

1st ACDC 2023

BOOK of ABSTRACTS



1st International Conference

of the Croatian Ceramic Society (CroCerS):

Advanced Ceramics in Derivative Configurations (1st ACDC 2023)



IMPRESSUM

1st International CroCerS (Croatian Ceramic Society) Conference thematically named

1st **ACDC** (Advanced Ceramics in Derivative Configurations) 2023 Web: <u>https://www.fkit.unizg.hr/CroCerS/1stCroCerSConference2023</u>

Organizer

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Zagreb, Croatia, 2023

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FOREWORD

The **Croatian Ceramic Society** is the last established national society in the European area. As defined in the Statute, the **CroCerS** strives to increase visibility of the activities related with ceramic materials of the researchers and processionals in Croatia and internationally. Needless to say, establishing a biyearly International Conference would serve as a useful platform to support the above-mentioned activities. Following that, we believe the time is just about right to put the **International CroCerS Conference** under the spotlight. In doing so, the Scientific-Organizing Committee would like to omit what has become fairly standard segmentation of the topics based on the application area or chemical composition. Instead we would like to foster proactive segmentation based on correlation of material configurations and functionalities.

Therefore, the submissions are welcomed in following topics:

(i) CERAMIC BODIES ...

bulk, porous, aerogels, shaping, green, composites, properties for ... biosensors

(ii) CERAMIC THIN-FILMS ...

and other configurations, optoelectrical, piezo, for ... energy conversion devices

(iii) CERAMIC POWDERS ...

nanoparticles, core-shell, doping & processing for ... catalysts and batteries

(iv) CERAMICS COMPUTATIONS ...

with respect to theoretical & design aspects, modelling, for ... various utility

On behalf of the Scientific-Organizing Committee of the 1st International CroCerS (Croatian Ceramic Society) Conference thematically named 1st ACDC (Advanced Ceramics in Derivative Configurations) 2023, I am honoured and pleased to invite you to support the Conference which will be held from the 26th to 29th of April 2023 in Student centre of the University of Dubrovnik in Dubrovnik, Croatia.

Looking forward to welcoming you at the 1st ACDC 2023 in Dubrovnik!

Chair of the 1st ACDC 2023 Vicepresident of the CroCerS assoc. prof. dr. Vilko Mandić

TIMETABLE

AGENDA		Wednesday 26.4.2023.	Thursday 27.4.2023.	Friday 28.4.2023.	Saturday 29.4.2023.
08:00	08:00		Registration (6o min)	Registration (60 min)	
09:00	09:00 09:20 09:40		OL1 FRP (15+5min) OL2 IKI (15+5min) OL3 AB (15+5min)	OL6 LĆ (15+5min) OL7 AŽ (15+5min) OL8 MW (15+5min)	free
10:00	10:00		Coffee Break1 (30min)	Coffee Break3 (30min)	
11:00	10:30 10:55		IL1 SK (20+5min) IL2 MB (20+5min)	IL6 PŠ (20+5min) IL7 AR (20+5min)	Dubrovnik Walking Tour (120min) (20 Eur pp)
	11:20 11:45		IL3 IP (20+5min) WS1 PV.WALL (20+5min)	IL8 FP (20+5min) WS1 SL.SLOPE (20+5min)	meeting in centre
12:00	12:10		Lunch1 (85min)	Lunch2 (85min)	Lokrum island boat excursion (27 Eur pp)
13:00	12/25		WS2 ITRLAB (20+5min)	WS2 ITRS (20+5min)	+ possible lunch
14:00	13:35 14:00		PL4 BM (30+5min)	PL5 CAGG (30+5min)	BAD WEATHER ALTERNATIVE
	14:35		IL4 YL (20+5min)	IL9 MG (20+5min)	Srđ hill cable car excursion (27 Eur pp)
15:00	15:00		POSTERS+Coffee2 (30min)	PANEL+Coffee4 (30min)	+ possible lunch
16:00	15:30 15:50		OL4 JM (15+5min) IL5 LP (20+5min)	OL9 MR (15+5min) Awards + Closing (25min)	(200min) meeting in centre
17:00	16:15	Registration (120min)	free	free	
	18:15	Opening (5min) PL1 YKM (30+5min)			
19:00	18:55	PL2 NLW (30+5min)			
20:00	19:30	PL3 SM (30+5min)			
21:00	20:05	Welcome Reception (115min)	Conference dinner (150min)	Freestyle dinner (185min)	

SHORT BIO – PLENARY

Sanjay Mathur

Sanjay Mathur is a Chair Professor and Director of the Institute of Inorganic Chemistry at the University of Cologne in Germany. He is also the Director of the Institute of Renewable Energy Sources at the Xian Jiao Tong University, Xian, China and a World Class University Professor at the Chonbuk University in Korea. He is a Visiting Professor in the Institute of Global Innovation Research at TUAT, Japan and a SPARC Faculty at IIT Madras, India. His research interests focus on application of nanomaterials and advanced ceramics for energy technologies. He holds 11 patents and has authored/co-authored over 500 original research publications (> 18,000 citations, h index, 70) and has edited several books. He serves as the Editor for Journal of Electroceramics, and for NanoEnergy. He is an Academician of the World Academy of Ceramics and Fellow of the American Ceramic Society, Fellow of European Academy of Science, Fellow of Indian National Academy and Fellow of ASM International. He was awarded the Honorary Doctorate of the Vilnius University in 2016. He is an Academician of the World Academy of Ceramics and has received the Medal of the Chemical Research Society of India. He was honored by the Frontiers of Materials Science award of the IUMRS. He was awarded the Lifetime Achievement Award of Indian Science Congress Association in January 2020. He is the current President of the American Ceramic Society (21/22). He serves on the Executive Council of the European Materials Research Society and is the Secretary of International Union of Materials Research Society (IUMRS).

Carlos Alberto García González

MSc Chemical Engineering (University Santiago de Compostela, USC, Spain), MSc Chemistry (USC), Postgraduate in Innovation Management (Autonomous University Barcelona) and PhD Chemical Process Engineering (Technical University Catalonia, UPC, 2009). Postdoctoral researcher (2010-2012) at the Technical Univ. Hamburg (Germany) and contracted R&D Engineer (2012-2014) by the chemical company Solvay (Brussels, Belgium). In 2014 he joined the Department of Pharmacology, Pharmacy and Pharmaceutical Technology in the University of Santiago de Compostela (Spain) where he is currently a Full Professor.He participated in R+D projects in the field of Engineering of Materials for biomedical applications, mainly focused on the development, process optimization and valorization of nanostructured porous materials using green and emerging technologies (supercritical fluids, aerogels, 3D-printing). He has worked in the frame of more than 40 R+D projects funded by the European Union, national and regional governments and private companies. He is the coordinator (Chair) of AERoGELS COST Action aiming at the valorisation of aerogels for environmental and biomedical applications, and MC-member of GREENERING COST Action aiming at upscaling sustainable processes. As a result of the technology transfer and industrial application of his research, he has filed 2 patents, which are currently under results valorisation.

Barbara Malič

Barbara Malič is Scientific Councillor and Head of Electronic Ceramics Department, Jožef Stefan Institute, and Professor of Chemistry of Materials at Jožef Stefan International Postgraduate School, Ljubljana, Slovenia. She obtained her PhD (1995) in Chemistry at the University of Ljubljana, Slovenia. She is the author or co-author of more than 250 papers, 10 book chapters, one EU/US/Chinese patent in the field of electrocaloric cooling and 4 Slovenian patents in the field of electroceramics. Her research topics include lead-based and lead-free ferroelectric, piezoelectric and electrocaloric ceramics, multilayer structures, solution-derived functional-oxide thin films and inkjet printed nanostructures. She is a Fellow of the European Ceramic Society (2019), a Fellow of the American Ceramic Society (2022), and a member of the World Academy of Ceramics (2021). She is a recipient of the national science award - Zois award (2020) and IEEE 2022 Ferroelectrics Recognition Award.

Nae-Lih Wu

Dr. Nae-Lih Nick Wu is currently a Distinguished Professor at the Department of Chemical Engineering, at National Taiwan University. Dr. Wu's research interests include the synthesis and characterization of electrode and component materials for electrochemical devices, including supercapacitors and rechargeable batteries; development of advanced in-situ/in-operando analytic methodologies based on synchrotron facilities in characterizing these materials and devices, particularly for energy storage applications; and nanomaterials synthesis and applications. He is currently also serving as the program director of the Chemical Engineering Division, National Science and Technology Council in Taiwan, and an associate editor of Journal of the Electrochemical Society.

Yogendra Kumar-Mishra

Yogendra Kumar Mishra is Professor MSO at Mads Clausen Institute, NanoSYD, University of Southern Denmark (SDU), Denmark. Prior joining to SDU, he worked as group leader at Kiel University, Germany. He did Habilitation in Materials Science from Kiel University in 2015 and Ph. D. in Physics in 2008 from Jawaharlal Nehru University (Inter University Accelerator Centre), New Delhi, India. In Kiel, he introduced a new flame-based process for metal oxide tetrapod nanostructuring and their 3D networks, which showed many applications in engineering and biomedical fields. Additionally, tetrapods can be used as templates to create hybrid and new 3D materials. At NanoSYD, he is heading 'Smart Materials' group with the focus to develop new materials for green and sustainable technologies. He has published over 300 papers with over 15 500 citations and currently has an h-index of 67.

SHORT BIO – WORKSHOP

WORKSHOP: SLIPPERY SLOPE project

It is necessary to improve the efficiency indicators of functional materials. Lately, this vague statement has followed the research and development of advanced materials, composites and structured materials that strive for nanoscale order and functionality. This project aims at clarifying more complex but also fundamental aspects of material surface research. Particularly attractive and complex, and insufficiently researched, is the area of contact between porous or nanostructured materials with other materials prepared using wet chemistry methods, i.e. chemical deposition as well as physical deposition methods. Project PI is Vilko Mandić, and one of the project associates is Floren Radovanović-Perić.

Floren Radovanović-Perić

Floren Radovanović-Perić is a PhD student working at the Faculty of Chemical Engineering and Technology on the SLIPPERY SLOPE project, where he also got his master's degree in 2020. His research interests include the synthesis of metal oxides for semiconductors and batteries and the study of device physics of organic solar cells. So far, he has contributed to the project through 4 publications, two granted scholarships and multiple scientific missions.

WORKSHOP: "IRI2" project: Development of a technical solution for energy saving using VIS permeable or semi-permeable and IR reflective thin layers

This project explores the possibilities of preparing a multifunctional energy-efficient solar panel. The goal is to design a multifunctional system in which the plastic substrate would be coated with an advanced multilayer nanosized coating that would enable the achievement of desired properties. The goal is to test the production setup, which requires the interplay of processes that are technologically well known with the most advanced methods of control and application of materials at the nanoscale, which still require extensive scientific testing. Project PI at FCET is Vilko Mandić.

Vilko Mandić

After obtaining PhD in bulk ceramic materials in 2012, Vilko Mandić shifted research area towards functional nanomaterials in more demanding configurations within several international postdocs. From 2017 he is an assistant professor at Faculty of Chemical Engineering and Technology, University of Zagreb. Under the frame of several national and EU projects, as a Group leader he strives for developing new nanostructured systems for the application in the areas of energy conversion and sensing nanomaterials as well as environmental nanomaterials, using high-end chemical and physical deposition techniques, as well as describing them using thin-film dedicated structural and morphological tools. He won the national science award in 2022 for advancement in the diffraction of nanomaterials. He authored about 100 papers with about 1000 citations. He is vicepresident of the Croatian Ceramic Society, member of many other, editor and reviewer.

WORKSHOP: Application note: ITR-LAB

ITR-LAB d.o.o. is a Slovenian private limited company founded in 2008 and restructured in 2017. Since establishment we have had exclusive representative rights for both sales and service in Slovenia for Thermo Scientific's Scanning Electron Microscopes, Transmission Electron Microscopes and DualBeam systems, including their accessories. We are constantly active promoting through positive experience and references toward increasing the number of successfully installed instruments and maintenance contracts. Supporting our primary market of EM ITR-LAB d.o.o. is an authorised distributor in Slovenia for all products manufactured by Oxford Instruments and Quorum Technologies Ltd, also responsible for sales and after sales service, supported by original spare parts and consumables. To further pursue our customer's needs we have extended our product portfolio with distribution privileges of Life Sciences Cryogenic Equipment manufactured by the leading global supplier Worthington Industries.

Matija Dolinar

Matija Dolinar, electrotechnic engineer, 15 years of experience in service and application support in the electron microscopy field, specialized for SEM and SDB (small dual beam / FIB) systems and the related sample preparation.

WORKSHOP: PW-WALL project

The idea behind the project is to use geopolymer paste in a conductive and/or transparent configuration, similar to transparent conductive oxide materials, in order to investigate the possibility of applying such materials in solar cells. Also, the aim is to expand the existing knowledge on the interfacing of nanostructured transparent conductive layers and charge transfer layers. The goal is to enable a better understanding of the stability of the mentioned layers, for the preparation of different elements of solar cells, for example various heterojunctions, hybrid-organic multiphase heterojunctions, photoactive perovskites and similar. Project PI is Vilko Mandić, and co-PI is Neven Ukrainczyk.

Neven Ukrainczyk

Neven Ukrainczyk leads since 2015 a Research Team at TU Darmstadt (Germany) and lectures in RILEM Computational Methods course and Building Chemistry. Neven's motivation is to improve sustainability of construction materials, with 23 years of materials engineering experience he has worked at TU Delft (Marie Curie grant), TU Graz, Uni Zagreb and UFRJ Brazil. Specializing in building chemistry, he likes to combine measurement with computational multiscale approaches for designing and improving mineral binders and concrete, the most used building material in the modern world. Neven generated some recognition leading national and EU research projects, and international Technical Committees. He is an avid guitar and piano player.

SHORT BIO – INVITED

Stanislav Kurajica

Stanislav Kurajica was born in Dubrovnik, he graduated in 1991 at Faculty of Chemical Enginering and Technology and received PhD in 1998 at the University of Zagreb. He works at the Faculty of Chemical Engineering and Technology from 1991, he is a full professor from 2009 and received tenure in 2013. He is the author of two textbooks, Introduction to nanotechnology and Powder X-ray diffraction. The scientific activity of S. Kurajica is focused on the field of materials science and engineering, especially nanomaterials, technical ceramics and catalysts. He led four scientific and one technological project and collaborated on 12 projects. He published 132 scientific papers, of which 90 were in journals cited in tertiary publications. He was the dean and vice-dean of the Faculty of Chemical Engineering and Technology, a member of the Senate of the University of Zagreb, a member of the Council of the Technical faculties of the University of Zagreb, head of the Department of Inorganic Chemical Technology and Non-metals of the Faculty of Chemical Engineering and Technology etc. S. Kurajica is a member of Croatian academy of technical sciences, he was a founder and first president of Croatian Ceramic Society, he was awarded with the State Prize for Science in the field of technical sciences, the "Fran Bošnjaković" prize of the University of Zagreb and the "Franjo Hanaman" prize of the Faculty of Chemical Engineering and Technology.

Yannick Lorgouilloux

After getting an engineer's degree from the National Advanced School of Ceramics in Limoges (France) Yannick Lorgouilloux prepared a PhD thesis on the synthesis of silicogermanate zeolites at the Université de Haute-Alsace, in Mulhouse (France, 2007). After three years of post-doc in Leuven (Belgium), in 2011 he obtained an associate professor position in the CERAMATHS laboratory of Université Polytechnique Hauts-de-France, in Valenciennes (France). Since 2022, he is director of the Materials and Processes Department (DMP) of CERAMATHS. His work focusses on ceramics preparation, from powder synthesis to shaping and sintering. His research activities deal with piezoelectric ceramics, transparent ceramics and monoliths for catalytic applications.

Ivana Panžić

Ivana Panžić obtained her PhD diploma in 2021 at the Faculty of Chemical Engineering and Technology (FCET) in Zagreb while working at Ruđer Bošković Institute at Division of Materials Physics. Research focus is mainly on functional nanostructured, materials as constituents of photovoltaic devices (especially hybrid-organic and perovskite), capacitors, and sensors (gas and humidity) and sol-gel derived (also other advanced deposition) mixed-metal-oxide materials in the form of thin films and coatings with upgrades in their photocatalytic, optical, electrical properties. During the post doc at FCET and international stays research interests broadened in to the area of aerogels, bioinspired porous ceramics, mesoporous metal/metal oxide films, 3D nano-printed structures. Currently she is on her postdoctoral research stay the Materials Center Leoben Forschung GmbH (MCL), in the group of Dr. Marco Deluca. The main focus of work is barium titanate and related ferroelectric and relaxor perovskite materials for applications in energy storage.

Andrei Rotaru

Andrei Rotaru obtained the Doctorate of Engineering in Mechanical Engineering (2011) from the University Politehnica of Bucharest, the PhD in Chemistry (2013) from University of St Andrews and the Doctorate of Science in Physics (2016) from the University of Craiova. Between 2014 and 2015 he was a postdoc Researcher at University of Craiova with a fellowship of Visiting Researcher at the University of Cambridge (United Kingdom). In 2006 he joined INFLPR-The National Institute for Laser, Plasma and Radiation Physics as an Assistant Researcher and was promoted up to Senior Researcher II in 2014. Further, in 2017 he became Associate Professor of Chemistry at the University of Craiova, and since 2019 he is also a Senior Researcher II at the Institute of the Physical Chemistry "Ilie Murgulescu" of the Romanian Academy. In 2021 he defended the Habilitation at the University of Craiova. Currently he is the President of European Society for Thermal Analysis and Calorimetry, President of Central and Eastern European Committee for Thermal Analysis and Calorimetry and Chairman of the Scientific Commission of International Confederation of Thermal Analysis and Calorimetry. He is the Editor-in-Chief of Hybrid Advances & Coordinating Editor of Applied Surface Science Advances, serving also as Member of the Editorial Boards of the journals: Surfaces and Interfaces, Journal of Thermal Analysis and Calorimetry, & Molecules. His research interests include: Solid state chemistry, Condensed matter physics, Thermophysical properties of materials, Dynamics of nonlinear systems, Surface science, Processing thin films of soft materials by laser-assisted techniques.

Michal Borysiewicz

Dr Michał Borysiewicz has been working at the Łukasiewicz Research Center - Institute of Microelectronics and Photonics in Warsaw, Poland since 2007. His research interests include the development of nanoscale functional thin film materials and their integration with devices for electronics, optoelectronics, energy storage and sensors. The two main areas of work concern: dense thin film materials with various degrees of nanoscale complexity (epitaxial, polycrystalline, inclusion-in-matrix, amorphous, etc.) as well as porous crystalline thin film materials including inorganic porous metal and metal-oxides, MXenes and MOF films, the latter studied during a research stay at the Massachusetts Institute of Technology in 2018-2019. Over 60 articles, Over 800 times cited, h=14, leader of EU and nationally funded projects, Co-author of several national and international patents.

Filippo Parisi

Filippo Parisi received a Master of Science degree in Chemistry with a curriculum in Physical Chemistry (2008) from the University of Palermo (Italy). After a research stage (2011) at "GFZ – Helmholz Zentrum", in Potsdam (Germany) he obtained a PhD in Mineralogy (2012) from the University of Triest(Italy). Defended thesis: "A Bader's topological approach for the characterization of pressure-induced phase transitions". In 2013 he received a Post-Doctoral Fellow in Physical Chemistry at the University of Palermo (Italy). Since 2020 he obtained a Fellow from INSTM (National Interuniversity Consortium of Materials Science). Since 2020 again at the University of Trieste after his habilitation in Mineralogy. In the last years dealing with spinels, nanotubes, drug delivery and specially chess.

Luka Pavić

Luka Pavić was born in 1984 in Rijeka, Croatia. He obtained a Diploma in Chemistry (2008) and PhD (2014) from the Faculty of Science, University of Zagreb. In doctoral thesis, under the supervision of Prof. A. Moguš-Milanković, he focused on the investigation of induced crystallization and its influence on electrical transport of binary iron phosphate glasses. During PhD he spent six months in the group of Prof. M.A. Valente at the University of Aveiro, Portugal (2011/2012). After postdoctoral stay at Ecole Polytechnique, France (2015-2016), with Prof. A. Constantinescu where he worked on preparation along with electrical and mechanical characterisation of CNT-based strain sensors, he returned to Ruđer Bošković Institute, Zagreb, where he is working as Research Associate (2019) in Laboratory for Functional Materials. His research is devoted to the structure-property relationship studies and investigation of transport conduction mechanism as well as crystallization processes in various phosphate-based glasses and glass-ceramics with transition metal oxides as well as other materials.

Pavel Šiler

Pavel Šiler got his master and Ph.D. degree in chemistry of materials at Brno University of Technology, Czech Republic. He works at the Institute of Materials Chemistry, Faculty of Chemistry, Brno University of Technology. Part of his Ph.D. completed at the Magnel Laboratory for Concrete Research, Department of Structural Engineering, Ghent University, Belgium. After completing the Ph.D. studies, completed a postdoctoral internship at the Department of Chemical Engineering, National Cheng Kung University, Taiwan. He currently teaches the courses Chemical Engineering and Characterization methods of inorganic materials. In recent years, he has been mainly concerned with the influence of zinc on the properties and hydration of cement. His research interest include: concrete, high performance concrete, supplementary cementitious materials, superplasticizers, thermal analysis and calorimetry methods, mechanics test of materials and X-Ray Fluorescence technique.

Mattia Gaboardi

Mattia Gaboardi got PhD diploma in Physics at the University of Parma (Italy) on topic of synthesis and structural/spectroscopic study of metal-intercalated fullerides for hydrogenstorage. Thereafter he went on the post-doc at the University of Parma on the topic of study of carbon nanostructures for batteries & H2-storage. After that, he was a Marie-Curie Fellow at the Rutherford-Appleton Laboratory (Harwell, Oxfordshire, UK) dealing with in-situ structural and spectroscopic investigation of carbon nanomaterials using neutron scattering (NPD, INS, QENS). Thereafter he worked as an instrument Scientist at the MCX beamline at the Elettra synchrotron light-source (Trieste, Italy) dealing with high-resolution synchrotron powder-diffraction; in situ & operando studies on batteries and ceramics. From 2023 he is a scientist at the Materials Physics Center, CSIC-UPV/EHU (San Sebastian, Spain) and deals with study of sustainable materials with neutron & synchrotron techniques (QENS, INS, XRPD). ABSTRACTS: PLENARY

Chemical nanotechnologies: from molecules to materials for energy and health applications

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Abstract

Chemical processing of functional ceramics has played a key role in converging disciplines, which is especially true for their bridge-building role in integrating the concepts of inorganic materials synthesis with fabrication and integration of ceramic device components. Inorganic nanostructures offer new opportunities in materials engineering due to their improved intrinsic properties resulting from the reduction of microstructural features, which also allows for engineering of interfacial properties. This talk will present how chemically grown nanoparticles, nanowires and nanocomposites of different metal oxides can be transformed into integrated advanced material technologies. Examples will include application of superparamagnetic iron oxide nanoparticles for magnetic resonance imaging (MRI) and drug delivery applications, vapour phase synthesis and electrospinning of nanowires for application as electrode materials and in PEC water splitting reactions. New sensing concepts based on the integration and correlation of complementary functionalities originating from multiple junctions in a singular nanostructure to palliate the current issues in gas sensor technologies such as low power consumption, low operating temperature and cost-effective production will be elaborated. Finally, the current challenges of integration of nanomaterials in existing device concepts will be discussed.

This talk will present how chemically grown ceramic nanostructures and nanocomposites of different metal oxides open up new domains of material properties, which can be transformed into advanced ceramics and materials processing technologies.

Keywords

functional ceramics; nanostructures; health applications

Advanced processing of aerogels for biomedical uses: Outlook and prospects

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Abstract

Aerogels have been recently recognized among the Top-10 emerging technologies by IUPAC association. These advanced porous materials are characterized by a light weight and an extremely high and open mesoporosity. These unique physicochemical properties of aerogels result in outstanding specific surface areas, which can be exploited for several biomedical applications, including biosensors, drug delivery and regenerative medicine (Figure 1). Ceramic and hybrid aerogels, as well as composites comprising aerogels or admixtures of ceramic origin are commonly used for these purposes. In terms of production, aerogels can be easily shaped to the intended morphology, scaled-up and have even the possibility of being manufactured under good manufacturing practices (GMP). In this work, recent advances in aerogel processing as well as current and forthcoming uses of aerogels for biomedical applications are herein evaluated and discussed.

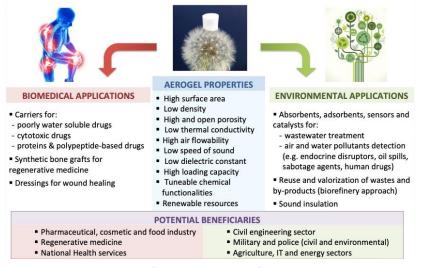


Figure 1. Main properties and applications of aerogels. For more information, please visit: https://cost-aerogels.eu

Keywords

aerogels; biosensors; porous materials; shaping

Acknowledgement

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Designing the microstructure of solution-derived lead-free ferroelectric perovskite-oxide thin films

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Abstract

Ferroelectrics – a group of dielectrics in which spontaneous polarisation can be switched by the applied electric field – are at the heart of numerous electronic devices. In the form of thin films (typically tens of nm to a 100 nm), they can serve as flexible and reconfigurable components that enable the miniaturisation of electronic devices. Besides physical vapour deposition methods such as sputtering or pulsed laser deposition, which rely on material transport in vacuum, Chemical Solution Deposition (CSD) of functional-oxide thin films is an alternative method in which the precursor is synthesised in liquid state, deposited on a substrate and usually heated to achieve crystallization of the target phase.

Since the early period of CSD, lead-based ferroelectric perovskite-oxides, exemplified by $Pb(Zr,Ti)O_3$ (PZT) have been extensively researched mainly for their outstanding piezoelectric properties, but the awareness of society about the toxicity of lead, supported by respective environmental legislation, has contributed to the research of lead-free alternatives. Material formulations extensively studied in the bulk ceramic form include sodium potassium niobate-, sodium bismuth titanate- and barium titanate-based solid solutions. In thin films not only the chemical and phase composition but also the presence of stresses, choice of the substrate and electrodes, and microstructure of the films, i.e., size and shape of the grains, strongly influence their functional properties.

In the contribution, we discuss microstructure evolution in selected lead-free ferroelectric perovskite thin films and the relation between the microstructure and their functional properties. Controlling the solution chemistry, deposition parameters, and heating regime enable the design of the microstructure of the studied films. Case studies include alkaline niobate- and barium titanate-based thin films.

Keywords

thin films; chemical solution deposition; microstructure; ferroelectric

Acknowledgement

This work was financially supported by Slovenian Research Agency (core funding P2-0105).

The interplay between microstructures and performance of Ni-rich Li(Ni,Co,Mn)O₂ cathodes for Li-ion batteries

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Abstract

Stimulated by the increasing awareness of the energy crisis and eco-friendly conservation around the world, research exploring lithium-ion batteries (LIBs) with great physio-electrochemical performance becomes an important issue. Ni-rich layer-structured Li(Ni,Co,Mn)O₂ (NCM) oxides have received noteworthy attention and are considered suitable cathodes for next-generation LIBs because of their lower cost, less toxicity, and high energy density. The state-of-the-art commercial NCMs are produced via a hydroxide-based precipitation process and are composed of secondary particles of 10~15 μ m in size. The adopted large particle size and interior microstructure have been designed to give high electrode packing density and hence high capacity and energy densities. The desire to increase the Ni content for achieving higher specific capacity unfortunately suffers from accelerated capacity fade due to increasing surface reactivity toward corrosion and volume variations, which bring about particle interior cracking and pulverization. To cope with these issues, there has been increasing research interest in NCMs with powder characteristics different from conventional ones. This research trend is also spurred up by the development of all-solid-state LIBs, which have rather different design considerations among the cell components from the liquid-electrolyte counterpart. This presentation will describe some key challenges faced by the NCM cathode materials for meeting the development of high-energy and fast-charging LIBs with enhanced safety and suggest, with examples, new perspectives in dealing with the challenges.

Keywords

Li-ion battery; nickel-rich cathode; powder microstructure

Acknowledgement

This work was financially supported by the National Science and Technology Council in Taiwan under contract numbers 110-2221-E-002-015-MY3 and 111-2923-E-011-001.

Section: (IV) Ceramic Computations

Tetrapods based smart materials for advanced technologies

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Abstract

Considering the size dependent utilization complexities of nanoscopic dimensions towards real applications, the focus of nanomaterials community is merging to three-dimensional (3D) form of materials which are built out interconnected nanostructures. This talk will briefly introduce the importance of complex shaped nanostructures towards smart 3D nanomaterials structuring. A simple flame based single step approach was developed for synthesizing zinc oxide tetrapods which demonstrated many applications in different technologies. These tetrapods have been used as building blocks to construct highly porous interconnected 3D nanonetworks in form of flexible ceramics which offer further new application avenues. Additionally, these 3D networks have been utilized as sacrificial templates to develop hollow tetrapodal 3D networks from almost any desired material, carbons, nitrides, oxides, polymers, hydrogels, etc. The sacrificial template-based strategy offers new and unique opportunities in the direction of 3D nanomaterials engineering and accordingly advanced technological applications. Some examples of 3D nanostructuring based smart materials in sensing, electronics, optoelectronics, energy, and biomedical engineering will be briefly highlighted in the talk.

Keywords

smart materials; tetrapods; hybrid nanomaterials; advanced technologies

ABSTRACTS: WORKSHOP

Pathways to porous ceramics: Shaping powders into structures

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Abstract

The field of ceramics as well as the flexibility of the material class has been expanding rapidly in the last decades. Application wise, porosity is one of the most important parameters for modern ceramic materials, whether the materials in question are solid state conductive ceramics for energy conversion and storage, self-standing photo- and electrocatalyst materials, thermoelectric materials or macro-microporous bioceramics which utilize both macro- and micropores to help repair or replace damaged bone tissue. In this workshop, we will try to present both conventional (sacrificial templating) as well as state-of-the-art (supercritical drying, lithography) techniques to obtain open porous bioceramic bodies and try to elucidate on the issues regarding porosity control, sample preparation and experimental setup. We will cover the basics of precursors synthesis and what type of porosity can be controlled from which aspect. We also comment on the compressive strength trade off as a consequence of achieving high porosity.

Keywords

porous bodies; bioceramics; supercritical drying; stereolitography; sacrificial templating

Acknowledgement

This work has been funded by the projects PZS-2019-02-1555 PV-WALL by CSF, ESF, UIP-2019-04-2367 SLIPPERY SLOPE by CSF and KK.01.2.1.02.0316 by ERDF.

Section: (II) Ceramic Thin Films

Spectroscopic characterisation of a solar collector

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Abstract

Utilisation of solar energy has become of utmost importance recently due to the global energy crisis. One of interesting and costly ways of minimising the expenditure of non-renewable energy sources is through the photothermal devices, or more commonly called solar collectors. They are devices engineered into various geometries with the purpose of heating and maintaining constant liquid temperature (~80 °C). Fabrication of such materials implies the choice of a transparent polymer with appropriate mechanical properties altered into advanced configurations through coating processes with inorganic materials which ensure absorption of irradiation in a controlled wavelength range, allowing for the liquid component to be heated to a constant temperature. In this workshop, we will cover the basics of polymer choice, coating material choice and methods of fabrication of these materials. Material characterization techniques which are crucial for the development of a good phototermal device, such as FTIR and UV-Vis reflectance and transmittance spectroscopy will also be discussed.

Keywords

absorption; photothermal; spectroscopy

Acknowledgement

This work has been funded by the projects PZS-2019-02-1555 PV-WALL by CSF, ESF, UIP-2019-04-2367 SLIPPERY SLOPE by CSF and KK.01.2.1.02.0316 by ERDF.

Section: (III) Ceramic Powders

Introduction of Clean connect and Inert gas sample transfer (IGST) workflow

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Abstract

With use of new technologies and worldwide production transformation, the understanding of the physical and chemical properties is becoming more and more challenging. That is also why standard analysis paths are being changed or adapted. One of these adaptions is a must have when working with atmosphere sensitive samples – like battery research. The growth of this market has led the equipment manufacturers to make changes in order to make analysis workflow more reliable, simplify sample handling and integrate to the various systems. The answer from ThermoFisher Scientific is Inert gas sample transfer workflow (IGST workflow) which connects glove box (inert environment) with sample preparation equipment as well as with SEM, TEM and other related equipment. Not only the battery research labs will gain from this solution, but it is also a great improvement for the rest of material science groups.

Keywords

electron microscopy; avoiding contamination

Section: (IV) Ceramics Computations

Computational approach for electrical conductivity of geopolymer-graphite composites

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Abstract

This invited workshop talk delves into the effective conductivity of geopolymer-graphite composites using mathematical modeling techniques. To simulate the percolation threshold, a particle-based Monte Carlo method is employed to generate virtual composite microstructures and simulate percolation phenomena. The effective medium model is then utilized to model the conductivity of the composite. The percolation and effective conductivity of a multi-phase mixture is closely related to the physicochemical characteristics of the mixed phases, such as the porosity and particle size distribution of individual components (metakaolin, graphite, and air) embedded in the geopolymer gel matrix. To investigate the effect of particle size distribution of the mixture phase on the percolation threshold, Hymostruc is used to generate a three-dimensional model of a mixture, which is then analyzed for connectivity of the conductive (graphite) filler using Matlab. The results indicate that the percolation threshold of a three-phase mixture of metakaolin, graphite, and pore is affected by the addition of segregated third phase, namely metakaolin or air (particle). To study the effect of metakaolin and porosity on the percolation threshold, the concept of effective volume fraction is introduced. This is defined as the ratio of the volume fraction of graphite to the total solid volume after excluding the segregated volume. Finally, a simple analytical effective medium model proposed by Bruggeman is used to simulate the variation pattern of conductivity of the mixture with increasing graphite content, in combination with the percolation threshold of the system simulated by Monte Carlo method. The simulation results show good agreement with the measurement results.

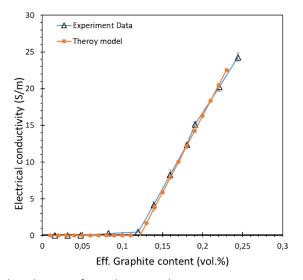


Figure 1. Electrical conductivity of geopolymer-graphite composites: comparison of modeling and measurement results.

Keywords

geopolymers; graphite; electrical conductivity; particle models; percolation; effective medium theory

Acknowledgement

This work was financially supported by the Research Initiative "Zukunft Bau" of the Federal Institute for Building, Urban and Regional Research (project: SWD-10.08.18.7-18.25) and the project "PV-WALL" PZS-2019-02-1555 in Research Cooperability Program of the Croatian Science Foundation funded by the European Union.

ABSTRACTS: INVITED Section: (I) Ceramic Bodies

Influence of composition on properties of bulk ZnO-Al₂O₃-SiO₂ glass ceramics

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Abstract

Samples with different ratios of ZnO, Al2O₃ and SiO₂, and a constant amount of TiO₂ were prepared by melting the batches at 1570 °C for 2 hours. The influence of glass composition on crystal phases, crystallite size, and appearance of prepared glass-ceramics has been investigated. As-prepared bulk glass samples were studied using differential thermal analysis (DTA). On the basis of glass transition temperature, the nucleation temperature was selected to be 740 °C. After 5 h of nucleation and heat treatment till 1150 °C, a part of the samples became white and opaque, while other samples turned out to be translucent and dichroic, i.e. bluish-grey coloured in reflected, and amber-coloured in transmitted light. Such behaviour is attributed to a combination of scattering and selective absorbance of light. On the basis of X-ray diffraction (XRD) analysis, it was established that gannite $(ZnAl_2O_4)$ is the main phase crystallizing in the course of thermal treatment, while corundum (Al_2O_3) , willemite (Zn_2SiO_4) , zinc titanate $(Zn_2Ti_3O_8)$, and rutile (TiO_2) appear in some of the samples as minor phases. A trend of crystallite size reduction with the increase of Al₂O₃ content has been noted. This was explained through a mechanism based on nucleation by nanoscale phase separation. High resolution transmission electron microscopy (HRTEM) analysis showed that the most translucent sample is densely populated with uniformly distributed and irregularly shaped gahnite crystal grains with average size of ~11 nm.

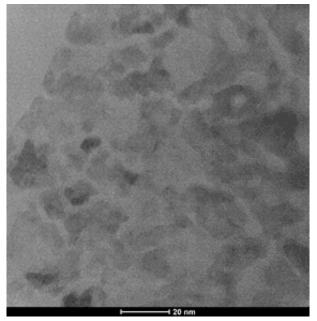


Figure 1. HRTEM micrograph of sample L nucleated at 740 °C for 5 h and crystallized by heating to 1150 °C.

Keywords

glass-ceramics; gahnite; nucleation; nanocrystals; dichroism

Acknowledgement

This work has been supported by University of Zagreb and GraFOx, a Leibniz science campus partially funded by the Leibnitz Association.

Optimization of (Ba,Ca)(Ti,Zr)O₃ lead-free piezoelectric ceramics properties by microwave sintering and by variation of the composition

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Abstract

Since a few years, the potential of piezoelectric ceramics for biomedical applications has become apparent and opened up a rapidly growing research field. Among others, efforts have been made to transfer established knowledge on classical piezoelectric applications to biosensors. PZT materials show piezoelectric properties much higher than other piezoelectric ceramics, but cannot be considered for such applications, due to lead toxicity. Among the lead-free alternatives, one is the family $(Ba_{1-x}Ca_x)(Ti_{1-y}Zr_y)O_3$ (BCTZ). These compounds have very promising piezoelectric properties, but a low Curie temperature. They have fewer applications than PZT but they can still be used at low temperatures as sensors.

In this work, focus was first put on the composition $(Ba_{0.85}Ca_{0.15})(Ti_{0.90}Zr_{0.10})O_3$ as reference. The effect of sintering conditions on piezoelectric properties was studied by comparing conventional sintering and microwave sintering.

In the second part of the work, the composition was slightly varied. The Zr/Ti ratio (y/(1-y)) on one hand and the Ca/Ba ratio (x/(1-x)) on the other hand were thus slightly modified. The aim was to finely modulate the piezoelectric properties. Each composition was characterized by X-ray diffraction and scanning electron microscopy. The piezoelectric, ferroelectric and dielectric properties were also studied.

This work made it possible to highlight a d₃₃ piezoelectric coefficient varying from ~290 pC/N to 400 pC/N for small modifications of the Zr content (from 0.07 to 0.09), as well as a Curie temperature reaching 90°C for some compositions (y = 0.07 – 0.08). Interesting electromechanical coupling factor kp values as high as 54% were measured when slightly decreasing Zr (y = 0.09) or increasing Ca content (x = 0.16), compared to the reference composition. The variation of Zr content also leads to improved ferroelectric properties when y decreases (Pr = 9.7 and Ps = 18.8 μ C/cm² for y = 0.08). Modification of the Ca/Ba ratio has lower effect on ferroelectric properties and almost no effect on the Curie temperature.

Keywords

BCTZ; powder X-ray diffraction; ferroelectricity; dielectric materials; piezoelectricity; microwave sintering

Acknowledgement

This work was financially supported by Région Hauts-de-France, UPHF and DGA, the French Defence Procurement Agency.

Explaining and exploiting relaxor properties in TTB electroceramic materials properties by thermal investigation and thermal processing

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Abstract

Tetragonal tungsten bronzes (TTBs), an important class of oxides known to exhibit ferroelectricity, undergo complex distortions, including rotations of oxygen octahedra, which give rise to either incommensurately or commensurately modulated superstructures. Many TTBs display broad, frequency-dependent relaxor dielectric behaviour rather than sharper frequency-independent normal ferroelectric anomalies, but the exact reasons that favour a particular type of temperature and frequency dependent dielectric response for a given composition remain unclear. Recently we have studied a family of ferroelectric (FE) and relaxor-ferroelectric (RFE) TTBs $Ba_4R_{0.67} \Box_{1.33}Nb_{10}O_{30}$ (R = La, Nd, Sm, Gd, Dy and Y and \Box = vacancy) with significant A-site vacancy concentrations using an array of diffraction techniques (high resolution neutron, synchrotron x-ray and selected area electron). The structural modulations are controlled by varying the A1-(perovskite) site cation size with different R, and allows manipulation of the nature of the dipolar ordering. For example, $Ba_4La_{0.67}\Box_{1.33}Nb_{10}O_{30}$ (R = La) is a commensurate RFE while R = Nd is incommensurate and FE. The crossover from these behaviours has also been examined in more detail *via* the solid solution $Ba_4La_{1-x}Nd_x\square_{1-33}Nb_{10}O_{30}La_{1-3}$ rich compounds display an additional feature of electrical field-driven ordering at temperatures between the RFE and paraelectric states, (demonstrated by 'pinched' polarisation-electric field (P-E) loops). The stability of this disordered regime has a clear thermal and electric field dependence and may be controlled by the average A-cation size (*i.e.* ratio of La:Nd).

We rationalise this behaviour in the context of our recent crystal-chemical model and demonstrate a test of the model *via* a simple compositional series. The key to understanding the variation in structure and electrical properties as a function of temperature is a multi-technique approach.

Keywords

relaxors; tetragonal tungsten bronze; dielectrics; electroceramics

Section: (II) Ceramic Thin-Films

Aspects and challenges of nanostructured metal oxide thin films

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Abstract

Zinc oxide (ZnO) and titanium oxide (TiO₂) are two widely studied metal oxide semiconductors due to their unique optical, electronic, and catalytic properties. These materials have attracted considerable interest in the development of advanced electronic device like sensors, solar cells, and photocatalysts, among other applications.

Nanostructured thin films of ZnO and TiO_2 , with thicknesses ranging from a few to several hundred nanometres, have received increasing attention in recent years due to their exceptional properties arising from the quantum confinement and large surface area-to-volume ratio. These properties include enhanced optical absorption, improved charge transport, and increased reactivity, making them suitable for the above-mentioned various applications.

In this work several chemical and physical methods of synthesis were utilized to produce nanostructured thin films, which were thoroughly characterized by SEM, STEM, TEM, AFM, GIXRD, UV-Vis, DLTS and ss-IS, and used in advanced configurations of thin-film based solar cells (perovskite) and as immobilized photocatalysts.

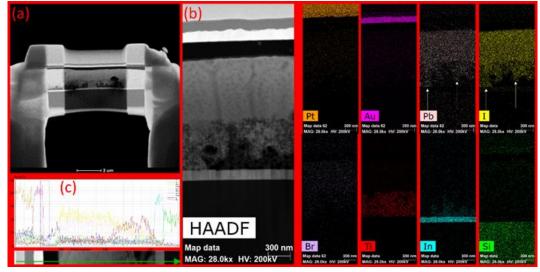


Figure 1. Micrographs of selected TiO₂ based solar cells; (a) STEM of the prepared lamella; (b) HAADF-STEM with mapping; (c) EDS line profiles.

Keywords

semiconductor metal oxides; thin-films; nanostructured; perovskite solar cells

Acknowledgement

This work has been funded by the projects PZS-2019-02-1555 PV-WALL by CSF, ESF, UIP-2019-04-2367 SLIPPERY SLOPE by CSF and KK.01.2.1.02.0316 by ERDF.

Controlling the band gap and transport properties of sputter-deposited AZO thin films by alloying with Mg - solutions for optoelectronic devices

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Abstract

The requirements of circular economy necessitate replacing critical raw materials with more sustainable alternatives. One such example is the need for replacing In-doped SnO₂, so called ITO, a transparent conducting oxide, widespread in touch screens. One of the candidates for that task is Aldoped ZnO (AZO) which consists of earth-abundant elements. ZnO is a material, the band gap of which can be broadened by alloying with Mg. However, there have been very few reports on ZnMgO:Al (or AZMO) wide bandgap transparent conducting electrodes.

In this report, we will present the results of the optimization of AZO and AZMO sputter deposition processes, with particular attention paid to the different sources that can be used for Mg introduction to AZO: metallic (Mg), ceramic (MgO) and compound ceramic (ZnO/Al₂O₃/MgO). We discuss how the differently deposited AZO and AZMO films behave as TCOs and show that it is possible to obtain materials with bandgap wider than the 3.2 eV of ZnO in a controlled manner (Fig. 1).

Finally, we test their performance in laser diodes using gallium nitride (GaN)-based structures and assess their applicability as contact structures to both sides of the diode (n and p).

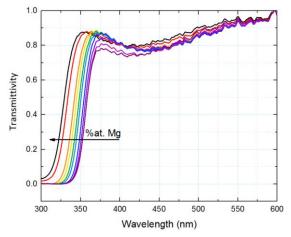


Figure 1. Optical transmission spectrum for AZMO samples with different Mg content, deposited using AZO and Mg and annealed after the deposition.

Keywords

AZO; transparent electrodes; laser diodes; thin films; sputtering

Acknowledgement

This work was financially supported by the National Centre for Research and Development, Poland, project 'OxyGaN' - M-ERA.NET2/2019/6/2020, by the Hungarian NRDI Fund, grant number 2019-2.1.7-ERA-NET-2020-00002 and by the Israel Ministry of Science, Technology and Space in the frames of the M-era.net Programme.

Section: (III) Ceramic Powders

New Insights into the oxidation process of normal spinels

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Abstract

The chromite end-member $Fe^{2+}Cr_2O_4$ is a typical example of a normal spinel where, with the term "normal", it is intended that the divalent cation fills only the tetrahedral site and the trivalent cations fill the octahedral one. This fact is due to the large excess octahedral crystal field stabilization energy of Cr³⁺ ensuring its preference for the octahedral site. Natural spinels can only approximate the ideal FeCr₂O₄, in fact other cations may enter into the crystal structure so that a (Mg, Fe²⁺)(Al, Cr)₂O₄ better describe natural Cr-bearing spinels. Another cation that could accommodate into the crystal structure in different amounts is Fe³⁺. Several Authors studied the effects of Fe³⁺ into the crystal lattice of Crbearing spinel and found out that it can be present both in T and M sites and that in large amounts it creates a magnetite/magnesioferrite component leading to an inverse spinel (Lenaz et al., 2011 among the others). The main cause of the presence of Fe³⁺ into the spinel structure is a higher oxygen fugacity of the original melt from which the spinels form, an oxidation process taking place after spinel formation or the unavailability of other trivalent cation in the melt. One crystal of synthetic chromite FeCr₂O₄ crystal has been annealed in air at 700°C for about 50 days at room pressure in order to verify changes in its physical-chemical condition. Once polished, its surface, analysed in backscattered image, showed areas with different compositions. Raman mapping revealed that the inner part is composed of pure chromite while the outer surface is composed of hematite. The rim area is composed of magnetite mixed in different ratios with hematite and/or chromite.

Keywords

chromite; magnetite; hematite; oxidation; Raman mapping

From glass to glass-ceramics: The pivotal role of (micro)structure in enhancing electrical and catalytic properties

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Abstract

Phosphate-based glasses and glass ceramics display remarkable properties that make them suitable materials for various technological applications. Recently, they have attracted special attention as solid electrolytes and/or electrode materials for the development of solid-state batteries. Despite numerous advantages of glassy materials, such as isotropic ionic conduction, lack of grain-boundary effect, and high compositional flexibility, their low electrical conductivity often limits their application. A cost-effective and scalable solution to this issue is the process of thermally induced crystallization, which leads to the formation of glass-ceramic, a composite material consisting of one or more crystalline phases embedded in a glassy matrix. In contrast to homogeneous structure of analogous glasses, glass-ceramics are heterogeneous systems whose macroscopic properties depend largely on microstructural features such as the type, size, distribution, and relative abundance of crystal grains within the amorphous matrix.

A powerful technique that facilitates the resolution of electrical processes in complex systems, such as multicomponent phosphate-based glasses and glass ceramics, is Solid-State Impedance Spectroscopy (SS-IS). This method is non-invasive, efficient, and easy to implement, and provides valuable insight into the electrical and dielectric properties of a system under study. The method involves applying a known voltage at different frequencies and measuring the resulting current. The impedance of the system is obtained by changing the excitation frequency across a broad range, from 10–2 Hz to 106 Hz, forming an impedance spectrum. The overall electrical response of the material-electrode system is due to various frequency-dependent microscopic processes, occurring either within the material itself (ion and/or electron transport and polarization) or at the interface between the electrode and the material (the transport or accumulation of charge carriers based on the type of electrode). Consequently, the materials that can be studied using SS-IS range from ionic and mixed ionic-electronic conductors and semiconductors to dielectrics and insulators.

In this talk the application of SS-IS will be illustrated through a series of case studies, with a particular focus on phosphate-based glass-(ceramics) systems with different conduction mechanisms: (i) mixed ion-polaron conductive glasses, (ii) mixed glass-network former glasses, (iii) mixed-conductive-mixed-glass-former-systems and (iv) glass-ceramics.

Mixed conductive systems that incorporate both mobile alkali ions and transition metal (TM) ions, such as V, Nb, Mo, W, and Fe, have been demonstrated to be highly effective as electrode materials in solid-state batteries. What's more, since these TMs are generally known to be highly active and selective catalysts in oxidation processes, prepared mixed conductive glasses-(ceramics) materials may be used as heterogeneous catalysts. This opens up new possibilities for the application of TM-containing glassy and glass-ceramic materials in a variety of fields.

Keywords

phosphate glass; phosphate glass-ceramic; electrical properties; catalytic properties

Acknowledgement

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Section: (III) Ceramic Powders

Effect of zinc on the properties of portland cement

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Abstract

In recent years, the amount of zinc in cement and subsequently in concrete has been constantly increasing, mainly due to the use of alternative fuels and the large use of secondary raw materials containing zinc. The presence of zinc in particular significantly prolongs hydration. The aim of this work is to show the rate of hydration delay using isothermal and isoperibolic calorimetry, the formation of new products using differential thermal analysis and the formation of new phases using the X-ray diffraction method. At the same time, morphology was studied using scanning electron microscopy with EDS microanalysis. Based on the obtained results, the possibilities of eliminating the negative effect of zinc with the help of appropriately chosen hydration accelerators were also monitored. It is precisely thanks to appropriately chosen accelerators that even in the presence of zinc, it is possible to obtain a material with a similar hydration time as that of ordinary Portland cement, but with better mechanical properties.

Keywords

Portland cement; zinc; hydration; hydration accelerators

Acknowledgement

This work was financially supported by the project GA-16646S "The elimination of the negative impact of zinc in Portland cement by accelerating concrete admixtures", with financial support from the Czech Science Foundation.

Section: (IV) Ceramic Computations

Long range order and local disorder in boosted-entropy ceramics

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Abstract

When a large number of similar atoms is compelled to share the same structural site in (nearly) equivalent fractions within a crystalline framework, the resulting boost of configurational entropy can lead to stabilizing new materials. This phenomenon was first predicted in 1981 but only observed for the first time in 2004 on metal alloys. Such materials are ordinarily referred to as high-entropy (HE).

Since 2015, this class of compounds has been successfully extended to ceramics with the first synthesis of high-entropy oxides, shortly followed by high-entropy carbides, nitrides, and diborides. Unlike highly-symmetric alloys, characterized by a unique crystalline site, HE ceramic structures display a minimum of 2 sublattices: typically, one or more for the metal cations and one site for the anion (O, C, N, or B). Among these freshly discovered classes of high-entropy ceramics, diborides stand out for their singular bi-dimensional arrangement, composed by alternating layers of boron and randomly distributed metals, forming a strongly anisotropic hexagonal lattice (similar to that found in intercalated graphite), yielding to unexpected properties and boosting the areas of application, such as wear-resistant coatings, ultra-high temperature resistance, or environmental barriers for the next-generation nuclear fusion-shielding materials. This work is meant to foster the understanding of high-entropy ceramics, which were therein investigated using high-resolution synchrotron powder diffraction and EXAFS analysis in order to disclose the real extent of homogenization and localize the structural disorder.

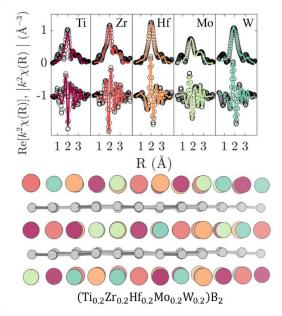


Figure 1. EXAFS analysis and modelized structure of a high-entropy diboride.

Keywords

ultra high temperature ceramics; high-entropy ceramics; local structure; X-ray scattering

Acknowledgement

We are grateful to Elettra synchrotron light source (Trieste, Italy) for providing beamtime and financial support (proposals nr. 20200101 and 20200077). This work was also financially supported the U.S. National Science Foundation through grant CMMI-1902069.

ABSTRACTS: ORAL LECTURES

3D volume morphology characterization of ceramic materials using advanced automated DualBeam technology

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Abstract

Ceramics are widely used in a variety of applications due to their unique combination of strength, hardness, and durability. However, these materials can also be brittle and prone to failure under certain conditions. Therefore, direct quantitative revelation of three-dimension morphology is of great importance in understanding the inner microstructure and properties of ceramic materials, providing the insights of overall performance and potential failure.

The DualBeam, a combination of a scanning electron microscope (SEM) and a focused ion beam (FIB), is an increasingly popular tool for advanced characterization and failure analysis of ceramic materials. The DualBeam has been used to study ceramic materials through multimodal imaging and visualization at the nanoscale. The SEM allows for the high-resolution imaging of surface features, while the FIB can be used to cut and mill away layers of material to reveal internal structures. This can be useful for identifying defects or variations in material composition that may contribute to the performance of the ceramic. Another use of the DualBeam in ceramic material research is through chemical analysis, especially utilizing analytical instruments such as EBSD/EDS.

In this abstract we present a few examples of ceramic materials characterization using the latest DualBeam technology. We share the experience of using automated serial sectioning capabilities combined with analytical detectors such as EBSD/EDS to obtain 3D structure and chemical distribution of porous ceramic materials. The data was acquired in an unattended manner and subsequently segmented and reconstructed using cutting edge data processing software. The pore structure, volume distribution, tortuosity were quantified and visualized respectively.

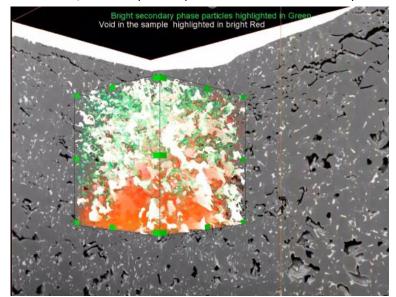


Figure 1. High resolution 3D reconstruction of a ceramic sample, 15 µm HFW. Data is segmented to analyse the distribution of different phases: Bright secondary phase (green) and Pores (white).

Keywords

3D volume; FIB; morphology; electron microscopy

Section: (I) Ceramic Bodies

Corrosion alumina ceramics corrosion in nitric acid

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Abstract

In this study, the corrosion behaviour of alumina ceramics was observed under different conditions: 0.5, 1.25 and 2 mol dm⁻³ of nitric acid at 25, 40 and 55 °C through 10 days. The results show that Al2O3 ceramics exhibit good corrosion resistance at higher HNO3 concentration. Al2O3 corrosion behaviour varies depending on the temperature, time and HNO3 concentration. In view of the reaction kinetics, it proceeds in the parabolic law in HNO3 aqueous solution and decreases with increase of the HNO3 concentration. The corrosion rate and activation energy were calculated and compared for all experimental conditions.

It was found that the corrosion rate of Al_2O_3 was found to be in the range 0.08 - 0.34 μ g² cm⁻⁴ h⁻¹ where the highest rate was achieved at lower HNO₃ concentration and highest temperature. Finally, the highest activation energy was determined for the 0.50 mol dm⁻³ HNO₃ concentration, which was more than twice as high as the activation energy observed at the 2.00 mol dm⁻³ HNO₃ concentration.

Keywords

alumina; corrosion activation energy; corrosion rate

Acknowledgement

This work was financed by the Croatian Science Foundation within the project Monolithic and composite advanced ceramics for wear and corrosion protection (WECOR) (IP-2016-06-6000). The authors would also like to thank Matt Sertić and Tomislav Jurašinović from Applied Ceramics, Inc. for green and hard machining alumina ceramics samples.

Mechanochemical approach in zero waste synthesis of geopolymer-zeolite composite

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Abstract

Kaolinite is a 1:1 clay mineral with the chemical formula $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$ that has a dioctahedral layer structure without charge. Each layer consists of one tetrahedral layer of silicon dioxide and one octahedral layer of aluminum oxide. The high proportion of SiO_2 and Al_2O_3 makes kaolinite an excellent source of aluminosilicates, which is why it is one of the most commonly used natural precursors in the synthesis of geopolymers and zeolites. However, highly ordered crystalline systems are more resistant to alkaline attack than amorphous systems. Therefore, kaolin as a crystalline precursor is often used in its amorphous form - metakaolin, which requires temperature treatment, usually in the range between 600 and 800 °C. In recent years, mechanochemistry has begun to be studied as an alternative to the high-temperature processing (calcining) of kaolin into metakaolin. Due to the reduction of solvent consumption, energy, and faster chemical reactions, mechanochemistry is considered a greener approach in the synthesis of aluminosilicate materials such as geopolymers and zeolites. In this work, the possibility of mechanochemical synthesis of geopolymer-zeolite composite was investigated. The composite synthesis parameters were approximately set to a molar ratio of 1 mol Na₂O: 1 mol Al₂O₃: 2.5 mol SiO₂, while the amount of water and the curing temperature of the samples varied. The obtained samples were characterised by XRD, SEM, and ATR-FTIR analysis.

Keywords

mechanochemistry; kaolin; zero waste; zeolite

Acknowledgement

This work has been funded by the projects PZS-2019-02-1555 PV-WALL by CSF and ESF, KK.01.2.1.02.0316 by ERDF, and UIP-2019-04-2367 SLIPPERY SLOPE by CSF.

Section: (II) Ceramic Thin-Films

Cellulose aerogels – SnO2 composites as potential photocatalysts

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Abstract

A huge focus worldwide is devoted to the production of novel tailored materials to improve electronic devices through sustainable processes. Nowadays, as reagents for the production of metal oxide– carbon composites that could be used in catalysis, sensing, and electrochemical devices, biopolymers are leading candidates. Various TiO2-based nanostructured materials fabricated using different cellulose templates such as nano and microcellulose fibres have been reported previously in the literature. Cellulose, as the most abundant renewable biopolymer, was used as well in our research to control the morphology, porosity, and therefore the properties of the derived composite samples at the nanoscale. Moreover, the tin(IV) oxide has a similar bandgap as already well-studied TiO2, while has not yet reached as wide applicability. In this work, to broaden the research in the field of SnO2 nanostructures, we investigated the properties of cellulose-immobilised SnO2 samples. Additionally, the influence of post-reaction treatment of the samples was investigated.

This work focuses on the course of non-hydrolytic sol-gel freeze-drying synthesis in combination with the reductive mineralization of cellulose. Samples' post-treatment was carried out under both inert and atmospheric conditions, at three chosen temperatures (60 °C, 700 °C, and 800 °C). Thin films of the resulting composite samples were prepared and characterised using grazing incidence X-ray diffraction (GIXRD), which overall showed the degree of chemical homogeneity of studied composites. Morphologic and surface geometries were studied by scanning electron microscopy (SEM). Finally, the photocatalytic activity was investigated by photocatalytic degradation with a model micropollutant with previously calculated bandgap values from diffuse reflectance spectroscopy (DRS) measurements.

Keywords

tin(IV) oxide; cellulose; photocatalysis; thin film

Acknowledgement

This work has been funded by the projects UIP-2019-04-2367 SLIPPERY SLOPE by CSF and PZS-2019-02-1555 PV-WALL by CSF and ESF, and KK.01.2.1.02.0316 by ERDF.

Section: (II) Ceramic Thin-Films

Spark plasma synthesis of defective titanium oxides: phenomena and peculiarities

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Abstract

Nowadays, the need for clean energy solutions is rapidly growing. Systems of high interest include versatile nanomaterials capable of energy conversions and storage, whether as photocatalysts, battery materials or transport layers in photovoltaic devices. Such materials need to satisfy multiple criteria, depending on their application, while the method of their synthesis needs to be reproducible, low-cost, green and scalable. Here we report on a solid state synthesis of titanium oxide nanoparticles through spark plasma discharge. We obtained primary particles of sizes as low as 10 nm with a mixed phase composition. Microscopy revealed multiple stages of assembly, stemming from coalescence and aggregation to agglomeration of web like structures. All of these result point out to a highly nanoporous material with an increased number of oxygen defects introduced into the structure which will hopefully serve as a superior electron transporting layer for flexible organic solar cells.

Keywords

spark plasma; clean energy; self-assembly

Acknowledgement

This work has been funded by the projects PZS-2019-02-1555 PV-WALL by CSF, ESF, UIP-2019-04-2367 SLIPPERY SLOPE by CSF and KK.01.2.1.02.0316 by ERDF.

Why do we publish? The reproducibility crisis in ceramic synthesis.

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Abstract

Once there was a time that scientists published papers in order to share their investigations and contribute to overall scientific knowledge. Current pressures to "publish (a lot) or perish" have distorted the incentives, and publishing has become a goal in and of itself. Instead of sharing the details of one's work, it is an established practice to hide the relevant details to avoid getting "scooped" by the competition. We have come to expect a dearth of information on synthesis details when reading the papers, which mostly focus on promising properties of a pure ceramic material under investigation, and less on nitty-gritty how to actually obtain the pure ceramic material in question. The number of published papers and journals has ballooned, but the usefulness of the publications has steadily decreased.

The aim of this presentation is to provide several examples of both very useful or very useless papers, to show that there is room to write interesting and useful papers focused solely on synthesis of ceramics, and hopefully to encourage the audience to share even the less "publishable" results with the world – someone, somewhere, will certainly be glad of it.

Keywords

chemical synthesis; infrared spectroscopy; precursor analysis; reproducibility

Acknowledgement

This work has been supported in part by Croatian Science Foundation under the project IP-2018-01-5246 and UIP-2020-02-9139.

Influence of the synthesis method on structural and magnetic properties of doped lanthanum manganites

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Abstract

Strontium-doped lanthanum manganites, described with the general formula La_{1-x}Sr_xMnO₃ (LSMO) are attractive, among others, for use in magnetic refrigeration and data storage due to their remarkable magnetic and electric properties at room temperature. In this study, LSMO powders were prepared by the citrate-nitrate autocombustion (CNA) and coprecipitation synthesis to investigate how the synthesis procedure impacts the structural characteristic of the materials. Both synthesis methods yielded materials with rhombohedral crystal structures. The lattice parameters and atoms occupancy factors were determined by Rietveld refinement analysis. The oxygen nonstoichiometry was determined by permanganate titration and the amount of Sr-dopant was confirmed by energydispersive X-ray spectroscopy. The obtained results showed that target Sr-doping was achieved and that materials prepared by coprecipitation possessed higher oxygen nonstoichiometry in comparison to the materials prepared by CNA. The magnetization curves as a function of temperature were recorded in a temperature range of 2-400 K at applied magnetic fields of 10 Oe, 100 Oe and 1000 Oe for all prepared materials. The Curie temperatures (T_c) increased with increasing Sr-doping meaning that materials with $x \ge 0.2$ are suitable for room-temperature applications as ferromagnets. Furthermore, Sr-doped materials showed low remanent magnetization making them applicable as soft ferromagnets. For materials prepared by coprecipitation, a large magnetic entropy change was observed near T_{C} . The maximum entropy changes of 1.5 and 1.3 J kg⁻¹ K⁻¹ were observed for LSMO materials with x = 0.1 and 0.2 at temperatures of 381 K and ~400 K, respectively. These two materials had the highest oxygen nonstoichiometry leading to the increase of Mn⁴⁺/Mn³⁺ mixed valences ratio and overall crystal lattice entropy, accompanied by magnetic phase transitions around room temperature. Therefore, coprecipitation synthesis is presented as an efficient synthesis method of manganites leading to the structural defects, i.e., oxygen nonstoichiometry resulting in improved overall magnetic properties.

Keywords

lanthanum manganites; magnetic properties; magnetocaloric effect; synthesis

Acknowledgement

This work has been supported in part by Croatian Science Foundation under the project IP-2018-01-5246 and the Croatian Academy of Sciences and Arts [grant Manganites for sustainable development and decreased air pollution]. D.B and D.P. acknowledge the support of projects CeNIKS co-financed by the Croatian Government and the European Union through the European Regional Development Fund–Competitiveness and Cohesion Operational Programme (Grant KK.01.1.1.02.0013.)

The preparation of manganese-doped ceria nanoparticles and their utilization for BTEX gas mixture oxidation

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Abstract

The commencement of ceria use for advanced catalytic applications took place a few decades ago and regardless of being widely used in catalysis, it is still being researched to improve its properties and catalytic efficiency. Besides in catalysis, owing to its fluorite structure and easy shift between Ce³⁺ and Ce⁴⁺ oxidation state, ceria utilization is investigated in solid oxide fuel cells and water purification membranes. Manganese, having multiple oxidation states, is assumed to improve many of CeO₂ properties including catalytic activity. As a catalyst, ceria is mostly utilized as a part of exhaust gas oxidation catalyst in three-way automobile catalytic converters and soot oxidation catalysts in diesel particulate filters, as well as for the oxidation of volatile organic compounds. In this investigation ceria (CeO₂) nanoparticles doped with manganese in various amounts were prepared via hydrothermal synthesis. The prepared samples were thermally treated at 500 °C for 2 hours to prevent change of properties in the course of catalytic test at elevated temperatures. Thermally treated samples were investigated via X-ray diffraction (XRD) and energy dispersive X-ray spectrometry (EDS) to determine the phase and elemental composition, while N2 adsorption-desorption isotherms were used for the determination of specific surface area. Finally, catalytic activity of the prepared catalyst was tested on benzene, toluene, ethylbenzene and o-xylene (BTEX) gas mix. It was established that doping of manganese into the ceria structure has noticeable benefits for the catalyst activity in BTEX oxidation process.

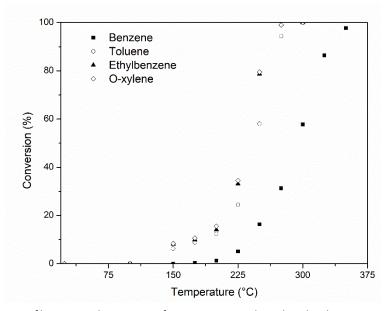


Figure 1. Conversion rates of benzene in the presence of ceria ceramic catalysts doped with various amounts of manganese.

Keywords

ceria; ceramics; nanoparticles; BTEX

Acknowledgement

This work was financially supported by the project Croatian Science Foundation under the project IP-01-2018-2963.

ABSTRACTS: POSTER COMMUNICATIONS

Development of simple rugged apparatus for solid and powder density determination

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Abstract

Measurement of density of solids and powders occurs frequently in the R&D and industrial quality control laboratories. The apparent and true density of the bulk material depends on the open and closed porosity and thus influences raw material and/or final product quality, mechanical and thermal properties. If the sample consists of the materials of different density, e.g. wood wool and cement binder, rapid density determination could indicate uniformity of dosing and mixing of raw components thus aiding in process control. The possibility of rapid and inexpensive density check during any stage of production of wood wool cement composite board is needed as a tool for early detection of process anomalies that could deteriorate product quality and increase production costs. It this work a simple and rugged apparatus for solid and powder density determination had been described. It consists of only one metal cell containing the sample and inexpensive pressure transducer. The various compressed technical gases or dry air is used as working gas and the pressure transient due to the gas flow through the capillary is recorded. The parameters of the fitted mathematical model indicate the volume of sample material in the cell, thus giving the density of solid.

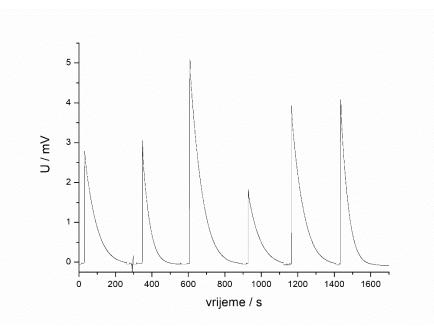


Figure 1. Pressure transients in the measuring cell containing powder sample due to the gas flow to the atmosphere through thin metal capillary.

Keywords

powder; density; gas pycnometer; capillary flow

Acknowledgement

This work was financially supported by the project "Acoustic incombustible plate" IRI2 KK.01.2.1.02.0299 FRAGMAT H d.o.o., HR-49223 Sveti Križ Začretje, Croatia.

Section: (II) Ceramic Thin-Films

Properties of sol-gel Ce-TiO₂ film

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Abstract

In this study, sol-gel process was used for the preparation of Ce-TiO₂ film with the following amount of cerium: 0.08 wt. %, 0.40 wt. %, 0.80%, 2.40 wt. %, 4.10 wt. % and 73.3 wt. %. For the preparation of the sol titanium isopropoxide and cerium (III) nitrate hexahydrate as a precursor, 2-propanol as a solvent, acetylacetone for complexation, distilled water for hydrolysis, and nitric acid as a catalyst were used. Ce-TiO₂ sol was deposited on a borosilicate glass substrate using a flow coating method, dried at 100 °C for 60 min, and heat-treated at 450 °C for 120 min (3 °C/min). Photocatalytic degradation of the pharmaceutical ciprofloxacin (CIP) dissolved in water was investigated by means of Ce-TiO₂ films irradiated with UV-A and solar-like radiation.

The film's surface morphology and roughness parameters were analysed by atomic force microscopy (AFM). Thermogravimetric analysis and differential scanning calorimetry (DSC/TG) were used to determine weight loss and endothermic and exothermic reactions during heat treatment. X-ray diffraction (XRD) was used to determine phase composition and parameters and volumes of unit cells.

Obtained results showed that $Ce-TiO_2$ film with 0.80 wt. % Ce improves photocatalytic degradation of CIP under UV-A and solar-like radiation compared to the pure TiO₂ film.

Keywords

Ce-TiO₂, ciprofloxacin, photocatalytic degradation, UV-A radiation, solar radiation

Acknowledgement

This research was funded by the Croatian Science Foundation under the project Fate of pharmaceuticals in the environment and during advanced wastewater treatment (PharmaFate) (IP-09-2014-2353).

Section: (III) Ceramic Powders

Paramelaconite Cu₄O₃ controversies and facts

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Abstract

Intermediate copper oxide Cu_4O_3 (paramelaconite) has been investigated in past decades in the mineral sample. The examinations of the structure by Frondel and O'Keeffe and Bovin (FO'KB) revealed the tetragonal unit cell containing 4 unit formulae Cu_4O_3 . However, repeated XRD records on the same, as well as, as on other mineral sample don't support the structure described by FO'KB, presumably as a result of a high sample inhomogeneity. Laboratory preparations, of the pure paramelaconite indicated by FO'KB XRD structure failed in all attempts. Present author (DD) prepared the sample containing the mixture of paramelaconite and other oxides, like Ag₂O and PbO. In the meantime, the same author prepared the pure oxide indicated by the paramelaconite stoichiometry $CuO_{0.757}$, and samples exhibit all known properties of the mineral sample (antiferromagnetism, neutron scattering, spiral magnetism, DSC), but XRD reveals structure that of CuO, possibly giving rise to a novel type of copper oxide.

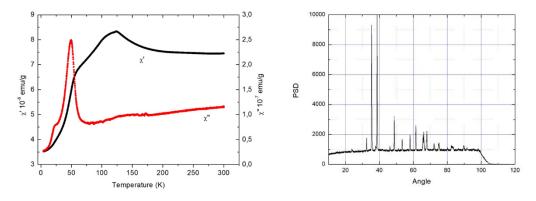


Figure 1. a) Temperature dependence of the magnetic susceptibility of $Cu_4O_{0.75}$, b) XRD of $Cu_4O_{0.75}$.

Keywords

paramelaconite; antiferromagnetism; stoichiometry; novel structures

Section: (III) Ceramic Powders

Copper doped ceria catalyst for soot oxidation

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Abstract

Diesel engines possess high durability, optimal fuel efficiency, and lower carbon dioxide emissions in comparison to petrol engines. However, particulate matter, i.e. soot resulting from incomplete fuel combustion presents a serious environmental and health hazard. Therefore, the advancement of catalytic converters for elimination of harmful exhaust pollutants, or more precisely the development of adequate catalysts for soot oxidation is of great importance. This work deals with catalytic activity of copper doped ceria-alumina nanocatalyst aimed for use in soot oxidation process as a costeffective, sustainable alternative to noble metal based catalysts. Samples without and with 2 mol.% of copper were prepared by combustion synthesis method with glycine as fuel and metal nitrates as oxidants, and subsequently thermally treated at 800 °C for 2 hours. The physicochemical properties of the prepared catalysts were determined using X-ray diffraction analysis, scanning electron microscopy, energy dispersive X-ray spectroscopy, Fourier transformed infrared spectroscopy and differential thermal and thermogravimetric analysis. Kissinger-Akahira-Sunose method was applied for non-isothermal kinetic analysis of the soot oxidation process in the presence of prepared catalysts. The crystallite size of the Cu doped sample is significantly smaller in comparison to the undoped sample, but both catalysts exhibit an extremely porous morphology. The copper doped sample showed the best catalytic activity in the soot oxidation process, as well as a continuous decline in activation energy with increasing degree of conversion.

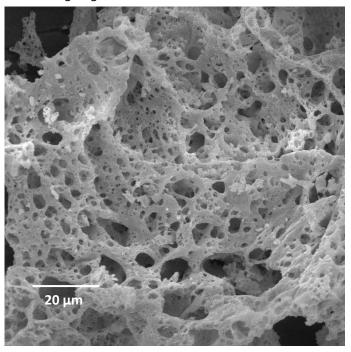


Figure 1. SEM micrograph of 1 mol.% copper doped ceria-alumina sample.

Keywords

ceria; nanocatalysts; combustion synthesis; soot oxidation

Acknowledgement

This work has been fully supported by Croatian Science Foundation under the project IP-01-2018-2963.

Imidacloprid photocatalytic degradation under artificial and natural solar light with sol-gel immobilized TiO₂ and TiO₂/CNT

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Abstract

Suspended photocatalysts tend to be more reactive in photocatalytic degradation, however immobilized photocatalysts are more practical. Accordingly, sol-gel method has been recognized as suitable for synthesis and immobilization of photocatalysts. The most researched photocatalysts is titanium dioxide (TiO_2), yet various modifications have been made to intensify photocatalytic activity in visible spectra range among which is modification with multiwalled carbon nanotubes (CNTs).

Therefore, in this paper, TiO_2 and nanocomposite of TiO_2/CNT were immobilized on fiber glass mesh by modified sol-gel method. The photocatalysts degradation efficiencies were determined based on photocatalytic degradation of imidacloprid, commonly used pesticide, in laboratory and pilot compound parabolic collector (CPC) reactor systems under artificial and natural solar light. By applying the pseudo-first-order reaction kinetics, degradation rate constant was determined (Figure 1). Results indicated that sensitization of solar photocatalysis by the addition of CNT to TiO_2 films, led to intensification of imidacloprid photocatalytic degradation, as well as faster degradation under natural solar light.

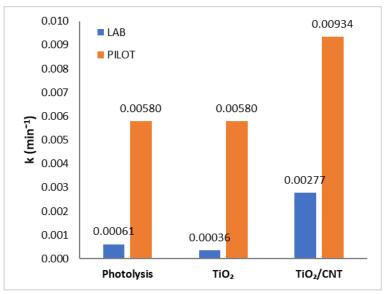


Figure 1. Photolytic and photocatalytic (TiO₂, TiO₂/CNT) degradation rate constants k (min⁻¹) according to pseudo-first-order kinetic model for imidacloprid degradation in lab and pilot CPC reactor setup.

Keywords

imidacloprid; TiO₂ films; TiO₂/CNT nanocomposites; photocatalysis

Acknowledgement

This work has been supported by following project "Waste & Sun for photocatalytic degradation of micropollutants in water" (OS-Mi), KK.01.1.1.04.0006, supported by European Regional Development Fund.

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