concentrations of metals, by using excluding strategies. Geopolymer materials used in this experiment were synthesized from a natural raw kaolinite clay with the addition of 2 wt.%, 4 wt.% and 6 wt.% of brushite (GPB2, GPB4 and GPB6). The main goal of this study is to examine the influence of brushite-metakaolin geopolymer materials on the phytostabilization efficiency of Pb(II) by Festuca rubra. The plant samples were grown using commercial substrate and seeds in eight different series, under controlled conditions. The series involved soil contaminated by Pb(II) treated with selected samples GPB2, GPB4 and GPB6, and the one untreated series and control series. Plant samples were collected after six weeks, and the plant height, fresh biomass, and dry biomass were measured. Germination rates of the plant samples were monitored during the experiment. Heavy metal content in the plant and soil samples was analyzed using atomic absorption spectrophotometer (AAS), and BCF, BAF and TF were calculated. Besides that, pH and organic carbon content in the soil samples were measured. Due to the fact that the starting materials which were used for GPBs synthesis were abandoned kaolinite clay and brushite obtained using green chemistry processes, byproducts of the synthesis are not harmful for the environment. In addition, this procedure contributes positively to the waste reduction.

OC-95

# STUDY OF STRUCTURAL AND SURFACE CHANGES OF SIO<sub>2</sub> FLINT AGGREGATE UNDER THERMAL TREATMENT FOR POTENTIAL VALORIZATION

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In order to extend its use, the fabrication and investigation of controlled SiOH SiO<sub>2</sub> surfaces are performed. The study of the effect of the heat treatment on the structural and surface changes of a natural flint SiO<sub>2</sub> aggregate after an optimized chemical treatment was carried out. The obtained samples were submitted to thermal treatment at three different temperatures, 500, 700 and 1000 °C. Then, the samples were investigated using different techniques. X-ray diffraction (XRD) was used in order to follow the structure's evolution with heat treatment. The decrease of the FWHM of the SiO<sub>2</sub>-(101) peak shows that the crystalline quality is improved upon heating. This result was confirmed by Fourier transform infrared spectroscopy (FTIR). The morphology of SiO<sub>2</sub> samples were characterized using variable pressure scanning electron microscope (VP-SEM), revealing the presence of disordered needles nanometric sizes (~500 nm) on the surface of the grains, which can be removed by heating at higher temperatures. Furthermore, FTIR spectroscopy also confirmed that the heating induces a reduction of OH groups on the surface. Thermogravimetry (TG) was used as a reference method to determine the hydroxyl group content. The OH groups obtained on the surface of the samples without and with heat treatment at 500, 700 and 1000 °C are 0.83, 0.44, 0.28 and 0.2 mmol/g, respectively. This study allows us to obtain the controlled SiO2 surface and contributes new insights into the use of SiO<sub>2</sub> flint surfaces for new applications as functional filler in polymers/asphalts composites.

Keywords:  $SiO_2$  natural aggregate, valorization, microstructure, silanol SiOH, circular economy

## SIC PARTICLE SIZE EFFECT ON CERAMIC THERMO-MECHANICAL PROPERTIES

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In this study, we report on the impact of particle size on thermo-mechanical properties of ceramic structures. Varying sizes of SiC particles were synthesized using high energy ball milling, Kaolin, clay, Al<sub>2</sub>O<sub>3</sub>, talc, mullite, chamotte and SiC were used to synthesize ceramic rods and plates. Ceramic samples were moulded, dried between 30–50 °C for 48 hours and fired at 1350 °C for  $\approx 3\frac{1}{2}$  hours. The XRD analysis of milled samples illustrated that increasing milling speed increased rate of crystal size reduction, amorphization and lattice strain. The SiC containing ceramic samples showed characteristic 2 $\theta$  SiC peaks at  $\approx$ 35.8°, 41°, 60° and 70°. SEM characterization of samples revealed that Particles milled at 100 and 150 rpm were smooth with rounded edges while particles milled above 200 rpm were angular with rough morphology. The grain boundary of SiC ceramic samples was poorly integrated and as a result exhibited intra/ intergranular fracture mode, resulting in lower flexural strength. All ceramic samples fabricated using SiC particles size range  $488.95 \ge mps > 1 \mu m$  exhibited inferior flexural strength compared to conventional cordierite-mullite ceramic structures. However, ceramic samples containing 15% SiC milled at 200 rpm for 1 hour (mps≈1µm) exhibited significant reduction in apparent porosity (12.4%) and increment of bulky density (1.99  $g/cm^3$ ). The observed physical properties of ceramic samples were attributed to SiC particle size.

Keywords: particle size distribution, thermo-mechanical properties, XRD, SEM

## PHASE EQUILIBRIA IN THE La<sub>2</sub>O<sub>3</sub>-Lu<sub>2</sub>O<sub>3</sub>-Ho<sub>2</sub>O<sub>3</sub> SYSTEM AT 1500 AND 1600 °C

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Lanthana ( $La_2O_3$ ) based solid solutions are transparent enough in the wide optical diapason to be used in optoelectronic devices. The complex oxide compounds LnLn'O<sub>3</sub> (Ln, Ln' = rare earth) with perovskite-type structure possess various electric (high dielectric constant), magnetic and optical properties (non-isotropic optics).

Powders of La<sub>2</sub>O<sub>3</sub>, Lu<sub>2</sub>O<sub>3</sub>, and Ho<sub>2</sub>O<sub>3</sub> (99.99 %) were used as raw materials. The samples were prepared in steps of 1 and 5 mol% from nitrate solutions with their subsequent evaporation and decomposition at 800 °C for 2 h. Powders were pressed at 10 MPa into pellets of 5 mm in diameter and 4 mm in height. Thermal treatments were carried out in the furnaces with heating elements based on H23U5T (1100 °C) and MoSi<sub>2</sub> (1500 and 1600 °C) in air. The phase compositions were investigated by X-ray diffraction and electron microprobe X-ray analyses.

The X-ray analysis of the samples was performed by powder method using DRON-3 at room temperature (CuK $\alpha$  radiation) with a step size of 0.05–0.1° in the range  $2\theta = 15-90^{\circ}$ . Lattice parameters were refined by the least-squares fitting using the LATTIC code. The accuracy in the lattice parameter of cubic phases was within 0.0002 nm. Microstructures were examined on polished sections and rough fractured surfaces of annealed samples in secondary electron (SE) and backscattered electron (BSE).

The isothermal section of the phase diagram for the La<sub>2</sub>O<sub>3</sub>-Lu<sub>2</sub>O<sub>3</sub>-Ho<sub>2</sub>O<sub>3</sub> system at 1500 and 1600°C has been developed. It was established that in the ternary  $La_2O_{3-}$  $Lu_2O_3$ -Ho<sub>2</sub>O<sub>3</sub> system there exist fields of solid solutions based on hexagonal (A) and monoclinic (B) modification of La<sub>2</sub>O<sub>3</sub>, cubic (C) modification of Lu<sub>2</sub>O<sub>3</sub>(Ho<sub>2</sub>O<sub>3</sub>), as well as perovskite-type ordered phase of orthorhombic symmetry  $LaLuO_3$  (R). The refined lattice parameters of the unit cells for solid solutions and microstructures of the definite field of compositions for the systems solid were determined. In the ternary system  $La_2O_3$ -Lu<sub>2</sub>O<sub>3</sub>-Ho<sub>2</sub>O<sub>3</sub> at 1500 and 1600 °C, the ordered phase of perovskite-type with rhombic distortion has been revealed. The maximum solubility of Ho<sub>2</sub>O<sub>3</sub> in the R phase is ~8 mol% along section Ho<sub>2</sub>O<sub>3</sub>-(50 mol% La<sub>2</sub>O<sub>3</sub>-50 mol% Lu<sub>2</sub>O<sub>3</sub>) (1500 °C). Using the concentration dependence of the lattice parameters it has been found that the homogeneity fields of solid solutions based on R phase spreads from 47 to 54 mol% La<sub>2</sub>O<sub>3</sub> along section La<sub>2</sub>O<sub>3</sub>-(50 mol% Lu<sub>2</sub>O<sub>3</sub>-50 mol% Ho<sub>2</sub>O<sub>3</sub>). Lutetium and holmium oxides form a continuous series of C-REE oxides solid solutions. The solubility of La<sub>2</sub>O<sub>3</sub> in the C-phase along the section  $La_2O_3$ -(50 mol%  $Lu_2O_3$ -50 mol%  $Ho_2O_3$ ) is about 4 mol% at 1500 °C and 5 mol% at 1600°C.

OC-98

# INFLUENCE OF HAFNIUM OXIDE ON THE STRUCTURE AND PROPERTIES OF POWDERS AND CERAMICS OF THE YSZ-HfO<sub>2</sub> COMPOSITION

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Nowadays ceramic materials are widely used in the industry, engineering and medicine [1, 2] due to the high level of their mechanical properties, in particular, wear resistance and hardness. In the series of oxide ceramics, especially important materials are those based on zirconium dioxide ( $ZrO_2$ ). The main part of zirconia based ceramics of industrial importance is binary solid solutions of  $ZrO_2-Y_2O_3$  or  $ZrO_2-MgO$  type. Doping with the third component is a promising way to obtain new properties of ceramics based on zirconium dioxide. The aim of this work is to study of the effect of hafnium oxide addition on the structure and properties of yttrium-stabilized zirconia nanopowders and to investigate hafnium oxide powders and ceramics based on them.

Study of the nY<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-mHfO<sub>2</sub> system in the range of hafnium concentrations from 1 to 15 wt.% and yttrium oxide concentration from 0 to 12 mol% showed that it is not full analogy of yttrium and hafnium oxides complete. It was shown that the density of three-component YSZ-HfO<sub>2</sub> - ceramics increases in the entire range of hafnium concentrations and an extremum corresponding to a 5 wt.% concentration of HfO<sub>2</sub> is observed in the concentration dependences of the strength and Young's modulus.

It was found that fractures of YSZ -  $HfO_2$  - ceramics at  $HfO_2$  concentrations in the range of 1–15% have an intercrystallite character. The homogeneous distribution of yttrium and hafnium impurities over the entire fracture surface was established and the absence of hafnium and yttrium along the grain boundaries was shown which prevents material softening.

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## DFT STUDY OF BULK AND EPITAXIAL LaMnO<sub>3</sub> FILM

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Research on lanthanum manganite-based materials, known for their complex electronic and magnetic properties, holds significant importance in the development of high-technology applications such as sensors, memory devices, and energy storage devices. Thin film materials provide an additional advantage by allowing control over their properties through the presence of strain, determined by the lattice parameters of the chosen substrate. In this study, we calculated the electronic and magnetic properties of bulk LaMnO<sub>3</sub> and LaMnO<sub>3</sub> under uniaxial strain, simulating the strain conditions that occur during the epitaxial growth of lanthanum manganite heterostructures. These investigations were conducted using computational methods based on density functional theory (DFT) with the Quantum Espresso 7.2 software package. During the process of geometric optimization and energy minimization, we established the ground state, which led to the determination of structural parameters that were subsequently employed in electronic structure calculations. The electronic property calculations included the band structure generation, determination of the bandgap energy, and the generation of the density of electronic states (DOS). For these calculations, we employed the SSSP PBEsol Efficiency v1.3.0 pseudopotentials, together with Hubbard potentials for correcting d-states of manganese. The results were evaluated by comparing them with literature values obtained through experimental studies.

# INVESTIGATION OF THE MICROSTRUCTURE AND MAGNETIC PROPERTIES OF SILICATE GLASS-CERAMICS WITH HIGH IRON OXIDE CONCENTRATION

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The synthesis of oxide glasses and glass-crystalline materials with high 3d transition metals concentration is of both theoretical and practical interest due to their possible application in electronics and medicine.

The present work reports the synthesis of samples from the compositional series  $16Na_2O/10CaO/(74-x)SiO_2/xFe_2O_3$ , x = 20, 25 and 30 mol% by the melt-quenching technique. Furthermore, thermal treatment at 580 °C for different times has been applied to the obtained materials in order to precipitate the Fe-containing phase magnetite. The phase composition is studied by X-ray diffraction analysis and the microstructure by scanning electron microscopy. The magnetization curves are recorded at room temperature by using vibrating sample magnetometer and magnetic characteristics such as coercive force and maximum magnetization are determined. Ferromagnetic nature of the measured samples is established. Subsequently, thermomagnetic curves are recorded in the temperature range 300–900 K and from them the Curie temperatures are determined.

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# INVESTIGATING THE EFFECTS OF Zr DOPING ON THE TITANIUM DIOXIDE NANOFIBRES

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In this work, titanium dioxide (TiO<sub>2</sub>) nanofibers doped with 0.5–5 mol% zirconium ions (Zr<sup>4+</sup>) were synthesized by combining the sol-gel process and electrospinning method, and calcined at 500 °C. The morphological, structural and optical properties of pure and Zr-doped TiO<sub>2</sub> nanofibers were investigated. According to the XRD and FTIR analyses, the addition of Zr as a dopant suppressed the transformation of anatase to rutile phase. Scanning electron microscopy showed that all fibers were smooth, fragile and randomly oriented after the calcination process. HRTEM analysis revealed that  $Zr^{4+}$  ions were incorporated at the substitutional sites in the anatase TiO<sub>2</sub> crystalline lattice. The photocatalytic efficiency for degradation of methylene blue (MB) was examined for both pure and Zr-doped TiO<sub>2</sub> samples. Nanofibers doped with 1% of Zr<sup>4+</sup> ions have shown the highest photocatalytic activity of 98%, wich can be explained by considering lower PL intensity in the PL spectrum of this sample, indicating suppressed electron-hole recombination.

#### OT-102

# COMPREHENSIVE STUDY ON CHARACTERIZATION, LEACHING BEHAVIOUR AND AGRICULTURE PREFORMANCE OF GLASS FRIT AS A SLOW-RELEASE FERTILIZER

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This research presents a comprehensive study on the characterization, leaching behavior, and agricultural performance of glass frit as a slow-release fertilizer. The glass fertilizer, synthesized from a K<sub>2</sub>O-CaO-P<sub>2</sub>O<sub>5</sub>-SiO<sub>2</sub> glass system was subjected to various analytical techniques. Extensive characterization techniques including were employed to assess the chemical composition, phase structure, molecular bonding, thermal behavior, and microstructure. Furthermore, plant breeding experiments were conducted to evaluate the effect of Glass fertilizers, NPK (Nitrogen-Phosphate-Potassium) fertilizer and combination of glass fertilizer with N(Nitrogen)-fertilizer on plant growth, with corresponding measurements of plant length. The plants treated with the glass fertilizer alone exhibited a plant length of 9.5 cm, while those treated with conventional NPK (Nitrogen-Phosphate-Potassium) fertilizer reached a length of 11 cm. The combination of glass fertilizer and N(Nitrogen)-fertilizer resulted in a plant length of 11.9 cm, indicating the potential synergistic effect of the two fertilizers. The leaching behavior of the glass fertilizer composition was investigated by conducting leaching experiments using water and a 2% citric acid solution as leaching solutions to simulate the soil and plant root environment. The results showed that nutrient release occurred over time, with higher concentrations observed in the citric acid solution compared to water. The concentrations of phosphorus, potassium, silicon, and boron in the glass fertilizer reached after 72 hours of leaching as 926, 2283, 58 and 303 mg/l, respectively, in water, while the concentrations increased to 9024, 25324, 898 and 1823 mg/l in the citric acid solution. These findings indicate the potential of the glass fertilizer to release nutrients and contribute to plant growth in agricultural applications. In conclusion, this study provides valuable insights into the characterization, leaching behavior, and agricultural performance of glass frit as a slow-release fertilizer. The plant breeding experiments demonstrate the importance of combining glass fertilizer with N-fertilizers to enhance plant growth.

# NOVEL POROUS ORGANOSILICA NANOPARTICLES FOR UV PROTECTION

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Porous organosilica nanoparticles, containing covalently embedded UV active organic molecules, provide an innovative method of preparing particles that can absorb UV light for skin protection [1,2]. Herein, research efforts on synthesizing novel porous organosilica nanoparticles (PMO), where the role of an organic component is to provide potent absorption of UV irradiation, while inorganic silica framework ensures the stability of the absorbing particles. This material is synthesized in basic aqueous environment, at room temperature, using cetyltrimethylammonium bromide as a template for the mesopores and organosilica precursors such as 4.4'bis(triethoxysilvl)biphenyl and a UV-blocking organosilica precursor prepared in situ from 4.4'.4"-s-Triazine-2.4.6-trivl-tribenzoic acid. Characterization of the prepared particles was performed using Fourier-transform infrared spectroscopy (FTIR) spectroscopy, Braunauer-Emmett-Teller adsorption method (BET), and two-photon excitation microscopy (TPEF). The characterization of the material showed the structure of mesoporous materials, CH vibrations at 2976 and 2922 cm<sup>-1</sup> and Si-C stretching peaks at 1132 cm<sup>-1</sup>, as well as vibrations at 1380 cm<sup>-1</sup> that represent aromatic CH groups from organic silil precusor. The BET analysis of the starting material displayed a type IV isotherm and a high surface area (282  $m^2/g$ ), while SEM analysis confirmed the particle size distribution from 800 to 1000 nm. UV/VIS measurements of the suspension of the particles revealed the capabilities of prepared materials for blocking UV irradiation. In addition, this material showed promising fluorescent properties upon two-photon excitation at 730 nm and laser power of 29 mW.

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OC-104

## EFFECTS OF PYROLYSIS CONDITIONS ON THE PRODUCTION OF ACTIVATED CARBON FROM OLIVE SEEDS

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In this study, the effect of gas type, rotation effect of the furnace (static, dynamic) and production method was investigated and activated carbon (AC) was produced from olive pit, an organic waste, in an oxygen-free environment (pyrolysis) with an ecosystem-friendly (environmentally friendly) method. The raw material was first kept in a nitrogen ( $N_2$ ) atmosphere at certain temperatures (600, 700 and 800 °C) and product characterization (SEM, BET, XRD, FESEM) was performed. Full activation of the product taken from  $N_2$  atmosphere was carried out in carbon dioxide (CO<sub>2</sub>) atmosphere at 700 and 800 °C. As the temperature and waiting time increased, pore development was observed in the product, therefore the specific surface area increased. It has been shown by SEM images and BET analysis that the CO<sub>2</sub> atmosphere creates a larger micropore in the raw material compared to the N2 atmosphere. Activated carbon was produced with the method called the traditional method (two-stage) in the literature, and the effect of two gases on the product properties was examined simultaneously. Activated carbon was obtained by the one-step physical activation method (CO<sub>2</sub>), which is an environmentally friendly production method alternative to the traditional method. The product was characterized and in the light of experimental studies, the highest specific surface area (1313  $m^2/g$ ) was obtained by the single-step production method. BET, SEM, XRD, FTIR analyzes of the products produced by both methods were performed and comparisons were made with studies reported in the literature. The findings have shown that products equivalent to commercial activated carbon can be produced under suitable conditions.

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