VŠB – Technical University of Ostrava
Nanotechnology Centre, CEET

NanoOstrava 2021

7th Nanomaterials and Nanotechnology Meeting

May 17-20, 2021
Ostrava

Book of Abstracts, Programme and General Information
(International conference)
NanoOstrava 2021 – 7th Nanomaterials and Nanotechnology Meeting
Nanotechnology Centre, CEET, VŠB – Technical University of Ostrava,
Czech Republic
May 17-20, 2021 (on-line)

VSB TECHNICAL UNIVERSITY OF OSTRAVA
ENERGY AND ENVIRONMENTAL TECHNOLOGY CENTRE
NANOTECHNOLOGY CENTRE

Book of abstracts was designed by
Ing. Sylva Holešová, Ph.D.

Dear Conference participants, Visitors and Colleagues,

We welcome you to the NanoOstrava 2021 - 7th international biennial 'Nanomaterials and Nanotechnology Meeting' in Ostrava. The conference will be held at **VŠB–TU of Ostrava**, and has been organized under cooperation of scientists from the **Nanotechnology Centre, CEET, VŠB-TU Ostrava** and **Institute of Geonics of CAS**.

Due to the global situation of COVID-19, the international conference NanoOstrava 2021 will be **VIRTUAL** for this year! NanoOstrava 2021 will use a professional virtual platform that will contain all the components of the conference online (oral presentations, e-poster presentations and exhibitions of industrial partners), which will enable participants, exhibitors and sponsors to build effective and long-term relationships.

The meeting is a platform to facilitate open discussions between scientists, students and representatives from companies with an interest in the development of nanomaterials and nanotechnology. **Five scientific sessions** in the field of nanoscience will be introduced by eminent world-known lecturers and a number of scientists and **one session** for presenters from the **industry**. Conference supports young scientists and enthusiastic students helping them to open doors to scientific careers.

Five scientific sessions in the field of nanoscience will be introduced by eminent world-known lecturers and a number of scientists and one session for presenters from the industry.

1. Advanced Nanomaterials
2. Nanomaterials Characterizations and Devices
3. Nanotech for Energy
4. Nanotech for Medicine and Pharmacy
5. Nanotech for Environmental Solution
6. Industrial Forum

We wish you a successful and fruitful conference!

Organizing committee of NOM 2021

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Ladies and gentlemen, dear colleagues,

Allow me, on behalf of the Organizing Committee, to welcome you to the 7th Nanomaterials and Nanotechnology Meeting, known as NanoOstrava 2021. Unfortunately, due to the COVID-19 pandemic, we cannot welcome you personally at this 7th year of the conference in Ostrava and share fruitful scientific communication or establish social contacts at various events, although it would be a great pleasure for us. We therefore thank all of you who have decided to support our virtual conference with active participation. Thanks to you, we can continue the continuous sequence and tradition of the international NanoOstrava conference intended for scientists, students and representatives of companies dealing with nanomaterials and nanotechnologies.

We hope that we have managed to prepare an interesting and informative scientific program for you, which outlines the current progress and activities in various areas of nanotechnology. Over 100 participants from more than 20 countries around the world will present their work in four days.

All conference participants are cordially invited to contribute their original research or review contributions to the Special Issue of the Open Access Journal of MDPI - *Nanomaterials* and/or to the Special Issue of the Open Access Journal of Elsevier - *Materials Today Proceedings*.

I wish you to spend a pleasant and rewarding time at the NanoOstrava 2021 meeting, to discover new and interesting scientific stimuli, to find further inspiration for your work and also to make new contacts, even if only through a virtual meeting. I sincerely hope that in two years, in 2023, we will be able to warmly welcome you directly in Ostrava.

Daniela Plachá
Chairman, NanoOstrava2021

SPONSORS

The organizers would like to thank the following companies for generously sponsoring this meeting:

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CONFERENCE PAPERS

Conference full papers should present these substantial conditions: **high quality research**, original ideas, not published anywhere else. Conference full papers will be published in following journals.

Special papers in Open Access Journals:

Nanomaterials

is an international and interdisciplinary scholarly open access journal. It publishes reviews, regular research papers, communications, and short notes that are relevant to any field of study that involves nanomaterials, with respect to their science and application. Thus, theoretical and experimental articles will be accepted, along with articles that deal with the synthesis and use of nanomaterials. **Nanomaterials** is dedicated to a high scientific standard. All manuscripts undergo a rigorous reviewing process and decisions are based on the recommendations of independent reviewers.

Current IF= 4.324 (2019)

Participants of the conference are cordially invited to contribute original research papers or reviews to special issue **"Nanomaterials and Nanotechnology Meeting 2021"**.

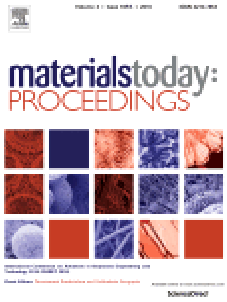
For publication in Nanomaterials, please contact: **Daniela Plachá** (daniela.placha@vsb.cz).

Deadline for manuscript submissions: **31 December 2021**

The agreement established between conference organizers and MDPI publisher allows for manuscripts submission to these special issues at a discount on the APCs.

Conference proceedings:

Materials Today: Proceedings



Materials Today: Proceedings provides the materials science community with a fast and flexible route to the publication of research presented at leading scientific conferences spanning the field of materials science, technology and engineering.

For publication in Materials Today: Proceedings send your manuscript to: **Grażyna Simha Martynková** (grazyna.simha@vsb.cz).

Deadline for manuscript submissions: **1 June 2021**

PROGRAMME

NanoOstrava 2021

Monday
17 May, 2021

8:00				
9:00	10:00	Opening Ceremony		
10:00	10:30	**Plenary presentation T1**	**Rummeli Mark H.**	*Towards atom precise synthesis and engineering of nanomaterials with electron beams*
10:30	10:45	15 min break		
10:45	11:15	**T1 (In)**	**Lafdi Khalid**	*Nanocomposites Wireless Sensing Design for Composite Degradation*
11:15	11:30	T1	**Tamayo Vegas Sebastian**	*Effect of CNT additives on the physical properties of derived nanocomposites (experimental and modelling)*
11:30	11:45	T6 (PREVAC)	**Walczak Lukasz**	*Boosted research of nanomaterials by XPS and HPXPS*
11:45	12:00	T1	**Bakandritsos Aristides**	*Advanced nanomaterials for catalysis*
12:00	12:15	T1	**Gulyaeva Irina**	*Gas-sensitive properties of ZnO-SnO2 films prepared via sol-gel method*
12:15	13:00	45 min break		
13:00	13:30	**T1 (In)**	**Pacáková Barbara**	*Game of clays - from single layers to functional multilayers*
13:30	13:45	T6 (Anton Paar)	**Špringer Jiří**	*Surface zeta potential analysis of novel graphene oxide coatings for membranes*
13:45	14:00	T1	**Fang Qichen**	*Development studies of silver nanocomposite based sensors for acid penetration*
14:00	14:15	T1	**Muhsan Ali**	*Fabrication and characterization of graphene based de-icing heater*
14:15	14:30	T1	**Starnikova Alexandra**	*Investigation of the electrophysical and gas-sensitive properties of thin nanocomposite materials based on ZnO (Sn) and ZnO (Au) nanorods*
14:30	14:45	15 min break		
14:45	15:00	T6 (Anton Paar)	**Stavárek Martin**	*Nanoscale Investigation of Multilayer Graphene Flakes using Contactless Electrical Modes of Tosca Atomic Force Microscope*
15:00	15:15	T1	**Kubáň Vít**	*Graphene oxide thin film with silver nanoparticle*
15:15	15:30	T1	**Skoura Eva**	*The functionalization of polycaprolactone surface with organo-saponite and methylene blue nanoparticles*
15:30	15:45	T1	**Vaculíková Lenka**	*Preparation and characterization of manganese dioxide/montmorillonite composites and their catalytic activity*
15:45	16:00	T1	**Motoc Sorina**	*Graphene quantum dots-multiwalled carbon nanotubes paste electrode for sensitive determination of doxorubicin in aqueous solution*

Tuesday
18 May, 2021

8:00				
9:00	9:30	**T4 (In)**	**Richardson Des R.**	*Targeting metastasis in cancer through the metastasis suppressor, NDRG1, using innovative therapeutics*
9:30	10:00	**T4 (In)**	**Jampílek Josef**	*Nanomaterials as antiparasitics*
10:00	10:15	T4	**Pisárčik Martin**	*Structure-property relationship study of silver nanoparticles stabilised with ammonium and phosphonium surfactants*
10:15	10:30	T6 (Bruker)	**Unger Miriam**	*Infrared (IR) Chemical Identification at the Nanoscale – When AFM meets IR*
10:30	10:45	**15 min break**		
10:45	11:15	**T4 (In)**	**Sobczak-Kupiec Agnieszka**	*Albumin loaded PEG/HAp composite coatings with bone regenerative potential*
11:15	11:45	**T4 (In)**	**Dubiel Agnieszka**	*Modification of Al203 and ZrO2 surfaces dedicated as a filler for polymers used in biomedical engineering*
11:45	12:15	**T4 (In)**	**Gerber Sandrine**	*Multifunctional Harmonic Nanoparticles for Cell Tracking and Theranostic Applications*
12:15	13:00	**45 min break**		
13:00	13:30	**T4 (In)**	**Calin Manuela**	*Targeted therapies based on nanocarriers to reduce vascular inflammation*
13:30	14:00	**T4 (In)**	**Taratuta Anna**	*NiTi alloy substrate preparation effect on the adhesion of Ta2O5 layer applied by ALD method*
14:00	14:30	T6 (Specion s.r.o.)	**Steffen Robert**	*Combining ion beam and electron beam techniques to better understand LiB materials*
14:30	14:45	**15 min break**		
14:45	15:00	T4	**Ščuková Michaela**	*Thin polymeric films with antimicrobial nanofiller*
15:00	15:15	T4	**Bardoňová Lenka**	*Encapsulation of hydrophobic drugs in hyaluronic acid nanofibers: effect of the solution preparation procedure*
15:15	15:45	**T4 (In)**	**Weissig Volkmar**	*Mitochondria-Targeted Pharmaceutical Nanocarriers*

Wednesday
19 May, 2021

8:00				
9:00	9:30	**T3(In)**	**Çetinkaya Tuğrul**	*State of the art nanostructured cathode materials for lithium-oxygen batteries*
9:30	9:45	T3	**Yuliasari Fitri**	*Improved DSSC performance with coral-like shape ZnO nanorods grown by using Zn(II)AA/ZnO-NP seed layer*
9:45	10:00	T3	**Govatsi Katerina**	*Photoelectrochemical water splitting of core/sheath nanowires based on ZnO and transition metal dichalcogenides*
10:00	10:15	T3	**Bakandritsos Aristides**	*Two-dimensional organic materials for energy and the environment*
10:15	10:30	T3	**Kment Štěpán**	*Hybrid nanostructures for (photo)electrochemical water splitting*
10:30	10:45	**15 min break**		
10:45	11:00	T3	**Dombaycıoğlu Şeyma**	*Development of lithiated ion exchange membranes for Li-S batteries*
11:00	11:15	T3	**Gulyaeva Irina**	*Study of the verticallly oriented domain structure of nanosized PZT film*
11:15	11:30	T6 (Optik Instruments)	**Matoušek David**	*Modern FTIR spectroscopy techniques for characterization of ultra-thin films*
11:30	11:45	T3	**Simha Martynková Gražyna**	*Nanocarbons in composites*
11:45	12:00	T3	**Sathish Sajjan Kumar**	*Study of clay mineral composites for Lithium batteries*
12:00	12:15	T6 (Měřící technika Morava s.r.o.)	**Lukeš Jaroslav**	*Nanomechanical Characterization of Battery Materials*
12:15	13:00	**45 min break**		
13:00	13:30	**T5(In)**	**Rai Mahendra**	*Biogenic silver nanoparticles: What we know and what we need to know?*
13:30	14:00	**T5(In)**	**Šafařík Ivo**	*Magnetic materials in biosciences, biotechnology and environmental technology*
14:00	14:15	T5	**Zinicovscaia Inga**	*Assessment of metal nanoparticles accumulation in organs and their effect on cognitive abilities of mice*
14:15	14:30	T5	**Lazaratou Christina Vasiliki**	*Ca-palygorskite clay mineral for Fe2+ removal from water systemsfrom molecular simulations point of view*
14:30	14:45	**15 min break**		
14:45	15:15	**T5(In)**	**Piszter Gábor**	*Chemically selective vapor sensing of structurally colored butterfly wings*
15:15	15:30	T5	**Kurtinová Sindy**	*Hetero- and homo-aggregation of ZnO nanoparticles in different size fractions of colloids extracted from agricultural soil*
15:30	15:45	T5	**Smýkalová Aneta**	*Graphitic carbon nitride prepared from different precursors for photocatalytic degradation of pollutants in water*
15:45	16:00	T5	**Tokarčíková Michaela**	*Experimental verification of regenerable magnetically modified montmorillonite and its application for heavy metals removal from metallurgical waste leachates*

Thursday
20 May, 2021

8:00				
9:00	9:30	**T2(In)**	**Pálková Helena**	*Infrared spectroscopy as powerful tool to study clay minerals subjected to various types of modification*
9:30	10:00	**T2(In)**	**Ta Huy Q.**	*Stranski-Krastanov and Volmer-Weber CVD Growth Regimes to Control Stacking Order in Bilayer Graphene*
10:00	10:15	T2	**Dorovskikh Svetlana**	*Gas-phase deposition of noble metal coatings for medical applications*
10:15	10:30	T6 (Anamet s.r.o.)	**Dudák Michal**	*Analytical instruments for nanoparticles, nanofibers and nanopores*
10:30	10:45	**15 min break**		
10:45	11:15	T6 (NenoVision s.r.o.)	**Hegrová Veronika**	*Correlative Probe and Electron Microscopy using AFM-in-SE*
11:15	11:30	T2	**Volkova Maria**	*TiO2-SnO2 films: synthesis by low-temperature pyrolysis and electrophysical properties*
11:30	11:45	T2	**Plesník Lukáš**	*Structural and phase variability study of the PVDF nanocomposite materials*
11:45	12:00	T2	**Zeynep Aslan**	*Silver nanoparticles stabilized with organic acid*
12:00	12:15	T2	**Khammassi Sabrine**	*Micromechanical characterization of Carbon Black reinforced epoxy nanocomposite using micro indentation*
12:15	13:00	**45 min break**		
13:00	13:15	T4	**Pospíšil Miroslav**	*Halloysite nanotubes as suitable carriers for irinotecan from molecular simulations point of view*
13:15	13:30	T6 (HORIBA Scientific)	**Chaigneau Marc**	*Scanning NV Magnetometry with ProteusQ*
13:30	13:45	T5	**Kratošová Gabriela**	*Microfluidics and biotechnology for nanoparticles synthesis in a green way*
13:45	14:00	T2	**Gelnarová Zuzana**	*Deposition, laser lithography and wet etching of silicon nitride thin films*
14:00	14:15	T2	**Kohut Tomáš**	*Setting of exposure parameters for direct laser writing in optical lithography*
14:15	14:30	T2	**Horníček Jiří**	*Development of materials and 1D photonic crystals by reactive magnetron sputtering*
14:30	14:45	**Closing ceremony**		

LIST OF POSTER PRESENTATIONS

Topic 1 – Advanced Nanomaterials

P1_S

Michal Čičkan: Preparation of light conversion materials from aluminosilicate nanoparticles

P2

Lenka Pazourková: Study of interactions between bioapatite from human bone and Ca – deficient hydroxyapatite

P3_S

Julie Smijová: *Comparison of hydrothermal and microwave synthesis of nano-ZnS and their influence on photocatalytic activity*

P4_S

Monika Šuteková: *Improving photoactivity of colloidal systems of fluorubine dye and saponite*

P5

Nikolai Tarasenko: *Laser and discharge plasma assisted synthesis of cobalt doped zinc oxide nanostructures in liquids*

P6

Věra Valovičová: *Surface properties of smectites fine fractions as precursors of multifunctional composites*

P7_S

Jakub Zágora: *Preparation of polylactide nanoparticles by supercritical fluid technology*

Topic 2 – Nanomaterials Characterizations and Devices

P8_S

Ivan Kopal: *The photochemical reactions of 4-aminobenzenethiol on the plasmonic nanostructured surfaces*

P9_S

Alexey Rezvan: *Local anodic oxidation of silicon for crossbar architecture*

P10_S

Denis Tryakhov: *MOCVD growth and study of hybrid heterostructures based on Pd particles and metal phthalocyanines*

P11_S

Maria Volkova: *The optical and electrophysical properties of Al-ZnO thin films*

P12_S

Pravind Yadav: Controlled deposition of polymer brush films to develop sub 5nm zirconium oxide films by liquid phase ion inclusion

Topic 3 – Nanotech for Energy

P13_S

Alexadra Starnikova: *The use of rapid thermal annealing for the formation of cobalt oxide films*

Topic 4 – Nanotech for Medicine and Pharmacy

P14

Liliya Batyuk: *Study of UDD under experimental carcinogenesis*

P15

Josef Jampílek: *The use of different silica gel RP-TLC plates for the determination the lipophilicity parameters of metformin and phenformin*

P16_S

Nikita Salavatov: *Gold nanorods with organosilica shell as potential dual-therapeutic agents*

P17

Kateřina Škrlová: *Biodegradable polymeric materials with antimicrobial effects suitable for medical applications*

P18_S

Joanna Trzcinska-Wencel: *Green synthesized silver nanoparticles: antibacterial activity, biocompatibility, and analyses of surface-attached proteins*

P19_S

Zuzana Vilamová: *Ag nanoparticles fixation on polycaprolactone-polyvinyl alcohol membrane and biocompatibility evaluation study*

P20

Magdalena Wypij: *Mechanistic insights onto the synergetic effects of AgNPs and antibiotics*

Topic 5 – Nanotech for Environmental Solution

P21

Eva Plevová: *Sorption study on diclofenac removal using organo-beidellite*

P22

Jana Seidlerová: *Application of sorbents modified by nanoparticles*

GENERAL INFORMATION ABOUT POSTER SECTION

The poster section will be available throughout the conference programme under a special channel in MS Teams, where you can view each poster and chat with the authors.

The **best 3 student posters** will be awarded. A special evaluation committee will be set up and during the poster section all student posters will be evaluated and the best 3 will be awarded a diploma. The results will be announced during the closing ceremony.

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Topic 1 – Advanced Nanomaterials

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Topic 3 –Nanotech for Energy

Invited lecture (IL):

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Topic 4 –Nanotech for Medicine and Pharmacy

Invited lectures (IL):

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Poster presentations (PP):

Topic 5 –Nanotech for Environmenal Solution

Invited lectures (IL):

Oral presentations (OP):

Poster presentations (PP):

Topic 6 –Industrial Forum

TOPIC 1

Advanced Nanomaterials

Chair: Miroslav Pospíšil

Nanoparticles – natural and synthetic
Nanocarbons, Nanoclays and Nanoceramics
Functionalization of nanomaterials
Nanocomposites and nanocatalysis

Invited lectures (IL):

Development of Nanocomposites Based Sensors Using Molecular/Polymer/Nano-Additive Routes

Khalid Lafdi[1,2] and Liu Chang[1]

[1]Chemical and Materials Engineering Department University of Dayton, Dayton, OH 45469-USA;
[2]Department of Mechanical and Construction Engineering, Northumbria University, Newcastle upon Tyne, UK

klafdi1@udayton.edu

In this study, nanocomposite durability sensors were developed in order to detect the diffusion of chemicals in real-time. Three nanocomposites-based sensors were fabricated using molecular/polymer/nanoadditives routes. Every approach has led to various sensors with large range of electrical conductivities. The first approach uses molecular additive such as a liquid crystal which could improve the crystallite size and orientation. In the second approach, polymer based conductive network (polyaniline) was explored. Phase separation of the polymer blend was manipulated to construct a compacted conductive network of conjugated polymer nanoparticles (polyaniline). In the third approach, carbon nanoparticles (carbon nanotube and carbon black) were used by monitoring their weight %. The sensing behavior is represented by the decrease of electrical resistance. A numerical multi-stage model was developed to evaluate the formation of the conductive network and its sensing property. The modeling work shows two major parameters affect the sensing behavior: (1) The aspect ratio of conductive nano-additive. (2) Diffusion coefficient ratio of different ions in different phases.

In this study, we used Radio Frequency Identification (RFID) as wireless sensing technology. A sensor embedded composite panel was prepared and tested in a harsh environment, 30 wt% H2SO4. Additionally, we have modified an RFID tag with conductive nanocomposite for selective sensing. We have found that the RFID tags are sensitive sensors for detecting penetration of chemicals in composites.

Game of clays

B. Pacakova[1], M. Matejdes[2], M. Daab[2], P. Brito[1], O.T. Neto[1], K. Hunvik[1], S. Raaen[1], J. Breu[2], J. O. Fossum[1]

[1]Departement of Physics, Norwegian University of Science and Technology, Trondheim, Norway

[2]Bayerisches Polymer Institut und Lehrstuhl für Anorganische Chemie 1, Universität Bayreuth, Bayreuth, Germany

barbara.pacakova@ntnu.no

Smectite clays represent very interesting class of layered materials[1,2,3], that can be combined with other building blocks to create composites with specific functionality. The ability of smectites to relatively easily swell[2], exfoliate[1] or intercalate various types of molecules into the interlayers[4] make them the ideal confined containers and reactors, that allow formation of the layered composite materials.

Smectite clays belong to the family of layered silicates and exhibit the net negative charge of the layers which is compensated by the positively charged interlayer cation. Properties of smectites are strongly dependent on the type of the interlayer cation [1–5] and its characteristic parameters, such as the charge, ionic radii, hydration enthalpy or polarizability[7]. Moreover, one of the important parameters distinctive for smectites is the cation exchange capacity, with a preference for easy exchange of smaller cation for the larger one. In case of synthetic fluorohectorite, interlamellar distance can be easily varied from 0.9 up to 14 nm, which allows its use as a confined reactor for material growth.

Smectite clays can be exfoliated into the form of 1 nm thin sheets. Combination of exfoliated clay sheets with Van der Waals layered materials, such as graphene together with graphene oxide and graphite, brings promising ways towards applications in electronics (selective ionic transport, field-effect transistors as an example).

In this talk, we will focus briefly on basic properties of synthetic fluorohectorite clay and its applications, with emphasis on the clay-graphene multi-layered sandwich heterostructures. Synthesis of graphene/graphitic structures in the fluorohectorite interlayers allows simultaneous band-gap opening in graphene and formation of final heterostructures in one step. Such combination of graphene with layered silicate is a promising step towards the real applications[8].

Keywords: smectites, fluorohectorite, graphene-clay heterostructures, structural colours, single layer sheets

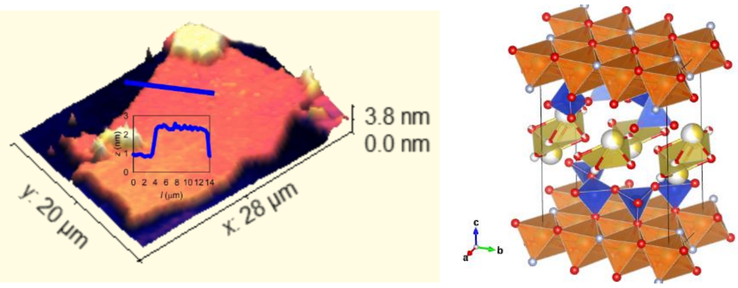

Figure 1. Left: AFM of single layer fluorohectorite sheet. Right: crystal structure of fluorohectorite.

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Towards Atom Precise Synthesis and Engineering with Electron Microscopy

Mark H. Rümmeli

[1]College of Energy, Soochow, Institute for Energy and Materials Innovations, Soochow University, Suzhou 215006, China;

[2]Centre of Polymer and Carbon Materials, Polish Academy of Sciences, M. Curie Sklodowskiej 34, Zabrze 41-819, Poland;

[3]Institute for Complex Materials, IFW Dresden, 20 Helmholtz Strasse, Dresden 01069, Germany;

[4]Center for Energy and Environmental Technologies, VŠB-Technical University of Ostrava, 17. Listopadu 15, Ostrava 708 33, Czech Republic;

Mhr1967@yahoo.com

A key feature of my research is to develop a transmission electron microscope (TEM) into an atomic-scale laboratory to fabricate, modify and characterize samples so that crucial structure property studies and synthesis studies can be conducted with high spatial resolution and high temporal resolution. Thus, the core of this presentation will look at a variety of electron beam driven chemical reactions and electron beam engineering techniques in which we can fabricate and manipulate nano-materials at the atomic level.

Keywords: TEM, nanomaterials, synthesis, engineering, in situ.

Oral presentations (OP):

Advanced nanomaterials for catalysis

A. Bakandritsos[1,2], R. G. Kadam[1], P. Kumar[1], G. Zoppellaro[1], M. B. Gawande[1],
P. Fornasiero[3], M. Otyepka[1,2], R. Zbořil[1,2]

[1]Regional Centre of Advanced Technologies and Materials, CATRIN, Palacký University, Olomouc, Czech Republic; [2]Nanotechnology Centre, Centre of Energy and Environmental Technologies, VŠB–Technical University of Ostrava, Czech Republic; [3]Department of Chemical and Pharmaceutical Sciences, INSTM Trieste and ICCOM-CNR Trieste Research Units, University of Trieste, Italy.

a.bakandritsos@upol.cz

Single-atom-catalysts aim at bridging the gap between homogeneous and heterogeneous catalysis. The challenge is the development of materials with ligands enabling coordination of metal atoms in different valence states, and preventing leaching or nanoparticle formation.[1]

Exploiting fluorographene's chemistry, a two-dimensional ligand was developed as a metal atom trap. Specifically, graphene functionalized with nitrile groups (cyanographene[2]) was employed for the robust coordination of Cu(II) ions, which were partially reduced to Cu(I) due to graphene-induced charge-transfer. Inspired by nature's selection of Cu(I) in enzymes for oxygen activation, this two-dimensional mixed-valence SAC was evaluated performing flawlessly in an O_2-mediated reaction involving the oxidative coupling of amines towards high-value pharmaceutical synthons. High conversions (up to 98%), selectivities (up to 99%), and recyclability were attained with very low metal loadings in the reaction. The synergistic effect/cooperation of Cu(II) and Cu(I) was identified as an essential part in the reaction mechanism.[3]

The developed strategy opens the door for a broad portfolio of other single-atom-catalysts via their coordination to thee functional groups of graphene, as demonstrated by the successful entrapment of Pt^{II}/Pt^{IV} single atoms to carboxy-graphene.

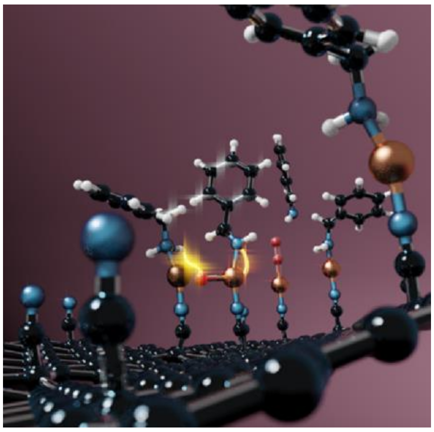

Figure 1. Cyanographene acts as a 2D coordination ligand for Cu ions and leads to a mixed valence, single-atom Cu(II)/Cu(I) catalyst. Oxidative coupling of amines takes place very efficiently through oxygen activation and opens the doors for heterogeneous and cooperative mixed valence single-atom catalysis.

Acknowledgments: The work was supported by the ERDF/ESF project "Nano4Future" (No. CZ.02.1.01/0.0/0.0/16_019/0000754) and by GACR (project No 19-27454X).

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Development studies of silver nanocomposite-based sensors for acid penetration

Qichen Fang[1], Khalid Lafdi[1,2]

[1]Chemical and Materials Engineering Department University of Dayton, Dayton, OH 45469-USA;
[2]Department of Mechanical and Construction Engineering, Northumbria University, Newcastle upon Tyne, UK

fangq1@udayton.edu

Polymer composites are widely used to store liquid chemicals. Monitoring their structural health has attracted a lot of attention recently. The geometry of nanomaterial in polymer composites plays an important role in affecting the penetration progress, thus altering the durability of polymer composite. In this study, the silver nanocomposite-based SHM sensors are designed to detect the progress of chemical diffusion and material degradation as a function of time. A comparison between the AgNW sensor and AgNP sensors is used to study the geometry of silver nanomaterial's effect on acid penetration. It appeared that the SHM sensors' resistance decrease in three stages with the diffusion time increase. Moreover, with the volume percentage of silver nanomaterial increase, the response time decrease. And the AgNW based sensors have shown a shorter response time. An aggregation model was developed to better understand the nanomaterial's geometry effect on acid penetration progress.

Keywords: Silver nanocomposite, sensors, acid penetration, structural health monitoring.

Acknowledgments: We gratefully thank Chang Liu (Lerner Research Institute, Cleveland Clinic Foundation) for his help in the simulation.

Gas-sensitive properties of ZnO-SnO$_2$ films prepared via sol-gel method

I.A. Gulyaeva[1], A.P. Starnikova[1], M.G. Volkova[2], V. Yu. Storozhenko[2], E.M. Bayan[2], V.V. Petrov[1]

[1]Research and Education Centre «Microsystem technics and multisensory monitoring systems", Southern Federal University, Russia; [2]Department of Chemistry, Southern Federal University, Russia;

iten@sfedu.ru

Thin nanosized ZnO-SnO$_2$ films with a ZnO:SnO ratio of 0.5: 99.5, 1:99, and 5:95 mol% were synthesized by the sol-gel method. XRD data confirmed the structure of cassiterite regardless of the concentration of additives added. The largest particle size and the best crystallization are noted for

films with a Zn: Sn ratio of 1:99. This was due to the maximum potential barrier for these films. Gas sensors based on films made of these materials can have higher gas sensitivity coefficients.

Keywords: ZnO-SnO_2 films, sol-gel, tin dioxide

Acknowledgments: This work was financially supported by the RFBR, project 20-07-00653 A.

Study of modified graphene oxide with silver nanoparticles

V. Kubáň[1,2], G.S. Martynková[2], G. Kratošová[2]

[1]Faculty of Materials Technology, VSB-TUO, Czech Republic; [2]Nanotechnology Centre, CEET, VSB-TUO, Czech Republic;

vit.kuban.st@vsb.cz

This work deals with the preparation and characterization of thin films of modified graphene oxide with bio-nano-silver. Graphene oxide prepared by the modified Hummers method was preserved in liquid form. The modification with silver nanoparticles was carefully tailored using phystosynthetically prepared colloid. The creation of thin films from pure and modified graphene oxide. The films differ in the flow rate of the silver nanoparticle reactant during their biosynthesis in the microfluidic reactor and the thickness of the films. The resulting materials were characterized by basic and instrumental analytical methods, namely XRD, STEM, FTIR and DSC. The individual films of the modified GO were compared with each other and compared with reference pure GO films. Comparison of prepared materials and confrontation with commercial material reduced GO, it was found that modification with nanosilver even in such a small content has an effect on the properties of the material. The film prepared based on Ag-modified GO is more temperature stable, its surface is smoother and hydrophobicity was observed.

Keywords: Graphene oxide, nanosilver, morphology, graphite exfoliation

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Graphene quantum dots-multiwalled carbon nanotubes paste electrode for sensitive determination of doxorubicin in aqueous solution

Sorina Motoc[1], Bianca Schinteie[2], Florica Manea[2], Aniela Pop[2], Sorina Negrea[3,4], Elisabeta I. Szerb[1]

[1]"Coriolan Dragulescu" Institute of Chemistry, Romanian Academy, 24 Mihai Viteazu Bvd., 300223-Timisoara, Romania; [2] Politehnica University of Timisoara, 2 Victoriei Square, 300006-Timisoara, Romania; [3]National Institute of Research and Development for Industrial Ecology (INCD ECOIND), 300431, Timisoara Branch, Romania; [4]"Gheorghe Asachi" Technical University of Iasi, Department of Environmental Engineering and Management, 700050, Iasi, Romania.

sorinailies@acad-icht.tm.edu.ro

In this work, a new electrochemical sensor consisted of graphene quantum dots (15 %, wt) and multiwalled carbon nanotubes (35%, wt.) in paraffin oil (50%, wt), named GRQD-CNT, was obtained

for determination of anticancer chemotherapy agent adriamycin, doxorubicin (DOX), in the aqueous solution. GRQD-CNT was selected based on the electrochemical response from carbon-based electrodes, *i.e.*, multiwall carbon nanotubes (33.3 %, wt) in paraffin oil (66.66%, wt), named CNT, and commercial boron-doped diamond electrode (BDD). The content of the grapheme quantum dots of GRQD-CNT composition was selected in relation with the stability of the electrochemical response. GRQD-CNT was characterized morphostructurally by scanning electron microscopy (SEM) and electrochemically through ferry/ferrocyanide classical method using cyclic voltammetry (CV) technique. In comparison with BDD and CNT electrodes, the CV response of DOX was enhanced, meanwhile the peak potential negatively shifted and higher current signals due to GRQD-CNT exhibited a large specific surface area and specific electroactive surface area, and good electronic properties. The content of DOX was effectively accumulated on the surface of the GRQD-CNT electrode and generated a pair of redox peaks at around 0.31 and 0.53 V (vs. Ag/AgCl) in 0.1 M Na_2SO_4 supporting electrolyte. Cyclic voltammetry (CV), differential-pulsed voltammetry (DPV) and square-wave voltammetry (SWV) were tested to optimize the best voltammetric procedure for DOX determination. Also, chronoamperometry (CA) and multiple-pulsed amperometry (MPA) allowed to find the best amperometric conditions for DOX detection. Under the optimized conditions of SWV applying, there was a linear correlation between anodic peak current and concentration of DOX in the range of 10–60 μgL^{-1} with the detection limit of 1.24 μgL^{-1}. The adsorption of DOX onto GRQD-CNT within the medium DOX concentrations range led to the electrode surface fouling but for trace levels of DOX concentrations, the adsorption aspect favored the development of a preconcentration-prior detection step that enhanced at least ten times the lowest limit of detection. The preconcentration step was assured by simple immersing of the electrode into DOX-containing aqueous solution for a 5 minutes period. This electrochemical sensor displayed excellent precision, sensitivity, and repeatability in the determination of DOX in the aqueous solution. The detection results were validated through DOX determination in real surface water.

Acknowledgments: Funding for this study was provided by a grant of the Romanian Ministry of Research and Innovation, CNCS - UEFISCDI, project number PD 88/2020, project code PN-III-P1-1.1-PD-2019-0676, within PNCDI III, and by a grant of the Romanian Ministry of Education and Research, CNCS - UEFISCDI, project number PN-III-P4-ID-PCE-2020-1958, within PNCDI III

Fabrication and characterization of graphene-based de-icing heater

A. Muhsan[1,2], K. Lafdi[1,3]

[1] University of Dayton, Dayton, Department of Chemical and Materials Engineering, OH, 45469, USA; [2] University of Technology, Department of Materials Engineering, Baghdad, 10066, Iraq; [3] Northumbria University, Department of Mechanical Engineering, Newcastle upon Tyne, NE1 8ST, UK

muhsana1@udayton.edu

In this study, graphene-based paper was utilized as a potential deicing material. Vacuum bagging technique was used to fabricate the composite panel which consisted of graphene-based paper and epoxy/glass fiber. The techniques were used to fabricate graphene-based paper were chemical vapor deposition (CVD) and hot-pressing techniques. Scanning electron microscopy (SEM), optical microscopy, X-ray diffraction and Raman spectroscopy were used to characterize graphene based paper and composite panels. The thermal conductivity and electrical conductivity were measured for graphene-based paper. The temperature distribution in the composite panel during deicing tests was studied using infrared thermal imaging. The deicing test based on the joule heating of the composite panel by applying electrical potential. Various electrical potential applied to the composite panel to investigate the role of potential applied on the deicing process. Based on the results, the optimum potential applied 5 V was sufficient to remove ice from panel's surface with low power consumption. The temperature profile of composite panels showed higher temperature for composite panel of CVD graphene-based paper compared by hot pressing sample because of higher in-plane thermal conductivity for CVD graphene-based paper.

Keywords: Deicing technique, Graphene-based paper, Composite panel, Joule heating.

The functionalization of polycaprolactone surface with organo-saponite and methylene blue nanoparticles

E. Skoura[1], P. Boháč[1,2], J. Bujdák[1,3]

[1]Slovak Academy of Science, Institute of Inorganic Chemistry, Bratislava, Slovak Republic; [2]Centre of Excellence for Advanced Materials Application (CEMEA), Slovak Academy of Sciences, Bratislava, Slovak Republic; [3] Comenius University in Bratislava, Faculty of Natural Sciences, Department of Physical and Theoretical Chemistry, Bratislava, Slovak Republic

eva.skoura@savba.sk

Synthetic saponite (Sap) was modified with hexadecyltrimethylammonium (HDTMA) cations leading to highly hydrophobic material. Subsequently, the organoclay was functionalized with variable amounts of methylene blue (MB). The suspensions of Sap/HDTMA/MB were filtered through Teflon membranes to prepared thin films. The synthesis of nanocomposites with polycaprolactone (PCL) with the functionalized organoclays was performed via melt diffusion at the interface of the film and the polymer. Pristine silicate and modified materials, as well as the final nanocomposites with the modified surface, were characterized by a combination of analytical techniques including UV-Vis absorption and fluorescence spectroscopy.

Keywords: smectite, photosensitizer, organic dye, polymer nanocomposite

Acknowledgments: This work was supported by the Slovak Research and Development Agency under contracts No. APVV-15-0347, APVV-15-0741, APVV-18-0075. Supports from the VEGA grant agency (1/0227/20, 2/0141/17) are also gratefully acknowledged.

Investigation of the electrophysical and gas-sensitive properties of thin nanocomposite materials based on ZnO (Sn) and ZnO (Au) nanorods

A.P. Starnikova[1], I.A. Gulyaeva[1], Kh. A. Abdullin[2], V.V. Petrov[1]

[1]Research and Education Centre «Microsystem technics and multisensory monitoring systems”, Southern Federal University, Russia [2] Al Farabi Kazakh National University, National Nanotechnology Laboratory of Open Type, Kazakhstan

starnikova@sfedu.ru

Recently, the most widespread materials for gas sensors are nanocomposites based on semiconducting metal oxides. Arrays of ZnO nanorods were synthesized on glass substrates by the hydrothermal method. Formed ZnO nanorods with a predominantly vertical orientation have an average transverse size of about 30-40 nm and a length of 500-600 nm. On top of the obtained ZnO nanorods, a thin layer of gold was deposited onto one of the samples, and a thin layer of tin was deposited on the other. The responses of the sample with the composition ZnO (Sn) showed the highest value of the gas sensitivity index equal to 47 at 200 °C in comparison with the material of the composition ZnO (Au).

Keywords: ZnO nanorods, tin dioxide

Acknowledgments: This work was financially supported by the RFBR, project 20-07-00653 A.

Effect of CNT additives on the electrical properties of derived nanocomposites (experimentally and modelling)

S.Tamayo-Vegas[1], A. Muhsan[2], L. Chang[2], M. Tarfaoui[3], K. Lafdi[1]

[1]Department of Mechanical Engineering, Northumbria University, United Kingdom; [2]Department of chemical engineering, University of Dayton, United States; [3]Department of Mechanical Engineering, ENSTA Bretagne, France

w18002002@northumbria.ac.uk

Polymeric nanocomposites (PNCs) are in continuous demand in many industrial applications. Especially with the rapid progress in electronics, PNCs with enhanced electrical and mechanical properties are being developed with the help of nanotechnology. Among carbon fillers, carbon nanotubes (CNTs) are extraordinarily superior in their physical properties. With the addition of small amounts into polymeric matrices, the electrical conductivity is greatly improved. However, different parameters affect the final conductivity. Thus, in this study, we have investigated the effects of MWCNT parameters i.e. aspect ratio, orientation, and intrinsic conductivity. throughout modelling in DIGIMAT software with two approaches, mean-field homogenization and finite element method. In addition, the numerical data were compared with our experimental data finding a great correlation. Also, the findings suggest that the mean field homogenization has a good accuracy below the percolation threshold while FE simulations showed an exceptional correlation above the percolation threshold. Moreover, the intrinsic conductivity of the MWCNT was found is the critical parameter to correlate with experimental data. Finally, this study provides sufficient evidence that both tools are powerful to predict the electrical conductivity of nanocomposites.

Keywords: Nanocomposites, Carbon nanotubes, Electrical properties, Modelling.

Acknowledgments: Authors can acknowledge funding agencies or any person that has made a significant contribution to the work.

Preparation, characterization and catalytic activity of manganese dioxide/montmorillonite composites

L. Vaculíková[1], V. Valovičová[1], E. Plevová[1], B. D. Napruszewska[2], D. Duraczyńska[2], R. Karcz[2], E. M. Serwicka[2]

[1]Institute of Geonics of the Czech Academy of Sciences, The Czech Republic; [2]Haber Institute of Catalysis and Surface Chemistry, Polish Academy of Sciences, Poland

lenka.vaculikova@ugn.cas.cz ; ncserwic@cyf-kr.edu.pl

Manganese oxides are currently a very promising materials for the development of new catalysts for total oxidation reactions of volatile organic compounds (VOCs), due to their high catalytic combustion performance, environmental friendliness and low cost. The aim of the experimental study was to prepare MnO_2/clay composites using the reductive procedure with $KMnO_4$ from different types of montmorillonites, and verify their catalytic activity in combustion of toluene and ethanol, a model reaction used for determination of the catalyst ability to destroy toxic volatile organic pollutants of air. Detailed characterization of the structure and physico-chemical properties of the obtained composite materials confirmed the presence of cryptomelane - type manganese dioxide (K-MnO2), which is a particularly suitable material for catalytic reactions. In catalytic tests, all cryptomelane/montmorillonite composites showed higher activity than the reference MnO_2.

Keywords: MnO_2/montmorillonite composite, cryptomelane, catalytic combustion of VOCs

Acknowledgments: This work was supported by Mobility program of the Czech Academy of Sciences and the Slovak Academy of Sciences, Mobility Plus Project, ID number SAV-21-08. Part of this work was supported financially by the statutory research fund of ICSC PAS. Procedures for catalytic testing were developed within the project financed by the National Science Centre, Poland (OPUS, UMO-2017/27/B/ST5/01834).

Poster presentations (PP):

Preparation of light conversion materials from aluminosilicate nanoparticles

M. Čičkan[1], M. Janek[1,2]

[1]Comenius University in Bratislava; Faculty of Science; Department of Theoretical and Physical chemistry; Slovak Republic

[2]Slovak Technical University; Faculty of Chemical and Food Technology; Department of Inorganic Materials; Slovak Republic

cickan2@uniba.sk

Aluminosilicates are among the hydrated oxide inorganic materials commonly found in nature and can be also prepared synthetically. The structure of 2:1 aluminosilicates is formed by layers build-up by network of octahedrons sandwiched between network of tetrahedrons. Due to the isomorphous substitutions in the networks, excess negative charge is located on the layers. The negative charge is compensated by exchangeable cations between the individual layers, that can be exchanged for other cations in cation exchange reaction. The number of exchangeable cations is referred to as cation-exchange capacity (CEC). Rare earth elements (REE) are known as suitable phosphors used in light-emitting devices. Their ability to emit light after excitation of their valence electrons and subsequent relaxation to the ground levels was intensively studied many years already. REE show their spectral properties due to the numerous possible energy states of f-orbital levels. In presented study, aluminosilicates were used for REE doping, to modify their properties. These materials can be used in several interesting applications, e.g. medicine, photovoltaic panels, etc. In this work, the optical properties of selected aluminosilicates after their REE modification were studied with respect to the changes of their structural and optical properties after their fixation in the interlayer space.

Keywords: Nanoparticles, up-conversion, down-conversion, rare earth elements

Acknowledgments: This work was supported by the Slovak Research and Development Agency under contract No. APVV-18-0075. Support from the VEGA grant agency (1/0227/20) and grant UK/177/2021 is also gratefully acknowledged.

Study of interactions between bioapatite from human bone and Ca – deficient hydroxyapatite

L. Pazourková[1], G. Simha Martynková[2], M. Šupová[3]

[1]IT4 Innovation Centre, VŠB – Technical University of Ostrava, Czech Republic; [2]Nanotechnology Centre, CEET, VŠB – Technical University of Ostrava, Czech Republic; [3]Institute of Rock Structure and Mechanic, Academy of Science of the Czech Republic, Czech Republic

Lenka.pazourkova@vsb.cz

Bioapatites (A), prepared from natural bones, are used as implants for bone grafts or powder bone fillings. The bioapatites are obviously obtained by calcinations of natural bones. The main advantage of BA from natural bones is the right ration between Ca, P and other minerals. The Ca-deficient hydroxyapatite (CDH) is biocompatible and cheap material very often used in biological and non-biological applications. The aim of this study is to prepare CDH particles on surface of bioapatites.

The bioapatites are prepared by two calcinations temperatures (500 and 700°C, respectively) and subsequently crushed in mortar or milled in jett mill. The Ca-deficient hydroxyapatite is prepared on the surface of bioapaties by easy precipitation route to decrease possible influence of more invasive preparation techniques. The resulting composites of A + CDH were characterised by X-ray powder diffraction (XRD), infrared spectroscopy with Fourier transformation (FTIR) and scanning electron microscopy (SEM). The XRD shows broadening of basal reflection of A in composite, which could confirm presence of CDH. The scanning electron microscopy shows creation of small CDH particles and film-like structure. The interactions between CDH and A are probably by physical and non-bonding base.

Keywords: Bioapatite, Bone, Ca-deficient hydroxyapatite

Acknowledgments: This work was supported by the European regional development fund in the IT4Innovations national supercomputing center – path to exascale project, project number CZ.02.1.01/0.0/0.0/16_013/0001791 within the operational programme research, development and education.

Comparison of hydrothermal and microwave synthesis of nano-ZnS and their influence on photocatalytic activity

J. Smijová[1], P. Peikertová[2], K. Mamulová Kutláková[2], J. Tokarský[23]

[1]Faculty of Materials Science and Technology, VŠB – Technical University of Ostrava, Czech Republic; [2]Nanotechnology Centre, CEET, VŠB – Technical University of Ostrava, Czech Republic; [3]Institute of Environmental Technology, CEET, VŠB – Technical University of Ostrava, Czech Republic

julie.smijova@vsb.cz

Zinc sulfide (ZnS) having broad band gap (~3.6 eV) is a semiconductor studied for use in various areas such as photocatalysis, electroluminiscence, biosensing, and others. Size of the band bap can vary with the particle size of the ZnS, and affects the resulting photocatalytic activity – one of the most promising properties of ZnS in terms of practical applications, e.g. wastewater treatment. Aim of the study is to compare structure and photocatalytic activity of nano-ZnS prepared by hydrothermal and microwave synthesis using $ZnCl_2$ and Na_2S as precursors. X-ray powder diffraction and scanning electron microscopy analyses were used in this study to determine the crystallographic phase and morphology of the nano-ZnS prepared by both methods. Samples were also characterized using Fourier-transform infrared spectroscopy and Raman microspectroscopy in order to confirm the ZnS presence as well or to determine the other byproducts. Degradation tests performed under ultraviolet irradiation using model dye Acid Orange 7 revealed that both preparation methods lead to well-acting photocatalytic nanomaterial. These results are a promising step in the search for a time-saving and cost-effective synthesis that has the potential to be used in the large-scale production of nano-ZnS with high photocatalytic activity.

Keywords: ZnS, hydrothermal synthesis, microwave synthesis, photocatalytic activity.

Acknowledgments: This work was supported by the project No. SP2021/106 “Study and development of composite nanomaterials and nanofillers”.

Improving photoactivity of colloidal systems of fluorubine dye and saponite

M. Šuteková[1], J. Bujdák[1,2]

[1]Department of Physical and Theoretical Chemistry, Faculty of Natural Sciences, Comenius University in Bratislava, Slovakia

[2] Institute of Inorganic Chemistry, Slovak Academy of Sciences, Slovakia

lackovicova10@uniba.sk

Fluorubines are highly fluorescent substances belonging to the group of pyrazinacene dyes. Hybrid colloidal systems based on a fluorubine derivative (BF) and layered silicate saponite (Sap) were the subject of this study. Compared to dye solutions in organic solvents, aqueous solutions showed significantly lower photoactivity, probably due to the formation of inactive molecular aggregates. The main objectives of this work were to find conditions for the preparation of hybrid systems to achieve an optimal photoactivity of the dye. UV-VIS absorption and fluorescence spectroscopy methods detected the presence of molecular aggregates also in BF/Sap hybrid colloids. By increasing the temperature of the hybrid systems, photoactive spectral substances like those in organic solvents were formed. By heating, the dye aggregates are likely to break down to form monomers, which are stabilized by adsorption on the surface of the layered particles, thereby improving the BF photoactive properties and the active form of the dye remained stable after re-cooling the systems. It is known that dye molecular aggregates are not formed in organic solvents and it is possible to achieve higher photoactivity. For practical applications, the use of organic solvents is not the optimal solution. Therefore, BF/Sap systems are very promising luminescent hybrid materials.

Keywords: layered silicates, photoactivity, fluorubine dye, luminescence.

Acknowledgments: This work was supported by the Slovak Research and Development Agency under contract No. APVV-18-0075. Support from the VEGA grant agency (1/0227/20) is also gratefully acknowledged.

Laser and discharge plasma assisted synthesis of cobalt doped zinc oxide nanostructures in liquids

N. Tarasenka, A. Nevar, M. Nedelko, V. Kornev, N. Tarasenko

B. I. Stepanov Institute of Physics, National Academy of Sciences of Belarus, Minsk, Belarus

n.tarasenko@ifanbel.bas-net.by

The processes of synthesis of doped (ZnO:Co) nanocrystals in electrical discharge and laser ablation plasmas in liquid with additional laser irradiation of suspensions have been studied. The combination of these non-equilibrium processes has been demonstrated as efficient tool for the synthesis of doped ZnO:Co NPs. The results of the characterization of inner structure, phase composition and morphology performed by means of TEM SAED, Raman, XPS and XRD techniques proved the formation of doped ZnO:Co nanocrystals under the found optimized experimental conditions.

Keywords: *laser ablation in liquid, electrical discharge in liquid, doped ZnO nanoparticles*

Acknowledgments: The work was partially financed by the National Academy of Sciences of Belarus under project Convergence 2.2.05.

Surface properties of smectites fine fractions as precursors of multifunctional composites

V. Valovičová[1], S. Dolinská[2], L. Vaculíková[1], E. Plevová[1], I. Znamenáčková[2], Z. Danková[3], D. Klichová[1], H. Yankovych[2]

[1]Institute of Geonics of the Czech Academy of Sciences, The Czech Republic; [2]Institute of Geotechnics of the Slovak Academy of Sciences, Slovak Republic; [3] State Geological Institute of Dionyz Stur, The Slovak Republic

vera.valovicova@ugn.cas.cz

The study focuses on evaluating surface properties of raw and activated smectites fine fractions in relation to their potentialities as precursors for multifunctional composites. Mineral standard samples of four montmorillonites as representatives of the smectite mineral group were purified and chemically activated using Na_2CO_3. Both the starting materials and the activated samples were characterized by X-ray diffraction analysis (XRD), changes in the surface and pore properties were studied by low nitrogen adsorption method and surface morphology by scanning electron microscope (SEM). On the basis of obtained results it can be concluded that activated materials exhibited changes of structural and surface properties. The modification mainly affected meso-macroporous montmorillonite samples. In these cases, the prepared samples are potentially suited to be precursors of multifunctional sorbents with enhanced sorption properties.

Keywords: smectite, activation, characterization, composites

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Preparation of polylactide nanoparticles by supercritical fluid technology

J. Zágora[1], D. Plachá[1], M. Fernández García[2], A. Muñoz Bonilla[2]

[1]Nanotechnology Centre, CEET, VSB – Technical University of Ostrava, Czech Republic; [2]ICTP, CSIC, Spain

jakub.zagora@vsb.cz

Development of new antimicrobial materials that are not toxic to higher living organisms is a major challenge today. Newly developed materials can have a high application potential in biomedicine, coatings, packaging etc. Combination of commonly used biopolymer polylactide with cationic polymers seems to be very successful in the fight against antimicrobial resistance [1]. PLA will play a key role in fulfilling the intention set out in the New Deal announced by the EU commission, as it is a bioplastic that is easily degradable, recyclable, and mass-produced. Also, the development of 3D printing in the context of this initiative, and the actual use of PLA as one of the main materials used for this printing, make the technology around the preparation and modification of PLA quite logical.

Moreover, the environmentally friendly and energy saving technology like supercritical fluid process (SFP) will be used for their preparation. In a first approach, polylactide nano- and microparticles and structures were prepared by supercritical fluid extraction. The RESS (rapid expansion supercritical fluid solution) method is easier to optimize and shows better particle size control. On the contrary, a highly porous structure was obtained using the SAS (supercritical antisolvent) method. In a second part the antimicrobial biobased polymer will be introduced by SFP.

Keywords: polylactide, antimicrobial polymers, supercritical fluid technology, micronization,

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TOPIC 2

Nanomaterials Characterizations and Devices

Chair: Jan Neuman

Advanced techniques for characterization in nanotechnology
Advanced testing methods for nanomaterials
Molecular modeling and simulations
Advances in computational methods for nanotechnology

Invited lectures (IL):

Infrared spectroscopy as a powerful tool to study clay minerals subjected to various types of modification

H. Pálková[1], J. Madejová[1]

[1]Institute of Inorganic Chemistry, Slovak Academy of Sciences, Dúbravská cesta 9, 845 36 Bratislava, Slovak Republic

helena.palkova@savba.sk

Clay minerals are abundant, inexpensive and environmentally friendly materials with a huge potential for diverse industrial and environmental applications. The preparation of innovative functional materials based on these layered materials persistently attracts the attention of researchers. Clay minerals are frequently subjected to various types of modifications in order to improve and change their structural and surface properties. Essential step to follow changes occurring upon preparation of such materials is detailed characterization using various experimental methods. Infrared (IR) spectroscopy have been commonly applied to clay minerals research as very effective in solving tasks connected not only to the characterization, but also for the identification of the interactions occurring in newly prepared systems.

The present contribution will be predominantly focused on the application of various IR techniques to study smectites, important group of clay minerals, frequently subjected to the mechanical, thermal or chemical handling. Further, the attention will be given to the effects of different aspects of the interactions of clay minerals with organic species. Mid infrared spectroscopy (MIR), 4000–400 cm^{-1} spectral region, is a useful and widely available tool for the characterization of modified smectites. In addition, the utilization of near IR (NIR) spectroscopy (8000–4000 cm^{-1}) will be discussed to show its benefits in evaluation of materials characteristics.

Keywords: Clay minerals, IR spectroscopy, modifications, organic surfactants, grinding, intercalation, acid treatment, thermal treatment.

Stranski–Krastanov and Volmer–Weber CVD Growth Regimes To Control the Stacking Order in Bilayer Graphene

Huy Q. Ta

Leibniz Institute for Solid State and Materials Research Dresden

P.O. Box 270116, D-01171 Dresden, Germany

q.h.ta@ifw-dresden.de

Aside from unusual properties of monolayer graphene, bilayer has been shown to have even more interesting physics, in particular allowing bandgap opening with dual gating for proper interlayer symmetry. Such properties, promising for device applications, ignited significant interest in understanding and controlling the growth of bilayer graphene. Here we systematically investigate a broad set of flow rates and relative gas ratio of CH_4 to H_2 in atmospheric pressure chemical vapor deposition of multilayered graphene. Two very different growth windows are identified. For relatively high CH_4 to H_2 ratios, graphene growth is relatively rapid with an initial first full layer forming in seconds upon which new graphene flakes nucleate then grow on top of the first layer. The stacking of these flakes versus the initial graphene layer is mostly turbostratic. This growth mode can be likened to Stranski–Krastanov growth. With relatively low CH_4 to H_2 ratios, growth rates are reduced due to a lower carbon supply rate. In addition, bi-, tri-, and few-layer flakes form directly over the Cu substrate as individual islands. Etching studies show that in this growth mode subsequent layers form beneath the first layer presumably through carbon radical intercalation. This growth mode is similar to that found with Volmer–Weber growth and was shown to produce highly oriented AB-stacked materials. These systematic studies provide new insight into bilayer graphene formation and define the synthetic range where gapped bilayer graphene can be reliably produced.

Keywords: Bilayer graphene, stacking order, stacking control, hydrogen role, growth mechanism.

Oral presentations (OP):

Silver Nanoparticles Stabilized with Organic Acid

Z. Aslan[1], G. Kratošová[2], G.S. Martynková[2]

[1]Engineering Faculty, Department of Metallurgical & Materials Engineering, Sakarya University, Turkey;
[2]Nanotechnology Centre, CEET, VŠB-TU Ostrava, Ostrava, Czech Republic

zeynep.aslan4@ogr.sakarya.edu.tr

In the biomaterials area, silver nanoparticles are commonly used owing to their antimicrobial properties. One of the most attractive approaches to AgNPs synthesis is a green synthesis. In the present study, silver nanoparticles were successfully created using biomass of *Urticia Herba* (UR) and *Arthemisia absinthium* (AR) with silver precursor $AgNO_3$. These plants have been selected for phytosynthesis due to chemical composition of medical constituents and non-toxicity. Moreover, formic acid was used as a stabilizing agent in experiments. Different concentration of precursors was tested to observe the speed of reduction via the color change. The fastest reduction observed was for UR, taken approximately 5 minutes. Furthermore, particle size and morphology of nanosilver were analyzed using the transmission electron microscope and scanning electron microscope whereas characterizations which are phase transformations and structure were performed using XRD and FT-IR methods. The method was effective, eco-friendly, and reproducible in synthesizing nanosilver. The long-term goal is to prepare bio nanoparticles in an inexpensive and clean way.

Keywords: Nano silver, biosynthesis, reduction, formic acid

Gas-phase deposition of noble metal coatings for medical applications

S.I. Dorovskikh[1], E.S. Vikulova[1], N.B. Morozova[1]

[1]Nikolaev Institute of Inorganic chemistry, Novosibirsk, Russia

dorov@niic.nsc.ru

In this work, we demonstrate the potential of application of a Metal-Organic Chemical Vapor Deposition (MOCVD) technique for deposition of Pt, Ir and $Pt_xIr_{(1-x)}$ films on polyimide substrates potential materials of modern medical implants. It is expected that such chemical surface modification of polymers opens perspectives to design implants with improving ossteointegration, X-ray visualization in to human bodies as well as long-term anti-inflammatory effect. The heteroligand $Ir(CO)_2(acac)$ and $(CH_3)_3Pt(acac)(Py)$ (acac – acethylacetonato, Py – pyridine) complexes are used precursors in MOCVD processes. The low-temperature MOCVD processes of formation of noble metal layers on polyimide were developed. The composition, structure and morphology of Pt, Ir and $Pt_xIr_{(1-x)}$ films deposited on polyimide and Si(100) substrates were studied.

Keywords: noble metal, polyimide, MOCVD, medical implants

Acknowledgments: This work was funded by the Russian Science Foundation, project No. 20-15-00222.

Deposition, laser lithography and wet etching of silicon nitride thin films

Z. Gelnarova[1], L. Halagacka[1,2], T. Kohut[1,2], T. Fördös[1,2], D. Cvejn[4], and K. Postava[2,3]

[1]Nanotechnology Centre, CEET, VŠB – Technical University of Ostrava, Czech Republic; [2]IT4Innovations, VŠB – Technical University of Ostrava, Czech Republic; [3]Faculty of Materials Science and Technology, VŠB – Technical University of Ostrava, Czech Republic; [4]ENET Centre, CEET, VŠB – Technical University of Ostrava, Czech Republic

zuzana.gelnarova@vsb.cz

Our aim is to develop a structured dielectric layer based on silicon nitride for electrical insulation of the Auston switch from spintronic multilayer stack, and for high-contrast gratings in multilayer lasers. Reactive RF magnetron sputtering from an intrinsic silicon target in the argon/nitrogen environment is used for deposition of silicon nitride thin films. The deposition parameters of silicon nitride are optimized in order to develop dielectric material with good insulation properties. After deposition, the UV laser optical lithography with direct writer is used to pattern the positive photoresist which acts as a mask for wet chemical etching. The final design is accomplished by the wet chemical etching of the silicon nitride layer in hydrofluoric acid. Various deposition, lithographic, and etching conditions are studied to establish deposition and etching rates and to find optimal conditions for fabrication of desired structure.

Keywords: Silicon nitride, reactive RF magnetron sputtering, UV laser optical lithography, wet chemical etching.

Development of materials and 1D photonic crystals by reactive magnetron sputtering

J. Horníček[1,3], L. Halagačka[1,2], K. Postava[2,3]

[1]Nanotechnology Centre, CEET, VŠB – Technical University of Ostrava, Czech Republic; [2]IT4Innovations, VŠB – Technical University of Ostrava, Czech Republic;
[3]Faculty of Materials Science and Technology, VŠB – Technical University of Ostrava, Czech Republic

jiri.hornicek@vsb.cz

We designed and developed a 1D photonic crystal based on stack of SiO_2 and Ta_2O_5 thin alternating layers. The crystal is designed to work as an ideal mirror at wavelength of 800 nm and at the 45° angle of incidence. The radiofrequency (RF) reactive magnetron sputtering was used for thin film deposition. The Ar/O_2 environment was used to deposit Ta_2O_5 and SiO_2 from Ta and SiO_2 targets, respectively. Deposition parameters (power, pressure, gas compositions) were optimized to achieve materials with negligible optical absorption at required operational wavelength. For the design, the knowledge of optical functions of used materials is essential. The optical properties of developed materials were characterized by Mueller matrix spectroscopic ellipsometry (SE). The sample was measured by SE after each SiO_2/Ta_2O_5 bilayer deposition. This allows us to monitor the deposition process and to adjust deposition parameters.

Keywords: sputtering, photonic crystal, ellipsometry, thin film

Micromechanical characterization of Carbon Black reinforced epoxy nanocomposite using micro indentation

S. Khammassi, M. Tarfaoui

ENSTA Bretagne, FRE CNRS 3744, IRDL, F-29200 Brest, France

sabrine.khammassi@ensta-bretagne.org

The effect of carbon black reinforcement on the Vickers hardness of epoxy resin-based composites was studied. The micro-indentation dynamic mechanical analysis test measures the micromechanical properties of carbon black reinforced DGEBA epoxy nanocomposites with varying weight percentages (0, 1, 2 and 5 wt%). The micro-indentation deformation was studied by in-situ imaging of the impression using KEYENCE. Mechanical properties, such as hardness, elastic modulus, and stiffness, gradually increase as the mass fraction of filler (CB) continuously increases. This progress depends on various parameters, such as nanoparticle-reinforced polymer composites' dispersion, interfacial bonding, and load transfer.

Keywords: Carbon Black, nanocomposite, micro-indentation, micro-hardness

Acknowledgments: The Region Bretagne, through the PhD projects, collaborated with ENSTA Bretagne kindly supported this work.

Setting of exposure parameters for direct laser writing in optical lithography

T. Kohut,[1,2] J. Toběrný,[1] and K. Postava[2,3]

[1]Nanotechnology Centre, CEET, VŠB – Technical University of Ostrava, Czech Republic; [2]IT4Innovations, VŠB – Technical University of Ostrava, Czech Republic;

[3]Faculty of Materials Science and Technology, VŠB – Technical University of Ostrava, Czech Republic.

tomas.kohut@vsb.cz

For correct preparation of lithographic nanostructures, precise knowledge of photoresist properties, parameters of exposure, and photoresist development time are essential. Our study is based on Gaussian beam properties of laser diode with operational wavelength of 405 nm. In the study we use the positive photoresist ma-P 1200 series. Main writing parameters of PicoMaster100 lithograph are the spot size, the power dose, the step between writing lines, and the vertical offset of the laser. Dependence of the developed photoresist thickness on the laser power and the development time were investigated by analysis of optical data measured by spectroscopic ellipsometry. Effects of the laser offset and the step size were studied on the developed 1D nanograting with atomic force microscopy. Knowledge of these dependencies enable us to reach the lithography writing limits and prepare photomask and high-quality security holographic structures with new forensic effects.

Keywords: Positive photoresist, UV DWL lithography, ellipsometry, diffractive grating

Structural and phase variability study of the PVDF nanocomposite materials

L. Plesník[1], K. Čech Barabaszová[1], S. Holešová[1]

[1]Nanotechnology Centre, CEET, VŠB - Technical University of Ostrava, 17. listopadu 15/2172, 708 00 Ostrava - Poruba, Czech Republic

lukas.plesnik.st@vsb.cz

The PVDF nanocomposite materials were prepared in the form of thin films. The natural PVDF were modified by various inorganic and organic nanofiller materials with known antimicrobial properties. The structural changes were investigated using scanning electron microscope (SEM). The hydrophilic character was demonstrated by water contact angle and the phase changes were evaluated by the Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction analyses. The PVDF nanocomposite samples had spherulites structure with a prevailing α and γ phases.

Keywords: PVDF nanocomposites, structural and phase changes, crystallite

Acknowledgments: This work was supported by the project No. SP2020/08 "Hybrid clay nanofillers for antimicrobial polymer films" and project No. SP2021/106 "Study and development of composite nanomaterials and nanofillers". Authors thank to M. Heliová for SEM micrographs.

TiO_2-SnO_2 films: synthesis by low-temperature pyrolysis and electrophysical properties

M.G. Volkova[1], V.Yu. Storozhenko[1], I.A. Gulyaeva[2], A.P. Starnikova[2], V.V. Petrov[2], E.M. Bayan[1]

[1]Department of Chemistry, Southern Federal University, Russia; [2]Research and Education Centre "Microsystem technics and multisensor monitoring systems", Southern Federal University, Russia

wolkowa-mg@yandex.ru

Thin films of pure SnO_2 and TiO_2-SnO_2 materials with a content of Ti:Sn=5:95 and Ti:Sn=50:50 mol.% were obtained by solid-phase low-temperature pyrolysis technique. According to XRD and SEM it was shown that regardless of the modified agents concentration, all materials were nanoscale and the resulting surface was homogeneous. The study of the temperature resistance dependence showed that the introduction of small titanium concentrations leads to a slight decrease in the films resistance; when the Ti/Sn amount is equal, the resistance changes significantly and doesn't depend on the temperature.

Keywords: Nanomaterials, films, pyrolysis, tin dioxide.

Acknowledgments: This work was financially supported by the RFBR, project 20-07-00653 A.

Poster presentations (PP):

The photochemical reactions of 4-aminobenzenethiol on the plasmonic nanostructured surfaces

I. Kopal[1], M. Švecová[2], T. Čapková[3], M. Dendisová[1]

[1]Department of Physical Chemistry, University of Chemistry and Technology Prague, Czech Republic;
[2]Department of Analytical Chemistry, University of Chemistry and Technology Prague, Czech Republic;
[3]Centre of Polymer Systems, University Institute, Tomas Bata University in Zlín, Czech Republic

kopali@vscht.cz

The increasingly dynamic development in the field of Surface-enhanced spectroscopies leads to the preparation of innovative nano-featured enhancing substrates, tested using suitable model analytes. 4-aminobenzenethiol (4-ABT) is widely used for this purpose, mainly due to its optical properties and adsorption abilities onto the surface of plasmonic substrates. On the other hand, there is not negligible risk of the photochemical dimerization of 4-ABT as a result of the incident radiation under certain circumstances. Therefore, it is desirable to assess the extent of dimerization on the selected plasmonic metals, same as the role of the excitation wavelength used.

As part of this work, we studied the photochemical reaction of prepared large-scaled substrates covered by 4-ABT molecules. The morphology of these substrates was monitored using optical and electron microscopy. Furthermore, we have measured the Surface-enhanced Raman scattering (SERS) spectra of the deposited molecules on the prepared Ag, Au, and Cu surfaces using five different excitation wavelengths.

The results showed that the formation of dimer molecules occurs most abundantly on the silver substrate, disproportionately less on the Au and Cu surfaces. In all cases, the degree of dimerization is strongly dependent on the morphology of the nanostructures and the excitation wavelength used.

Local anodic oxidation of silicon for crossbar architecture

V. Polyakova[1], A. Rezvan[2]

[1] Design Center for Microelectronic Component Base for Artificial Intelligence Systems, [2] Institute of Nanotechnologies, Electronics and Equipment Engineering, Southern Federal University, Russia

vpolyakova@sfedu.ru

That article presents the possibility of forming a neuromatrix using the crossbar architecture technology on silicon substrates, for the implementation of which it is necessary to study the technological parameters of the local anodic oxidation of silicon. The possibility of using the method of local anodic oxidation of silicon for the implementation of this neuromatrix is show.

Keywords: Silicon, localized anodic oxidation, neuromatrix, crossbar.

Acknowledgments: Scientific research was carried out within the framework of the project "Development and research of methods and tools for monitoring, diagnostics and forecasting of the state of engineering objects based on artificial intelligence" (task No. FENW-2020-0022, work number at SFU No. LAB0110 / 2020-

01ITs). We would like to express our gratitude to the center for collective use of nanotechnologies of the southern federal university.

MOCVD growth and study of hybrid heterostructures based on Pd particles and metal phthalocyanines

D.E. Tryakhov[1,2], S.I. Dorovskikh[1,2], D.D. Klyamer[2], T.V. Basova[1,2]

[1]Novosibirsk state University, Novosibirsk, Russia

[2]Nikolaev Institute of Inorganic chemistry, Novosibirsk, Russia

reter16@yandex.ru

We present the results on the fabrication of new hybrid film materials based on Pd particles deposited on cobalt phthalocyanine (CoPc) films using a pulse Metal-Organic Chemical Vapor Deposition (pulse-MOCVD) technique. The use of hybrid materials opens perspectives to design a multifunctional membrane to produce 99.999% hydrogen. To fabricate Pd particles, the series of high volatile fluorinated Pd beta-diketonates were synthesized and thoroughly examined by several physic-chemical methods and $Pd(hfac)_2$ (hfac - 1,1,1,5,5,5-hexafluoro-2,4-pentanedione) was used as a precursor. The composition and morphology of Pd particles deposited on Si(100) substrates are studied using XPS and microscopy methods. The influence of the deposition parameters on the morphology of Pd particles is also discussed.

Keywords: Pd beta-diketonates, MOCVD, Pd particles

The optical and electrophysical properties of Al-ZnO thin films

I.O. Ignatieva[1], M.G. Volkova[1], I.A. Gulyaeva[2], A.P. Starnikova[2], V.V. Petrov[2], E.M. Bayan[1]

[1]Department of Chemistry, Southern Federal University, Russia; [2]Research and Education Centre “Microsystem technics and multisensory monitoring systems”, Southern Federal University, Russia

wolkowa-mg@yandex.ru

Nanocrystalline films of pure ZnO and zinc oxide with a molar ratio Al:Zn=1:99 and 3:97 were obtained by solid-phase pyrolysis. The XRD method established that the prepared films have a wurtzite structure. The particle sizes and the crystallinity of the obtained materials were determined. Based on the study of the optical properties, it was found that the obtained films are almost transparent in the near UV range (the absorption coefficient is less than 16 %) and are transparent in the visible wavelength range. It is shown that the lowest resistance and activation energy of electrical conductivity is observed in the film with a molar ratio Al:Zn=1:99.

Keywords: Thin films; zinc oxide; aluminum oxide; optical properties.

Acknowledgments: This work was financially supported by the RFBR, project 20-07-00653 A. The authors are grateful to the Molecular Spectroscopy Centre of Southern Federal University for the registration of spectra.

Controlled deposition of polymer brush films to develop sub 5 nm zirconium oxide films liquid phase ion inclusion

P. Yadav [a], Riley Gatensby [a], M. Snelgrove [c], M. A. Morris [a]

a School of Chemistry, AMBER@CRANN, Trinity College Dublin, Dublin, Ireland

b Institute of Chemistry of Condensed Matter of Bordeaux, University of Bordeaux

c Dept. of Physics, Dublin City University, Dublin, Ireland

pyadav@tcd.ie

In recent years, down scaling of integrated circuit (ICs) and the powering up of electronic devices have created integration challenges. To meet the fabrication challenges of next generation electronic devices, novel materials and new processing methods are being discovered. In particular, area selective deposition (ASD) techniques have gained much interest as a way to produce direct deposition of the active materials while reducing processing step(1). In order to generate uniform metal oxide films it is critical to form a templating system that can enable the infiltration of metal precursors. We have demonstrated grafting of end functionalised polystyrene, polymethylmethacrylate, and poly-2(vinyl pyridine) as templates to produce highly coherent oxide films. A facile approach for rapid grafting (in seconds) of polymer brush films with complete coverage over large area with high uniformity is shown (pinhole free)(2). We developed monolayer coverage of polymer brush through careful optimization of polymer molecular weight, concentration, annealing temperature and time. Formed polymer brush film was then infiltrated with ethanolic solution of zirconium nitrate and subsequent oxygen plasma treatment eliminate polymer films and yield coherent zirconium oxide films.

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TOPIC 3

Nanotech for Energy

Chair: Ahmet Oguz Tezel

Nanomaterials for energy storage
Nanotechnology for renewable energy harvesting and conversion
Hydrogen storage
Sustainability of materials recycling for energy

Invited lecture (IL):

State of the art nanostructured cathode materials for lithium-oxygen batteries

Tugrul Cetinkaya[1,2,3]

[1]Sakarya University, Engineering Faculty, Department of Metallurgical & Materials Engineering, Esentepe Campus, 54187 Sakarya, Turkey

[2]Sakarya University, Research, Development and Application Center, Esentepe Campus, 54187 Sakarya, Turkey

[3]NESSTEC Energy & Surface Technology A.S., Technology Development Zones, 54050, Sakarya University, Sakarya, Turkey

tcetinkaya@sakarya.edu.tr

Lithium oxygen batteries (Li-O_2) are seen as next-generation energy storage systems among rechargeable lithium-based batteries. According to the European battery road map, it is reported as 5th generation batteries and expecting to commercialize after 2030. Although the number of publications has been decreased in 2018 and 2019 regarding the Li-O_2 batteries, recently, it has exhibited a dramatic increment. Recent works show that the development of nanostructured cathode materials will overcome one of the biggest issues of such batteries and will make them possible to be available in the market future.

Li-O_2 batteries consist of lithium metal anode, electrolyte, separator and porous cathode components and each component of batteries has been studying with researchers in whole of the world. Since the Li-O_2 formation and decomposition reaction takes place in the cathode, it is responsible for the reversibility, cycle life, efficiency, and C-rate. Therefore, the main challenge is to development of the optimal cathode for Li-O_2 batteries. In the literature researchers develop a metal, metal oxide (sulfide) cathodes with different nanostructured morphology, functionalized graphene-based cathodes with such metal, metal oxide (sulfide), heterostructure with other 2D materials and carbon free nanostructured cathode materials. The main target for the development of the cathode materials is to prevent side reactions, increase oxygen catalysis and provide reversible formation-decomposition reaction kinetics. In this talk, state-of-the-art nanostructured cathode materials for Li-O2 batteries will be reviewed, discussed in detail with future perspective.

Keywords: Li-O_2, graphene, 2D materials, metal oxide, metal sulfide, nanostructure, cathode, review, discussion, future perspective.

Oral presentations (OP):

Two dimensional organic materials for energy and the environment

A. Bakandritsos[1,2], J. Kolarik[1], I. Obraztsov[1], M. Otyepka[1], R. Zbořil[1,2]

[1]Regional Centre of Advanced Technologies and Materials, CATRIN, Palacký University, Olomouc, Czech Republic; [2]Nanotechnology Centre, Centre of Energy and Environmental Technologies, VŠB–Technical University of Ostrava, Czech Republic

a.bakandritsos@upol.cz

Chemical functionalization of graphene can mitigate its tendency for restacking and boost its ability to interact with target chemical species. Fucntionalization must be performed selectivly and covalently if high performance is required, combined with material-reusability and stability in challenging environents. For example, such properties are pre-requisites for sustainalbe and reusable sorbents for heavy metal recovery and in electrode materials for energy storage.

These challenges are together met in a densely carboxylated and conducting graphene derivative (graphene acid, GA). GA is prepared through fluorographene chemistry,[1] and currently represents the most conductive graphene derivative with such a high content of carboxyl groups (Figure 1). Carboxyls are strong metal-coordinating groups,[2] predisposing GA for capturing metal ions in water remediation applications and for lithium coordination in energy storage applications.[3] GA exhibited high proclivity for sorption of Cd^{2+} and Pb^{2+}, as well as for the valorization of waste Pt for developing electrocatalysts for the oxygen reduction reaction. The all-covalent and direct $C_{graphene}$-COOH bonds rendered GA particularly stable in highly acidic media, allowing for metal recovery and full regeneration of the sorbent.[4] As Li-ion battery anode, GA revealed i) high redox capacity stemming from its carboxyl groups, ii) high conductivity, boosting the rate capability, iii) high capacity, and iv) extra charge storage due to the co-presence of significant content in sp^2 moieties serving as Li intercalation sites.

The results indicate that the covalent, dense and selective grafting of carboxyl groups on the conductive skeleton of graphene paves the way for efficient and stable organic anodes for energy storage and environmental applications.

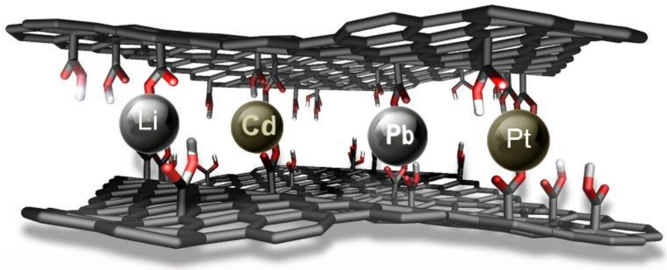

Fig. 1. Structural model of the densely and selectively functionalized graphene with carboxyl groups, offering robust binding sites for metal ions, finding application in water remediation and energy storage.

Acknowledgments: The work was supported by the ERDF/ESF project "Nano4Future" (No. CZ.02.1.01/0.0/0.0/16_019/0000754) and by GACR (project No 19-27454X).

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Development of lithiated ion exchange membranes for Li-S batteries

Ş. Dombaycıoğlu[1,2], H. Günsel[2,3], A.O. Aydın[1,2]

[1]Department of Chemistry/Arts&Sciences Faculty, Sakarya University, Turkey;

[2] Sakarya University Research, Development and Application Center (SARGEM), Sakarya, Turkey;

[3]Department of Fundamental Science in Engineering/Faculty of Technology, Sakarya University of Applied Sciences, Sakarya, Turkey

sdombaycioglu@sakarya.edu.tr

Lithium sulfur (Li-S) batteries attract attention due to their high theoretical energy density of 2600 Whkg^{-1}, which is 5 times more than the latest technology Li-ion batteries [1]. However, despite all the benefits, this electrochemical system have some disadvantages that cause capacity fade, such as dissolution of the polysulfides in the liquid electrolyte and their shuttle effect [2]. Recently, the use of ion exchange membranes in order to ensure capacity retention in Li-S batteries has been the subject of research [3,4].

In this study, lithiated Nafion and Aquivion based hybrid ion-exchange membranes were developed. First of all, Nafion and Aquivion ionomers in solution form were lithiated and produced in powder form. By using these powders, Li-Naf, Li-Aqu and by mixing these ionomers in certain proportions Li-Naf: Li-Aqu hybrid membranes were prepared.

The ion exchange capacity and polysulfide transition of the produced membranes were examined and structural characterizations were carried out via fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric analysis (TGA) and field emission scanning electron microscopy (FESEM). In order to perform electrochemical analysis, CR2032 half cells were designed. Electrochemical performances of membranes were examined by cyclic voltammetry, electrochemical impedance spectroscopy and galvanostatic charge-discharge tests.

Keywords: Nafion, Aquivion, ion exchange membrane, Li-S batteries.

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Photoelectrochemical water splitting of core/sheath nanowires based on ZnO and transition metal dichalcogenides

K. Govatsi[1], A. Antonelou[1], S. Neophytides[1] and S. N. Yannopoulos[1]

[1]Foundation for Research and Technology Hellas – Institute of Chemical Engineering Sciences
(FORTH/ICE-HT), P.O. Box 1414, GR-26504, Rio-Patras, Greece

kat.govatsi@iceht.forth.gr

The quick depletion of natural resources and moderation of carbon dioxide emissions are currently pursued by developing low-cost alternatives of electricity supply using renewable energy sources, such as solar light. Photoelectrochemical (PEC) water splitting stands out as a promising way towards hydrogen production by solar energy-driven water splitting. Metal oxide semiconductors such as TiO_2 and ZnO have traditionally employed as anodes in PEC cells in view of their stability and ease of morphology manipulation, especially for ZnO. Though, the wide optical bandgaps of these materials limit their light absorption only to the UV part of the solar spectrum, which prevents the efficient exploitation of the solar energy in the visible region. Hybrid nanostructures, for example core/sheath nanorods consisting of a metal oxide semiconductor core (ZnO) covered by transition metal dichalcogenides layers (MX_2, M: Mo, W, etc., and X: Se, S_2, Te) and other chalcogenides such as ZnSe have been explored to prepare visible light stimulated PEC anode materials. In this work we prepare various hybrid systems where ZnO nanorods (core) are covered by chalcogen-based thin films (sheath). ZnO are prepared by a chemical bath deposition method, while chalcogenide crystals are prepared by soft chalcogenation at moderate temperatures of pre-deposited metallic film on the ZnO surface. Structural and morphological characterization is followed by exploration of the influence of the type of chalcogenide crystal and the thickness of the pre-deposited metal on the PEC properties of these hybrid nanostructures.

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Study of the vertically oriented domain structure of nanosized PZT films

I.A. Gulyaeva[1], D.V. Stryukov[3], A.V. Nesterenko[2], A.V. Pavlenko[3], M.V. Il'ina[2],
O.A. Ageev[4], V.V. Petrov[1]

1,2Research and Education Centre “Microsystem technics and multisensory monitoring systems”, Southern Federal University, Russia; 2 Institute of Nanotechnologies, Electronics and Electronic Equipment Engineering, Southern Federal University, Russia 3Southern Scientific Centre, Russian Academy of Sciences, Russia; 2,4Research and Education Center “Nanotechnologies”, Southern Federal University, Russia;

iten@sfedu.ru

In this work, we investigated films of lead zirconate-titanate (PZT) formed by the method of high-frequency reactive plasma sputtering in oxygen. The films have a thickness of 610-660 nm and a developed relief with a predominant number of pointed "protrusions" 100-300 nm in height. It was shown by X-ray phase analysis and force microscopy of the piezoresponse that PZT crystallites are located on the surface of the protrusions. The magnitude of the piezoresponse is 273 ± 38 pm / V.

Keywords: Films of lead zirconate-titanate

Acknowledgments: The work related to the formation of PZT films was supported by the Russian Foundation for Basic Research (Grant No. 18-29-11019). The measurements were supported by the design part of the state assignment of the Ministry of Education and Science of Russia No. 2.3928.2017 / 4.6 at the Southern Federal University and REC "Nanotechnology" SFedU.

Hybrid nanostructures for (photo)electrochemical water splitting

Štěpán Kment

Nanotechnology Centre, CEET, VSB-Technical University of Ostrava, Czech Republic

stepan.kment@vsb.cz

The production of energy through solar light harvesting and conversion is considered among the most promising options for tackling the growing need for energy on global scale and environmental concerns caused by the huge dependence of the energy demand on fossil fuels. In spite of the social relevance of these energy-related issues, finding valuable alternatives to the use of oil-based energy sources remains highly challenging and thus sustainable technologies are still far from commercial and industrial applications.

Semiconductor metal oxides (*e.g.,* TiO_2, α-Fe_2O_3, $BiVO_4$) are the most widely adopted materials for the conversion of solar energy into transportable chemical energy such as di-hydrogen (H_2) produced *via* the photocatalytic and photo-electrochemical (PEC) splitting of water. Nowadays, the scientific research is mainly focused on the development of effective strategies that could couple (i) light absorption, (ii) steady-state production and transfer of photogenerated charge carriers, and (iii) solar water splitting reaction into one single device, and on the engineering of composite assemblies that could maximize the efficiency of the single processes. Unfortunately, none of the previously mentioned metal oxides simultaneously meet the thermodynamic and kinetic requirements that would ensure a high performance in the mutually interconnected (i) – (iii) processes.

This presentation will deal with the design and realization of hybrid nanostructuress, in which selected components are arranged to leverage their expected mechanistic functions. In particular we aim at covering the lack of efficient multifunctional PEC materials and devices by: i) controlling dimensionality (*i.e.*, 1D - 3D) for improved charge transport directionality; ii) engineering hybrid nanostructures (HNS), that could provide enhanced water splitting efficiency through the synergistic combination of the single semiconductor counterparts' properties; iii) selecting novel co-catalysts/sensitizers, to boost charge transfer processes and to extend light absorption properties.

Study of clay mineral composites for Lithium batteries

S.K. Sathish[1], G. Simha Martynkova[1]

[1]Nanotechnology Centre, CEET, VSB-Technical University of Ostrava, Czech Republic

sajjan56@gmail.com

The work deals with the laboratory preparation and characterization of clay mineral composites, which are prepared using the clays (montmorillonite and vermiculite), polymers (polyethylene oxide and polyvinyl difluoride), and with or without the addition of conductive carbonaceous material (graphene oxide). The polymer/ clay composites have applications in Li-batteries as solid state electrolytes. The properties of the samples were dependent on heat treatment, the chosen combination of different clays, polymers and carbon source, and its intercalation. The prepared powder samples were then characterized using X-Ray diffraction for structural and phase changes, Fourier transform-infrared spectroscopy for bonding interactions, Inductively coupled plasma atomic emission spectroscopy for elemental analysis, Differential scanning calorimetry for thermal properties of prepared samples, and measurement of electrical conductivity. The aim of the work was to analyse the prepared clay/polymer nanocomposites which could be employed as potential solid electrolytes in batteries.

Keywords: Clay minerals, polymers, intercalation, nanocomposite, lithium.

Nanocarbons in composites for Li-S batteries

G. Simha Martynkova[1]

[1]Nanotechnology Centre, CEET, VSB-Technical University of Ostrava, Czech Republic;

Grazyna.simha@vsb.com

The nanocarbons are playing in recent nanocomposite science unoverlooked role in various areas of science and industry. Their unique properties are giving the conventional matrix new perspectives and applicability in nontraditional areas. The application in energy saving materials is expected to be advantageous due to their conductive properties. Various allotropes of carbons will provide slightly different results based on the morphology.

Planar graphene is promising host for wrapping or anchoring sulfur to fabricate carbon/ sulfur composite cathodes for LSBs. It can build macroscopic architectures like porous carbons which serves as a perfect building block for preparing binder-free graphene-sulfur composite cathodes. On the other hand, nanotubes or nanofibers when aligned can provide hierarchical porous structure can significantly increase the specific surface areas and total pore volume of materials.

Comparison of both allotropes with traditional carbon black soot was evaluated regarding the sulfur morphology after mechanical treatment. The particles of ductile orthorhombic sulfur became variously shaped while milled with different carbons. The phenomenon of carbon influence had impact on the porosity and size of the surface itself. Using scanning electron microscopy techniques, the estimation of particles size was done confronting with laser diffraction methods particle size evaluation.

Keywords: Nanocarbons, sulfur, nanocomposite, lithium.

Improved DSSC performance with ZnO nanorods grown by using Zn(II)AA/ZnO-NP seed layer

F. Yuliasari[1], Y.S. Handayani[1], H. Pujiarti[2], A. Aprilia[3], H. Bahar[1],

R. Hidayat[1,*]

[1]Physics of Magnetism and Photonics Research Division, Faculty of Mathematics and Natural Sciences, Bandung Institute of Technology, Indonesia; [2]Department of Physics, Faculty of Mathematics and Natural Sciences, State University of Malang, Indonesia; [3]Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Padjadjaran, Indonesia.

rahmat@fi.itb.ac.id

We have investigated the performance of dye-sensitized solar cell (DSSC) with ZnO nanorod (ZnO-NR) as photoanode by using Zn(II)AA/ZnO-NP seed layer. We found that the morphology of ZnO-NR was affected by growth time, seed layer concentration and growth solution concentration of hydrothermally ZnO-NR. XRD characterization showed that ZnO-NR diffraction pattern are indicated to hexagonal wurzite structure and SEM image showed the coral-like nanorod morphology. Then, the ZnO-NR was used as a photoanode in DSSC with device structure FTO/ZnO-NR/D358-dye/mosalyte/Pt/FTO. The best current density-voltage (J-V) was obtained 6.6 mA/cm^2 and the efficiency was reached 2.20%.

Keywords: ZnO nanorod, DSSC, Seed Layer, Hydrothermal, Photovoltaic, Nanoparticles

Acknowledgments: The authors acknowledge the research support from PDUPT program of Ristek Dikti 2020, Indonesia.

Poster presentation (PP):

The use of rapid thermal annealing for the formation of cobalt oxide films

V.V. Bespoludin[1], A.P. Starnikova[2], Yu.N. Varzarev[1], E.M. Bayan[3], V.V. Polyakov[1], V.V. Petrov[2]

[1]Institute of Nanotechnologies, Electronics and Electronic Equipment Engineering, Southern Federal University, Russia; [2]Research and Education Centre “Microsystem technics and multisensory monitoring systems”, Southern Federal University, Russia; [3]Department of Chemistry, Southern Federal University, Russia;

starnikova@sfedu.ru

In this work, cobalt oxide films were formed by rapid thermal annealing (RTA) in the air. The temperature range of the formation of cobalt oxide films by rapid thermal annealing (RTA) was determined by X-ray phase analysis.

Keywords: cobalt oxide films, rapid thermal annealing, x-ray phase analysis.

TOPIC 4

Nanotech for Medicine and Pharmacy

Chair: Josef Jampílek

Nanopharmacology and targered drug delivery
Biocompatible and biodegradable nanomaterials
Nanotoxicology

Invited lectures (IL):

Targeted therapies based on nanocarriers to reduce vascular inflammation

M. Calin

“Medical and Pharmaceutical Bionanotechnologies” Laboratory/ Institute of Cellular Biology and Pathology
“N. Simionescu” of Romanian Academy, Bucharest, Romania

manuela.calin@icbp.ro

Cardiovascular diseases (CVD), a group of disorders affecting the heart and blood vessels remain the leading cause of morbidity and mortality in the world. Despite new therapeutics, a lack of innovative approaches for further improvement is apparent. CVD is primarily caused by atherosclerosis, a systemic disease characterized by lipid deposition in the arteries' walls with an associated, chronic inflammatory process. The aim of our research focuses on the development of advanced targeted therapies, based on nanoparticles, to mitigate the inflammatory process associated with CVD. Targeted nanoparticles can be potentially used as carriers for drug delivery to specific pathological sites. The endothelium, the inner cell layer of the blood vessel wall, and monocyte-derived macrophages are key players in vascular inflammation and are accessible targets for therapeutic approaches. In pathological conditions, the endothelial cells overexpress, on the surface, cell adhesion molecules (such as VCAM-1 and P-selectin) that can be used as targets for nanotherapy. The presentation will cover the development of biomimetic lipid-based nanocarriers directed towards cell adhesion molecules, designed to carry small molecules or nucleic acids to the dysfunctional endothelium or activated monocytes/macrophages. The validation of the developed nanotherapeutics, at the preclinical level, in vitro and in vivo animal relevant models, will be discussed.

Keywords: nanoparticles, endothelium, inflammation, cardiovascular diseases.

Acknowledgments: The work was supported by UEFISCDI, PN-III-P4_ID-PCE-2020-2465 and 13PCCDI/ 2018 (INTERA projects) within PNCDI III.

Modification of Al_2O_3 and ZrO_2 surfaces dedicated as a filler for polymers used in biomedical engineering

A.Dubiel[1], D.Nakonieczny[1], W.Walke[1], M. Antonowicz[1], J.Majewska[1], K.Matus[2], P.Nuckowski[2],

[1]Faculty of Biomedical Engineering, Silesian University of Technology, Zabrze, Poland, University, Country;
[2]Faculty of Mechanical Engineering, Silesian University of Technology, Gliwice, Poland;

agnieszka.dubiel@polsl.pl

One of the main problems with the use of ceramic materials in dental prosthetics is the uneven dispersion of particles in the polymer matrix. In addition, the unfavorable phenomena occurring at the interface between the veneered ceramic layer and the crown framework, caused by mechanical stress, lead to joint decohesion and, consequently, complete delamination of the veneered layer. In order to prevent this type of negative phenomena, the surface of both chemical and mechanical bonding between these elements should be increased. The authors of the study proposed the

functionalisation of the Al_2O_3 and ZrO_2 surfaces by using a solution of silicon nitride with sodium hydroxide (10M) and 3-aminopropyltetraethoxysilane (APTES). The test specimens were calcined at the temperature T = 250ºC, T = 350ºC and T = 450ºC in the atmosphere of air and nitrogen. To verify the effectiveness of the proposed modification method, thermogravimetric analysis (TGA), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR-AT) and scanning electron microscopy (SEM-EDS) observations were performed. The XRD and TGA analyzes revealed the presence of amorphous spaces. In turn, SEM-EDS analysis showed the deposition of silicon and a change in surface morphology. The presented research is a preliminary stage to the development of the silanization process of spherical aluminosilicates (cenospheres) for applications in medicine and dental prosthetics.

Keywords: silicon oxide, zirconium oxide, silanization, APTES.

Multifunctional Harmonic Nanoparticles for Cell Tracking and Theranostic Applications

A. Gheata[1], G. Gaulier[2], G. Campargue[2], J. Vuilleumier[1], D. Staedler[3], D. Diviani[3],
L. Bonacina[2], S. Gerber-Lemaire[1]

[1]Group for Functionalized Biomaterials, Institute of Chemical Sciences and Engineering, Ecole Polytechnique Fédérale de Lausanne, Switzerland; [2]Department of Applied Physics, Université de Genève, Switzerland; [3]Department of Biomedical Sciences, University of Lausanne, Switzerland

sandrine.gerber@epfl.ch

In comparison with luminescent probes, harmonic nanoparticles (HNPs), which are based on metal oxide nanocrystals lacking of inversion symmetry, benefit from excitation spectral flexibility, fully coherent emission and high photostability. These appealing properties constitute unique assets for bioimaging protocols beyond the classical NIR I tissue transparency window. Surface functionalization of HNPs offers the possibility to exploit these unique capabilities in highly demanding applications such as cell tracking in tissue depth, multiscale and multimodal imaging and on-demand light-triggered release of therapeutic payloads. We herein disclose efficient conjugation protocols for surface coating and post-functionalization of HNPs toward the development of theranostic nanodevices.

Keywords: Multifunctional nanoparticles, Multimodal imaging probes, Cell labelling, NIR-light triggered uncaging.

Acknowledgments: We acknowledge financial support from the France-Switzerland Interreg program (Interreg fédéral, Vaud state, Geneva state, projects NANOFIMT and OncoNanoScreen).

Nanomaterials as antiparasitics

J. Jampilek[1,2]

[1]Department of Analytical Chemistry, Faculty of Natural Sciences, Comenius University in Bratislava, Slovakia; [2]Regional Centre of Advanced Technologies and Materials, Palacky University Olomouc, Czech Republic

josef.jampilek@gmail.com

For many decades, diseases caused by parasites were among the so-called neglected diseases, and the development of antiparasitic drugs was minimal. However, with climate change, travel and global food distribution, more and more species of parasites are becoming a serious problem. In general, parasites can be classified into three main groups: ectoparasites (e.g., lice and fleas), helminths (i.e. worm parasites, e.g., roundworm, tapeworm, fluke) and protozoa (i.e. the single-celled organism, e.g., *Plasmodium*, *Trypanosoma*, *Leishmania*, *Entamoeba*, *Toxoplasma*, *Trichomonas*). Thus, it is evident that they are not only parasites causing tropical diseases, but many parasites also occur in temperate regions, although their diversity and activity increase with increasing temperature. The number of parasites harmful to agricultural/food valuable plants or animals is even greater. The choice of appropriate drugs is very limited, and therefore development is logically focused on reformulating older drugs to reduce toxicity, increase efficacy, and target distribution to parasite-affected tissues. Nanoscale science and nanotechnology have unambiguously demonstrated to have a great potential in providing novel and improved solutions. Nano-size materials change their physical and chemical properties in comparison with bulk materials and have helped to improve and innovate a variety of pharmaceutical, medical, industrial, and agricultural products. Thus, this contribution deals with the biological activity of various types of nanoparticles/nanomaterials against parasites.

Keywords: Antiparasitics, helmints, nanoparticles, protozoa.

Acknowledgments: This study was supported by the Slovak Research and Development Agency; project APVV-17-0373.

Targeting metastasis in cancer through the metastasis suppressor, ndrg1, using innovative therapeutics

D.R. Richardson[1]

Centre for Cancer Cell Biology and Drug Discovery, Griffith Institute for Drug Discovery, Griffith University, Nathan, Brisbane, Australia. 4111

d.richardson@griffith.edu.au

The metastasis suppressor, N-myc downstream-regulated gene-1 (NDRG1) inhibits a plethora of oncogenic signalling pathways by down-regulating the epidermal growth factor receptor (EGFR). Herein, we examined the mechanism involved in NDRG1-mediated EGFR down-regulation.[1]

NDRG1 overexpression potently increased the levels of mitogen-inducible gene-6 (MIG6), which inhibits EGFR and facilitates its lysosomal processing and degradation. Conversely, silencing *NDRG1* in multiple human cancer cell-types decreased MIG6 expression, demonstrating the regulatory role of NDRG1.

Further, NDRG1 overexpression facilitated MIG6-EGFR association in the cytoplasm, possibly explaining the significantly (p <0.001) increased half-life of MIG6 from 1.6 ± 0.2 h under control conditions to 7.9 ± 0.4 h after NDRG1 overexpression. The increased MIG6 levels enhanced EGFR co-localization with the late endosome/lysosomal marker, lysosomal-associated membrane protein 2 (LAMP2).

An increase in EGFR levels after *MIG6* silencing was particularly apparent when NDRG1 was overexpressed, suggesting a role for MIG6 in NDRG1-mediated down-regulation of EGFR.

Anti-tumor drugs of the di-2-pyridylketone thiosemicarbazone class that activate NDRG1 expression also potently increased MIG6 and induced its cytosolic co-localization with NDRG1. This was accompanied by a decrease in activated and total EGFR levels and its redistribution to late endosomes/lysosomes.

The delivery of such agents *via* special nanoparticles known as “nanochelators” will be discussed.

Keywords: N-myc downstream regulated gene-1, epidermal growth factor receptor, mitogen-inducible gene 6, nanochelators

Acknowledgments: D.R.R. thanks the National Health and Medical Research Council of Australia for a Senior Principal Research Fellowship and Project Grants.

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Albumin loaded PEG/HAp composite coatings with bone regenerative potential

A. Sobczak-Kupiec[1], D. Słota[1], W. Florkiewicz[1]

[1] Faculty of Materials Engineering and Physics, Cracow University of Technology, Poland

agnieszka.sobczak-kupiec@pk.edu.pl

The aim of the presented research was to create a ceramic-polymer coatings, demonstrating bioactive properties, for potential use in bone tissue regeneration. For this purpose, hydroxyapatite, known for its osteoconductive properties and polymer phase, which was (PEG), were used. This system was then modified by the addition of bovine serum albumin. Hydroxyapatite was obtained by wet precipitation method and characterized using X-ray diffraction and FT-IR. Ceramic-polymer coatings with albumin were prepared using a photopolymerization method. Coatings were then incubated in simulated biological fluid (SBF) and the changes occurring due to the interaction of the material with the fluid were observed using SEM imaging. Analysis of the surface morphology demonstrated that new apatite layers appeared after 14 days of incubation. This satisfactory information confirms the interactions between the sample and the incubation fluid, thus demonstrating the bioactivity of the materials obtained.

Keywords: Hydroxyapatite; ceramics; biomaterials; coatings; biomineralization;

Acknowledgments: The “Multifunctional biologically active composites for applications in bone regenerative medi-cine” project is carried out within the TEAM-NET program of the Foundation for Polish Science financed by the European Union under the European Regional Development Fund. The authors gratefully acknowledge financial support.

NiTi alloy substrate preparation effect on the adhesion of Ta_2O_5 layer applied by ALD method

A. Taratuta[1], J. Lisoń[1], D. Nakonieczny[1], Z. Paszenda[1], M. Basiaga[1], M. Antonowicz[1]

[1]Department of Biomaterials and Medical Devices Engineering, Silesian University of Technology, Poland

anna.taratuta@polsl.pl

This paper evaluates the suitability of Atomic Layer Deposition (ALD) method for surface modification of NiTi alloy used for implants in contact with blood under specific technological conditions. Selected material belongs to the category of shape memory alloys. In order to evaluate the suitability of thin film deposition technology, mechanical and physical property tests were proposed. NiTi alloy was subjected to the following surface modifications: electropolishing, passivation: in air at 450°C for 1 hour, etching with Kroll's reagent and boiling for 2 hours in 30% H_2O_2, passivation in autoclave at 135°C for 30 minutes, and boiling in distilled water for 1 hour. Then the Ta_2O_5 layer was deposited by ALD method. Tantalum was chosen due to its good fluoroscopic visibility. Subsequent variants of the process differed in deposition temperature and number of cycles. The mechanical and physical properties were then evaluated, which has a fundamental significance in the case of implants used in the cardiovascular system. The obtained results have a direct impact on the process of optimizing the deposition of Ta_2O_5 layer by ALD method on NiTi implants in contact with blood, by improving their functional properties.

Keywords: atomic layer deposition (ALD), shape memory alloy, tantalum oxide layer, biomedical materials

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Mitochondria-Targeted Pharmaceutical Nanocarriers

V. Weissig

Department of Pharmaceutical Sciences, College of Pharmacy Glendale, Midwestern University, Arizona, USA

vweiss@midwestern.edu

Mitochondria are a prime therapeutic target. Tremendous pharmacological efforts undertaken led to the emergence of “Mitochondrial Medicine” as a new field of biomedical research [1, 2]. The identification of molecular target sites at or inside mitochondria and the discovery of appropriate drug candidates will launch a multitude of new therapies for the treatment of mitochondria-related diseases. As underappreciated, however, appears the development of drug delivery systems for achieving a high bioavailability of the drug at its target site. A random observation at the laboratory bench has helped paving the way towards the development of mitochondria-targeted nanocarriers [3]. A fortuitous discovery in the mid-1990s [4] involving the self-assembly of a molecule, known to accumulate inside mitochondria, has led to the development of subcellular nanocarriers suited for the selective delivery of biological active molecules to mitochondria inside living mammalian cells [5]. In 2008 it was shown that the surface modification of nanocarriers with mitochondriotropic triphenylphosphonium cations facilitates the efficient subcellular delivery of a model drug to mammalian mitochondria [6] and the potential of mitochondria-targeted nanocarriers to enable the manipulation of the mitochondrial genome has been demonstrated in 2011 [7].

In this presentation, the current state-of-the-art of mitochondrial drug targeting technology will be reviewed.

Keywords: Mitochondria, Nanocarrier, Drug Targeting, DQAsomes, Liposomes

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Oral presentations (OP):

Encapsulation of hydrophobic drugs in hyaluronic acid nanofibers: effect of the solution preparation procedure

L. Bardoňová[1,2], A. Kotzianová[1], J. Kulhánek[1], O. Židek[1], K. Mamulová Kutláková[2], V. Velebný[1]

[1]Contipro a.s, Czechia; [2]Nanotechnology Centre, Energy and Environmental Technology Centre, VŠB – Technical University of Ostrava, Czechia;

bardonova@contipro.com

Perspective application of nanofibrous mats composed of hydrophilic polymers are the fast-dissolving drug delivery systems for immediate release of a wide spectrum of additives, including poorly soluble drugs. For the effective preparation of drug carriers, it is necessary to load additives with high encapsulation efficiency. In our study, we aimed to prepare hyaluronic acid/polyethylenoxide nanofibers with a hydrophobic antiseptic agent (ANT). We have used several approaches of solution preparation to achieve ideally 100 % encapsulation efficiency of ANT. The approaches included dispersing ANT in polymer solution (dispersion), creating an emulsion with the surfactant Tween 60 in water before dissolving the polymers (emulsion), and dissolving all the components in a single solvent system (blend). The morphology of the nanofibers was characterized by SEM, amount of encapsulated ANT was analyzed by HPLC, and the distribution of ANT was mapped by Raman spectroscopy. The best results were obtained by blend electrospinning. The encapsulation efficiency of ANT was over 95 % and ANT was distributed in the nanofiber mat homogeneously. The yield of the electrospinning process was also 3-fold higher than the yield of dispersion and emulsion electrospinning.

Keywords: hyaluronic acid, antiseptics, nanofiber, electrospinning

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Structure-property relationship study of silver nanoparticles stabilised with ammonium and phosphonium surfactants

M. Pisárčik

Department of Chemical Theory of Drugs, Faculty of Pharmacy, Comenius University, Bratislava, Slovakia

pisarcik@fpharm.uniba.sk

The present study shows the effect of alkyltrimethylammonium bromide and alkyltrimethylphosphonium bromide surfactants on physical properties and cytotoxicity of surfactant-stabilised silver nanoparticles (AgNPs). As the results indicate, the interaction of alkyltrimethylphosphonium surfactant molecules with AgNPs results in a slightly larger particle diameter and a less positive zeta potential value which is to relate to weaker association of bulkier phosphonium headgroups on AgNPs surface. The investigations of physical parameters of AgNPs stabilised with alkyltrimethylphosphonium bromides correspond with their increased cytotoxicity when compared with AgNPs stabilised with alkyltrimethylammonium bromides.

Keywords: silver nanoparticles, phosphonium, ammonium, cytotoxicity

Acknowledgments: This work was supported by the Grant Agency of Ministry of Education and Academy of Science of Slovak republic (VEGA), Projects No. 1/0686/21 and 1/0054/2019 and by the Slovak Research and Development Agency Grant No. APVV-17-0373.

Halloysite nanotubes as suitable carriers for irinotecan from molecular simulations point of view

M. Pospíšil[1], E. Gianni[2], K. Avgoustakis[3], M. Pšenička[1], D. Papoulis[2]

[1]Faculty of Mathematics and Physics, Charles University, Czech Republic; [2]Department of Geology, University of Patras, Greece; [3]Department of Pharmacy, University of Patras, Greece

miroslav.pospisil@mff.cuni.cz

Halloysite is a clay mineral with tubular morphology, where individual clay sheets create spiral-shape. Because of its compatibility with the human bodies, halloysite can be used as effective carrier for delivery of drugs which are usually connected on the inner or outer part of nanotube. The halloysite nanotubes are presented as a potential drug delivery system of irinotecan, which is used for colon cancer treatment and due to halloysite it is administered by the oral route. The whole system irinotecan-loaded halloysite nanotubes were coated with Eudragit S100, which is an anionic copolymer in order to control and limit the amount of the drug release in the stomach pH while allowing release in the intestinal environment. The capturing of irinotecan was very high, reaching 84.42 ± 3.10%.

The molecular simulations were used for the determination of the mutual interactions between the irinotecan cations and the halloysite nanotube at release pH value (pH 7.4). Results of optimized geometry showed that the cationic form of drug molecules remain closer to the outer part of the halloysite nanotubes. Longitudinal axes of the adjacent drug cations are positioned along the longitudinal axis of the tube and based on the calculations, these positions are energetically preferred.

Thin polymeric films with antimicrobial nanofiller

M. Ščuková, S. Holešová, K. Čech Barabaszová

Nanotechnology Centre, CEET, VŠB - Technical University of Ostrava, 17. listopadu 15/2172, 708 00 Ostrava - Poruba, Czech Republic

michaela.scukova.st@vsb.cz

Thin polymeric films with antimicrobial nanofillers were prepared for their possible medical applications. The biodegradable polymer polycaprolactone (PCL) was used as the polymer matrix and clay mineral vermiculite was used as carrier for antimicrobial components. The active organic antimicrobial compound is antifungal ciclopiroxolamine (CPX) which has been intercalated to vermiculite. For possible synergistic effects, a sample in combination with the inorganic antimicrobial active ingredient zinc oxide (ZnO) was also prepared. The structure of the prepared samples was characterized by X-ray diffraction (X-ray) analysis, infrared spectroscopy (IR) and scanning electron microscopy (SEM). Furthermore, the organic carbon content was determined for the powder samples and the particle size distribution (PSD) and ζ-potential were also measured. The antimicrobial activity of both the powder nanofillers and the resulting polymer films was tested against bacterial strains of *Staphylococcus aureus* and *Escherichia coli* and against the yeast *Candida albicans* and finally, the results were compared. The antimicrobial effect has been demonstrated at various time intervals. From the powder samples, the VER:CPX sample (1:1) showed the greatest effect. In the case of polymer films, there is a probability of response against individual strains in longer time intervals.

Keywords: Polycaprolactone, vermiculite, zinc oxide, ciclopiroxolamine, antimicrobial effect

Acknowledgments: This work was supported by the project No. SP2020/08 “Hybrid clay nanofillers for antimicrobial polymer films” and project No. SP2021/106 “Study and development of composite nanomaterials and nanofillers”.

Poster presentations (PP):

Study of UDD under experimental carcinogenesis

L.V. Batyuk[1], N.N. Kizilova[2,3]

[1]Department of Medical and Biological Physics and Medical Information Science/Kharkiv National Medical University, Ukraine; [2]Warsaw University of Technology, Institute of Aviation and Applied Mechanics, Poland; [3]PoreLab, Department of Chemistry, Norwegian University of Science and Technology, Norway

lv.batyuk@knmu.edu.ua

In article the protection ability of the ultra-disperse nanodiamonds (UDD) is studied based on the measurements of content of electrolytes in plasma and erythrocytes of Wistar rats with Guerin's carcinoma under conditions of irradiation by 10.0 Gy and use of UDD. The groups of rats with Guerin's carcinoma and with X-ray irradiation and both with used UDD and X-ray irradiation treatments were compared to the control group. In the experiment, the content of electrolytes in blood plasma and erythrocytes of rats was measured by flame photometry. It was shown, that UDDs have a radioprotective effect under conditions of external exposure of the body of rats. The radiation activities of UDDs are based on its specific membranotropic properties, which integrally increases the general nonspecific resistance of the organism.

Keywords: Carcinoma, X-ray, erythrocytes, blood plasma, nanodiamonds

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The use of different silica gel RP-TLC plates for the determination the lipophilicity parameters of metformin and phenformin

Małgorzata Dołowy[1], Klaudia Całus-Makowska[2], Josef Jampilek[3]

[1]Department of Analytical Chemistry, Faculty of Pharmaceutical Sciences in Sosnowiec, Medical University of Silesia in Katowice, Poland; [2]Student Scientific Group at the Department of Analytical Chemistry in Sosnowiec; [3]Department of Analytical Chemistry, Faculty of Natural Sciences, Comenius University, Bratislava, Slovakia

josef.jampilek@gmail.com

Silica gel in form of different TLC and RP-TLC plates is the major material used as stationary phase for chromatographic study of different drug substances. Variety of commercially available silica gel plates consisting of small particle size silica gel is also efficient in the determination the lipophilicity parameter of numerous bioactive compounds [1]. Because lipophilicity is essential parameter for pharmacokinetics, pharmacodynamics and toxicity of bioactive molecules and metformin and phenformin exhibit the biological activity i.e. antidiabetic and antitumor, the aim of this study was to determine the chromatographic parameter lipophilicity of both biguanide derivatives and to compare it with theoretical values of logP [2,3].

Keywords: Silica gel plates, lipophilicity, metformin, phenformin

Acknowledgments: This research was supported by the research grants from Medical University of Silesia in Katowice, Poland No. PCN-1-099/K/0/F in 2020 year.

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Gold nanorods with organosilica shell as potential dual-therapeutic agents

N.A. Salavatov, O.V. Dement'eva, V. M. Rudoy

Frumkin Institute of Physical Chemistry and Electrochemistry, Russian Academy of Sciences, Moscow

gop--nick@yandex.ru

The application of nanoparticles in the biomedical field opens up new possibilities in the diagnosis and treatment of cancer and other socially significant diseases. Recently, special attention has been paid to more complex composite nanoparticles. Such structures can combine several functions, for example, serve as diagnostic and therapeutic agents of dangerous diseases. The common known type of composite nanoparticles is nanoparticles with a noble metal core (for example, gold nanorod) and a silica shell. The main property of gold nanorods is longitudinal localized surface plasmon resonance (LSPR). The position of the LSPR band depends on their aspect ratio and can be easily “tuned” in a wide spectral range, including the near-IR spectral region (i.e. the region of biological tissue optical window). Therefore, gold nanorods are used in optical diagnostics and photothermal therapy of cancerous tumors. Silica shells of composite nanoparticles can serve as the container for dye molecules, drugs, etc. Tetraethoxysilane (TEOS) is usually used as a precursor for silica shells. In this case, to obtain a mesoporous shell, it is necessary to use a surfactant. We propose to use for this purpose another precursor – 3-mercaptopropyltrimethoxysilane (MPTMS) which has several advantages over TEOS. First, the thiol group has a chemical affinity for the surface of gold nanorods. Second, the asymmetric structure of MPTMS molecule provides the formation of a porous shell without the use of surfactants in comparison with TEOS. Also it should be noted that reactive SH-group of the MPTMS can be used for shell functionalization with various compounds. For example, it is well known that thiol groups coordinate the ions of different metals.

In this work, we demonstrate the advantages of $SiO_{1.5}SH$-shells by loading a popular and well-studied chemotherapy drug – cisplatin.

In the seedless synthesis of gold nanorods, in contrast to the classical “seeded approach”, particle nucleation and growth occur in one pot, which greatly simplifies the process. This protocol allows us to obtain monodisperse gold nanorods and to tune the position of the LSPR in a wide wavelength range. During the synthesis of $SiO_{1.5}SH$-shells, the formation of by-products as coreless organosilica nanoparticles is detected. To solve this problem, it is necessary to suppress homogeneous nucleation. We succeeded in achieving this aim by drop-wise injection MPTMS, increasing the concentration of gold nanorods (i.e. centers of heterogeneous nucleation) and the amount of ethanol in the reaction system. Moreover, the synthesis was carried out at an elevated temperature (40–60 °C). The optimized synthesis protocol allows us not only to completely suppress the formation of organosilica nanoparticles but also to provide fine control of the $SiO_{1.5}SH$-shell thickness of composite nanoparticles. The formation of $SiO_{1.5}SH$-shells predictably leads to a bathochromic shift of the longitudinal LSPR, which is associated with a change in the dielectric constant near the gold nanorods. After sorption of cisplatin from aqueous solutions in $SiO_{1.5}SH$-shells, the plasmon characteristics of composite nanoparticles change only slightly, that indicates the aggregative stability of the dispersion. Preliminary data on the peculiarities of cisplatin sorption were obtained. We should note that the sorption value is high enough and reaches approximately 0.2 g/cm^3. The loading of cisplatin into $SiO_{1.5}SH$-shells is confirmed by elemental mapping by energy dispersive analysis of X-rays. Besides the expected elements like Au, S, Si, the Pt is also detected in a rather high quantity. The assumption that thiol groups are responsive for the high sorption value of cisplatin is confirmed by the data of FTIR spectroscopy. The band near 2560 cm^{-1}, corresponding to the stretching vibrations of the SH-group, disappears after the sorption of cisplatin.

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Biodegradable polymeric materials with antimicrobial effects suitable for medical applications

K. Škrlová[1,2], K. Malachová[3], Z. Rybková[3], T, Stachurová[3], J. Zagora[1], Marta Fernández-García[4], and Daniela Plachá [1,5]*

[1]Nanotechnology Centre, VŠB–Technical University of Ostrava, 17. listopadu 15, 708 00 Ostrava-Poruba, Czech Republic; [2]Center of Advanced Innovation Technologies, VŠB–Technical University of Ostrava, 17. listopadu 15, 708 00 Ostrava-Poruba, Czech Republic; [3]Department/Research Institute, University, Country; [3]Department of Biology and Ecology, Faculty of Science, University of Ostrava, Chittussiho 10, 71000 Ostrava, Czech Republic; [4]Institute of Polymer Science and Technology (ICTP-CSIC), Juan de la Cierva 3, 28001 Madrid, Spain; [5]ENET Centre, VŠB–Technical University of Ostrava, 17. listopadu 15, 708 00 Ostrava -Poruba, Czech Republic

katerina.skrlova@vsb.cz

Biodegradable polymers are promising materials for use in medical applications. The properties of these materials are comparable to commercial non-biodegradable polymers. This paper focuses on the preparation of biodegradable polymer composites based on polylactide, which are modified with antimicrobial fillers based on silicate and graphene matrices, which have been enriched with antimicrobial agents: Ag, hexadecyltrimethylammonion and hexadecylpyridinium cations. These composites have been investigated for their chemical-physical, degradable and antimicrobial properties.

Keywords: Polylactide, antimicrobial, biodegradable, degradable, silver, medical applications

Acknowledgments: This research was funded by MSMT projects No. CZ.02.1.01/0.0/0.0/17_049/0008441 "Innovative Therapeutic Methods of Musculoskeletal System in Accident Surgery" within the Operational Programme Research, Development, and Education financed by the European Union and by the state budget of the Czech Republic; Doctoral grant competition VSB - Technical University of Ostrava, reg. no. CZ.02.2.69/0.0/0.0/19_073/0016945 within the Operational Programme Research, Development and Education, under project DGS/TEAM/2020-001 "Development of antimicrobial biobased polymeric material using supercritical fluid technology; Programme i-LINK+ 2020: Design of novel antimicrobial biobased materials using supercritical fluids processes, LINKA20364.

Green synthesized silver nanoparticles: antibacterial activity, biocompatibility, and analyses of surface-attached proteins

J. Trzcińska-Wencel[1], M. Wypij[1], M. Rai[1,2], P. Golińska[1]

[1]Department of Microbiology, Nicolaus Copernicus University, Poland

[2]Nanobiotechnology Laboratory, Department of Biotechnology, SGB Amravati University, India

trzcinska@doktorant.umk.pl

In this study, the antibacterial and cytotoxic effect of AgNPs biosynthesized from filamentous actinobacterium, strain SF23, was evaluated against bacteria by determination of MIC, MBC and ATP level, as well as against cancer (MCF-7) and murine macrophages (RAW 264.7) using MTT assay, measurement of LDH release and determination of ROS level. Moreover, the capping proteins of NPs were identified by LC-MS/MS. Nanoparticles exhibited antibacterial activity (MIC of 8-128 µg mL^{-1}, MBC of 64-256 µg mL^{-1}) and significantly decreased ATP level in bacterial cells. The *in vitro* analysis showed dose-dependent cytotoxicity of AgNPs. Cell viability decrease was found to be 42.2-14.2% and 38.0-15.5% while LDH leakage 14.6-42.7% and 19.0-45.0%, respectively. Moreover, MCF-7 cells released a greater amount of ROS than RAW 264.7 macrophages during stimulation with AgNPs. The SDS-PAGE analysis revealed the presence of 5 protein bands at a molecular weight between 31.7 and 280.9 kDa. These proteins showed the highest homology to hypothetical proteins and porins from *E. coli*, *Delftia* sp. and *P. rhodesiae*. Obtained results suggest antimicrobial and anticancer potential of biosynthesized AgNPs in medical applications in the future.

Keywords: biogenic AgNPs, capping proteins, antibacterial and anticancer agents, cytotoxicity

Acknowledgments: This research was funded by National Science Centre (NSC), Poland, grant Preludium (no. 2016/23/N/NZ9/00247).

Ag nanoparticles fixation on polycaprolactone-polyvinyl alcohol membrane and biocompatibility evaluation study

Zuzana Vilamová[1], Zuzana Konvičková[1,2,3], Richard Dvorský[1,4], Petr Mikeš[5], Ludmila Porubová[6], Jiří Bednář[1,4], Ladislav Svoboda[1,4], Gabriela Kratošová[1]

[1] Nanotechnology Centre, CEET, VŠB – Technical University of Ostrava, Czech Republic, [2] ENET Centre, CEET, VŠB – Technical University of Ostrava, Czech Republic, [3] Department of Machining, Assembly and Engineering Metrology, Faculty of Mechanical Engineering, VŠB – Technical University of Ostrava, [4] IT4 Innovations, VŠB – Technical University of Ostrava, Czech Republic, [5] Department of Chemistry, Faculty of Science, Humanities and Education, Technical University of Liberec, Czech Republic, [6] Public Health institute Ostrava, Czech Republic

Zuzana.Vilamova.st@vsb.cz

The air filtration through nanofibrous membranes possesses several obstacles. The most problematic is the dependence of the air filtration efficiency vs. particle size. The filter efficiency shows a different position of minimum for various particle sizes, called the Most Penetrating Particle Size (MPPS), which also depends on the electrostatic component of the Hamaker interaction causing the capture of particles on nanofibers. Modulating the electric field through a nanofibrous membrane doped by conductive nanoparticles (NPs) in dispersion below the percolation threshold changes NPs

polarization and affects both the depth of minimum and the position of the MPPS. We prepared polymer-metal based membrane using electrostatic processes for air filtration membrane. The silver nanoparticles (Ag NPs) were encapsulated in the polyvinyl alcohol (PVA) electrospun droplets and these were attached on the prepared polycaprolactone (PCL) fibers. We observed Ag NPs clusters incorporated in PVA droplets by SEM analysis. Ag NPs proved efficient antibacterial properties against *Escherichia coli (E. coli)* and *Staphylococcus aureus (S. aureus)*, and low cytotoxic level on Vero cell line. This test was chosen to investigate the membrane biocompatibility in case of damage and Ag NPs rapid release into the organism.

Keywords: Silver nanoparticles, air filter membrane, antibacterial properties, cytotoxicity

Acknowledgments: Our work has been supported by the Doctoral grant competition VSB - Technical University of Ostrava, reg. no. CZ.02.2.69/0.0/0.0/19_073/0016945 within the Operational Programme Research, Development and Education, under project DGS/TEAM/2020-001 “Organic and inorganic pathogenic nanoparticles and the formation of appropriate protective barriers based on electroactive nanomaterials”, by project Gamma PP1 No. TP01010036 by Technology Agency of the Czech Republic and internal project No. SP2021/106 and by the IT4Innovations national supercomputing center - path to exascale project (No. EF16_013/0001791) Ministry of Education, Youth and Sports. We especially appreciate the kindness of the PrimeCell Advanced Therapy Company for the use of its equipment in material preparation and Kateřina Rosenbergová for antibacterial testing.

Mechanistic insights onto the synergetic effects of AgNPs and antibiotics

M. Wypij[1,2], P. Golińska[1], V. Puntes[2]

[1]Department of Microbiology, Nicolaus Copernicus University, Poland

[2]Catalan Institute of Nanoscience and Nanotechnology (ICN2), Barcelona, Spain

mwypij@umk.pl

The antimicrobial activity of silver nanoparticles (AgNPs) and combination of AgNPs with selected antibiotics (amikacin, colistin, tetracycline and vancomycin) was studied against Gram-positive (*Staphylococcus aureus*) and Gram-negative (*Escherichia coli*, *Pseudomonas aeruginosa*) bacteria. The minimum inhibitory concentrations (MICs) and minimum biocidal concentrations (MBCs) were evaluated using micro-dilution method. Combined effect was determined by estimation of FIC (Fractional inhibitory concentration) index. The antimicrobial activity of AgNPs strongly depended on their size and was noticed for those of 21 nm in size. The lowest MIC of AgNPs was noticed against *Escherichia coli* (58 μg ml^{-1}), followed by *Staphylococcus aureus* and *Pseudomonas aeruginosa* (both 178 μg ml^{-1}). The synergistic effect of antibiotics with silver nanoparticles against tested bacteria was found mainly for tetracycline. The obtained results clearly indicate that combination of selected antibiotics with nanoparticles enhances antibacterial activity in a way that traditional antibiotics cannot do.

Keywords: AgNPs, antibacterial agents, antibiotics

TOPIC 5

Nanotech for Environmental Solution

Chair: Ivo Šafařík

Green chemistry for nanomaterials
Environmental chemistry
Ecotoxicology and Environmental Safety

Invited lectures (IL):

Chemically selective vapor sensing of structurally colored butterfly wings

G. Piszter[1], K. Kertész[1], Z. Bálint[2], L.P. Biró[1]

[1]Institute of Technical Physics and Materials Science, Centre for Energy Research, Hungary; [2]Hungarian Natural History Museum, Hungary

piszter@mfa.kfki.hu

The photonic-crystal-type nanoarchitectures occurring in the wing scales of the butterflies are mainly composed of chitin and air, and the structural coloration they generate is determined by the periodicity and characteristic dimensions of the nanostructure, and also by the refractive indices of the building materials. Small changes in the refractive index or in the periodicity, like those induced by different vapors in the surrounding atmosphere, can generate color variation of the wings detectable with optical spectrophotometry. The mechanism behind vapor sensing is the capillary condensation of the volatiles into the nanocavities of the photonic nanoarchitecture which results in a reversible color change. In the present work, the general findings regarding the mechanism of sensitivity and selectivity, and the parameters influencing the sensing will be discussed. We demonstrated that the butterfly wing-based sensor materials allow chemically selective detection of single-component vapors as well as binary vapor mixtures. The sensitivity of the wings was tuned by modifying the surface of the photonic nanoarchitecture, and the effect of long-term vapor exposure and how these sensors work in real life conditions, for example in simulated environmental humidity, were investigated.

Keywords: photonic nanostructure, vapor sensing, butterfly, chemical selectivity

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Biogenic silver nanoparticles: What we know and what we need to know?

Mahendra Rai

Visiting Scientist, Department of Microbiology, Nicolaus Copernicus University, Lwowska 1, 87 100, Torun, Poland

mahendra.rai@v.umk.pl

The green synthesis of nanomaterials has garnered the attention of nanotechnologists, and has given birth to a new branch of “Green nanotechnology”. The biosynthesis of silver nanoparticles is a green route and follows the principles of Green chemistry which is the basis of sustainable development. The process can be performed by various microorganisms such as fungi, bacteria, green algae, and plants. Biosynthesis has advantages over the physical and chemical processes being eco-friendly, cost-effective, can be performed at ambient temperature and pressure, with enhanced bioactivity, and less toxic. There are reports that the proteins and enzymes act as reducing and also capping agents. However, the actual mechanism of synthesis of silver nanoparticles by the green route has not been fully elucidated. The biogenically synthesised silver nanoparticles have demonstrated broad-spectrum bioactivities against various emerging and multidrug-resistant pathogenic microbes and are supposed to a new generation of antimicrobials. In the context of the current COVID-19 pandemic,

nanomaterials can be used for detection, drug/vaccine delivery, and personal protective equipment. The talk will be focused on the biogenic synthesis of nanoparticles, the mechanism of synthesis, biomedical applications, the challenges, key areas of research, toxicity and safety issues.

Keywords: Green synthesis, Biogenic, silver nanoparticles, mechanism, broad-spectrum, Biomedical

Magnetic materials in biosciences, biotechnology and environmental technology

I. Šafařík[1,2], J. Procházková[1], E. Baldíková[1], K. Pospíšková[2]

[1]Department of Nanobiotechnology, Biology Centre, ISB, CAS, České Budějovice, Czech Republic [2]Regional Centre of Advanced Technologies and Materials, Czech Advanced Technology and Research Institute, Palacký University, Olomouc, Czech Republic

ivosaf@yahoo.com

Magnetically responsive materials exhibit several types of responses to external magnetic field. Such materials can find many interesting applications in various areas of biosciences and biotechnology. The presentation will at first summarize the main characteristic features of magnetically responsive materials from the point of view of a bioscientist. In the second part developed procedures enabling to prepare magnetically responsive materials from diamagnetic particulate and high aspect ratio (bio)materials will be shown. Finally, the magnetic materials based research at the Department of Nanobiotechnology will be presented, showing the achieved results in main disciplines, namely in biochemistry, microbiology, algology, biotechnology, catalysis, polymer chemistry, (nano)textile research, (bio)analytical chemistry, environmental technology and medicine.

Oral presentations (OP):

Microfluidics and biotechnology for nanoparticles synthesis in a green way

G. Kratošová[1], J. Klusák[2], M. Večeř[2], D. Orlová[1], V. Rybníčková[1]

[1]Nanotechnology Centre, CEET, VSB - Technical University of Ostrava, Czech Republic; [2]Department of Chemistry, Faculty of Materials Science and Technology, VSB - Technical University of Ostrava, Czech Republic

gabriela.kratosova@vsb.cz

Tilia cordata and other plants as well as siliceous algae were used for a period in our laboratory for gold and silver nanoparticles biosynthesis[1]. Because of some issues of batch biosynthesis, typically process reproducibility and nanoparticles heterogeneity in sense of their shape and size, we are currently focusing on nanoparticles phytosynthesis using microfluidic 3D printed microreactors. Main advantage of low-cost biosynthesis, which is reduction and simultaneous nanoparticles stabilization, is thus enhanced by the benefits of a controlled flow process. Preliminary tests with silver on-chip synthesis revealed that different flow rates in the range 10 - 60 ml/ h on the same chip have no grate effect on silver nanoparticles morphology and size. However, when chip with more complicated channels geometry (Zig-Zag or 3D Zig-Zag) and micro-mixing system is used, phytosynthesized nanoparticles seem to be smaller and better stabilized by phytomolecules. Due to silver reactivity in the final colloid and agglomeration tendency, further experiments of oil microfluidic phytosynthesis were performed and showed that enclosure of reaction mixture in a drop leads to the formation of even smaller particles then in previous cases. In general, applying microfluidics completely different results regarding shape, size and stability of nanoparticles may be achieved compared to the batch synthesis and various process conditions must be investigated to find the optimal ones for a given plant, metal precursor and micro-reactor.

Keywords: biosynthesis, nanoparticles, microfluidics, bionanotechnology

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Hetero- and homo-aggregation of ZnO nanoparticles in different size fractions of colloids extracted from agricultural soil

S. Kurtinová

Institute of Laboratory Research on Geomaterials, Faculty of Natural Sciences, Comenius University in Bratislava, Slovakia

Slovak Spectroscopic Society, member of the Association of Slovak Scientific and Technological Societies

kurtinova2@uniba.sk

In the last decades, there is a significant increase in the application of zinc oxide nanoparticles (ZnO-NPs) in the agroindustry. With the increasing application of nanofertilizers, there is also a higher risk of negative impact of NPs' exposure on development and growth of crops[1,2]. Furthermore, the exposure of engineered NPs has generally more severe responses of crops in comparison to the effects of micro- and macro-sized analogues[3]. Nevertheless, it has been shown that NPs can alleviate the impact of biotic and abiotic stresses on crops[4]. Whether the impact of NPs is negative or positive depends primarily on their physico-chemical properties, concentration and nature of their interactions with the environment, e.g. aggregation with soil components[5,6]. To highlight the risks of the increased deposition of NPs into the soil environment, we have studied the sorptive interactions of ZnO-NPs with a colloid fraction that was extracted from agricultural soil. Our initial results indicated contribution of both homo- and hetero-aggregation on the ZnO-NPs distribution in soil colloid. The obtained results can be helpful for better understanding of ZnO-NPs' behavior in soil environment.

Keywords: zinc oxide nanoparticles, size fractionation, soil colloids

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Ca-palygorskite clay mineral for Fe^{2+} removal from water systems from molecular simulations point of view

C.V. Lazaratou[1], D. Panagiotaras[2], G. Panagopoulos[3], M. Pospíšil[4], D. Papoulis[1]

[1]Department of Geology, University of Patras, Greece; [2]Department of Environment, Ionian University, Greece; [3]Department of Mechanical Engineering, University of the Peloponnese, Greece, [4]Faculty of Mathematics and Physics, Charles University, Czech Republic.

lachrist93@yahoo.gr

Palygorskite fibers were used for the testing of Fe (II) uptake from aqueous solutions under various experimental conditions. Palygorskite samples were characterized by following methods: X-ray diffraction, Fourier transform infrared spectroscopy, scanning electron microscopy, cation exchange capacity and molecular simulations. The fully saturated Ca-palygorskite samples were characterized as well and applied in batch kinetic experiments series.

It was shown that Ca-palygorskite was more efficient adsorbents for the lowest ferrous concentrations removal (5 mg/L). Especially when the highest solid: liquid ratio (20 g/L) have been applied, Ca-palygorskite reach 99.8% removal within 10 min at the optimal room temperature (20 ± 1 °C). The pH value affected the adsorption’s efficiency, as Ca-palygorskite was more efficient adsorbent at acidic values (4–6). The competitive ions found to decrease Ca-palygorskite capacity for Fe (II), following the order K<Na=Mn<Zn<Cu<Mg<Ba, highlighting their preference to a Fe cations. According to the thermodynamic analysis the Fe (II) uptake is a physical and spontaneous process, Ca-palygorskite was fitted by Langmuir isotherm indicating the monolayer sorption. The exact mechanism of Ca-palygorskite efficient adsorption in detail was described in detail by molecular simulation methods in Materials Studio 4.3. modelling environment.

Graphitic carbon nitride prepared from different precursors for photocatalytic degradation of pollutants in water

A. Smýkalová[1,2], K. Foniok[1], P. Praus[1,2]

[1]Department of Chemistry, VŠB-TUO, Czech Republic, [2]CEET, Institute of Environmental Technology, Czech Republic

aneta.smykalova@vsb.cz

Graphitic carbon nitrides from mixtures of guanidine hydrochloride and melamine or guanidine hydrochloride and dicyandiamide with different weight ratios were synthetized. Obtained materials were characterized using UV/Vis diffuse reflectance spectroscopy, X-ray powder diffraction and Fourier transform infrared spectroscopy. The specific surface area and elemental composition (C, H, N) were also measured. Photocatalytic activity of prepared materials was tested on Rhodamine B, Phenol and Amoxicillin. The photocatalytic experiments on all three pollutants showed a maximal activity for one sample with a specific ratio of precursors from each series. The specific surface area measurement of both sample series also showed the maximum correlating to the photocatalytic activity of samples. In order to understand the increase and decrease of photodegradation activity and specific surface area of materials more measurements and characterizations had to be carried out, for example, thermal analysis.

Experimental verification of regenerable magnetically modified montmorillonite and its application for heavy metal removal from metallurgical waste leachates

M. Tokarčíková[1], O. Motyka[1], O. Životský[2], R. Gabor[1], J. Seidlerová[1]

[1]Energy and Environmental Technology Centre, Nanotechnology Centre, VŠB-Technical University of Ostrava, Czech Republic; [2]Department of Physics, VŠB-Technical University of Ostrava, Czech Republic

Michaela.tokarcikova@vsb.cz

Sorbents with magnetic properties seem to be an efficient, easy to apply and low-cost technique for heavy metals elimination from aqueous solutions. Most of the studies have been focused on eliminating pollutants only from model solutions, while the application to real wastewaters has been missing. In this study, the metallurgical waste leachates were prepared, and magnetically modified montmorillonite (MMt) was applied for Zn(II), Cd(II) and Pb(II) elimination. In the first step, the sorption properties of MMt, sorption kinetic and isotherm models were determined from experiments obtained from the model solutions. The data obtained from metallurgical waste leachates corresponded well with the data obtained from model solution experiments. The elimination efficiency of Zn(II), Cd(II) and Pb(II) corresponded with the initial heavy metals concentration. The reusability of MMt was studied by the fifth adsorption/desorption cycle. The elimination of Zn(II), Cd(II) and Pb(II) was higher than 98 % even after the fifth cycle, and the MMt was still magnetically active. The magnetic properties were verified by a magnet and determined using a vibrating sample magnetometer. MMt proved to be an efficient and regenerable sorbent, applicable for wastewater treatment.

Keywords: Magnetic montmorillonite, Metallurgical waste leachates, Sorption, Recycling

Acknowledgments: The work was supported by ERDF/ESF New Composite Materials for Environmental Applications (No. CZ.02.1.01/0.0/0.0/17_048/0007399).

Assessment of metal nanoparticles accumulation in organs and their effect on cognitive abilities of mice

Inga Zinicovscaia[a,b*], Alexandra L. Ivlieva[c], Elena N. Petritskaya[c], Dmitriy A. Rogatkin[c], Nikita Yushin[a], Dmitrii Grozdov[a], Konstantin Vergel[a], Kateřina Mamulová Kutláková[d]

[a]Joint Institute for Nuclear Research, str. Joliot-Curie 6, Dubna, Moscow Region, 141980, Russia, [b]Horia Hulubei National Institute for R&D in Physics and Nuclear Engineering, 30 Reactorului Str., MG-6, Bucharest - Magurele, Romania, [c]Moscow Regional Research and Clinical Institute named after M. F. Vladimirsky, str. Schepkina 61/2, Moscow,129110, Russia, [d]Nanotechnology Centre, VŠB-Technical University of Ostrava, 17. listopadu 2172/15, 708 00 Ostrava-Poruba, Czech Republic

zinikovskaia@mail.ru

Because of good optical, electrical ant antimicrobial properties, silver and TiO_2 nanoparticles are widely used in many fields of industry and medicine. To assess the effect of silver and TiO_2 nanoparticles on mice cognitive abilities and their accumulation in different organs, daily, up to 2-month period, experimental mice were administrated with silver or TiO_2 nanoparticles solution. Accumulation of silver and titanium in blood, brain, liver, kidney, lungs and feces was assessed by

neutron activation analysis. According to obtained data silver nanoparticles are able to reach and cross the placental barrier and blood-to-brain barrier in both mice female and their offspring. In mice female the highest silver concentration was determined in lungs, followed by brain, liver, kidney and blood. In offspring silver bioaccumulation changed in the following order lungs> brain> blood> liver>kidney. The average specific mass content of silver which crossed the blood-brain barrier was 373±75 ng (for female) and 385±57 ng (for offspring). The obtained results are important for studies on developmental and reproductive toxicity of nanomaterials. The amount of titanium in all organs was below the limits of detection of the method and in feces it constituted 74±7 µg/g.

Cognitive abilities in mice before and after metal nanoparticles administration were evaluated in the Morris water maze behavioral test. In the main Morris water maze behavioral test at the control points statistically significant differences were found in the parameters of treks between capable experimental and control animals. The obtained results are important for studies on developmental and reproductive toxicity of nanomaterials.

Poster presentation (PP):

Sorption study on diclofenac removal using organo-beidellite

E. Plevová[1], S. Vallová[2,3], L. Vaculíková[1], V. Valovičová[1], K. Smutná[2,3], M. Hundáková[4], S. Dolinská[5]

[1]Institute of Geonics of the Czech Academy Sciences, The Czech Republic; [2]Institute of Environmental Technology CEET, VSB-Technical University of Ostrava, The Czech Republic; [3]Department of Chemistry, VSB-Technical University of Ostrava, The Czech Republic; [4]Nanotechnology Centre, VSB-Technical University of Ostrava, The Czech Republic; [5]Institute of Geotechnics of the Slovak Academy of Sciences, The Slovak Republic

eva.plevova@ugn.cas.cz

The aim of this study is the assessment of ability of organo-beidellite for the removal of drugs from aqueous solutions due to increasing wastewater pollution related to high consumption of pharmaceuticals. The beidellite (BEI), often neglected clay mineral of smectite type, was modified with cetylpyridinium chloride (CP), and prepared BEI-CP was checked as potential sorbent for analgesic drug diclofenac (DC) from aqueous solutions. The BEI-CP samples were characterized by X-ray powder diffraction (XRD), simultaneous thermogravimetry and differential thermal analysis (TG/DTA) and infrared spectroscopy (FT-IR) along with UV-VIS spectroscopy. The enhancement of the basal spacing obtained by X-ray diffraction confirmed the intercalation process of organic substance into beidellite. Also the infrared spectra provided evidence of sorption and intercalation of CP. The results obtained by TG/DTA showed that CP intercalation caused decrease in dehydration and increase in dehydroxylation temperatures due to an increase of the hydrophobicity of modified beidellite. The sorption experiments exhibited the increasing of sorption capacity related to the increasing of initial concentration of DC in solution. The removal of DC from aqueous solution reached up to 96%. The sorption characteristics including the sorption isotherms were determinated too.

Keywords: beidellite, cetylpyridinium chloride, diclofenac, adsorption

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Application of sorbents modified by nanoparticles

J. Seidlerová[1,2], M. Tokarčíková[1], R. Gabor[1]

[1]Nanotechlology Centre CEET, VŠB-TUO, Czech Republic; [2]Department of Physical Chemistry and Theory of Technological Processes, Faculty of Materials Science and Technology, VŠB-TUO, Czech Republic

jana.seidlerova@vsb.cz

Phyllosilicates are a promising group of materials since they are native, non-toxic, and applicable in numerous industrial sectors. Phyllosilicates can be used as carriers of photoactive, degradable,

antibacterial, or fungicidal nanoparticles. They are efficient adsorbents of non-degradable pollutants due to their (e.g.) high specific surface area, chemical and mechanical stability, various structural and surface properties, higher cation exchange capacity. On the other hand, sorbent separation can cause substantial technological problems, mainly when the sorbent particles are of a very small size. However, the adsorbent can be modified by magnetically iron oxide micro/nanoparticles to facilitate removing the adsorbents from the solution – by using a magnet. The magnetically modified materials were prepared from montmorillonite, kaolinite, vermiculite, and Ghassoul (a unique mixture of stevensite and sepiolite) using the microwave-assisted method of magnetic particle preparation. The adsorption of Be, Cd, Ce, Pb, Sr, Tl, and Zn ions and selected organic compounds from water solution were studied. The experimental data were fitted by adsorption isotherms (Langmuir and Freundlich) and pseudo-first or pseudo-second kinetic model. The results proved that magnetic iron particles bonded strongly with the surface of the phyllosilicates and did not negatively affect the adsorbed amount of ions.

Keywords: Adsorption, modified phyllosilicates, remove pollutants

TOPIC 6

Industrial Forum

Oral presentations (OP):

Nanoscale Investigation of Multilayer Graphene Flakes using Contactless Electrical Modes of Tosca Atomic Force Microscope

Gregor Plohl

Anton Paar Gmbh / Austria

support-nsp@anton-paar.com/ www.anton-paar.com

ABSTRACT

Graphene is a 2D material with promising physical and electronic properties and has been widely studied because of its potential applications in next-generation electronics, photovoltaics and life science. The contactless electrical modes of atomic force microscope Tosca were used to accurately characterize the material and functional properties of graphene flakes on nanometer scale.

Keywords: AFM, KPFM, EFM, two pass technique, work function, surface potential microscopy, thin films

INTRODUCTION

Graphene was discovered, isolated and characterized by A.K Geim and K.S. Novoselov in 2004 (1). They were able to isolate a monolayer of graphene from a piece of graphite by using an ordinary Scotch tape. The authors received the 2010 Nobel Prize in Physics for "*groundbreaking experiments regarding the two-dimensional graphene*" (2).

Graphene is made of sp^2 – hybridized carbon atoms arranged in a one-atom-thick hexagonal (honeycomb) lattice structure (3), as shown in Figure 1. This two-dimensional (2D) material is a basic building block for all graphitic materials of other dimensionalities; it can form a zero-dimensional (0D) quantum dots, it can be rolled into one-dimensional (1D) nanotubes or stacked to form three-dimensional (3D) graphite.

Graphene has remarkable mechanical, electrical, magnetic and thermal properties. It is one of the thinnest (thickness of 0.345 nm), strongest (about 100 times stronger than steel) and lightest material known to exist with excellent electrical and thermal conductivity. Due to its unique electronic characteristics, graphene has been viewed as a promising candidate to replace conventional silicon-based electronics. Moreover, graphene has an enormous potential in many applications such as semiconductors, photovoltaics, life science, energy storage, printable transparent electronics and many others.

Nevertheless, the widespread implementation of graphene is, overall, still in the research and development stage. Therefore, there is a great scientific interest for an in-depth understanding and characterization of various physical properties of graphene-based materials on a nanoscale. The advanced modes of atomic force microscopy (AFM) are powerful tools which enable a great progress in the field of graphene related research. This report demonstrates the application of contactless electrical modes, namely the electrostatic force microscopy and Kelvin probe microscopy of Tosca atomic force microscope for nano-characterization of material functional properties of graphene flakes.

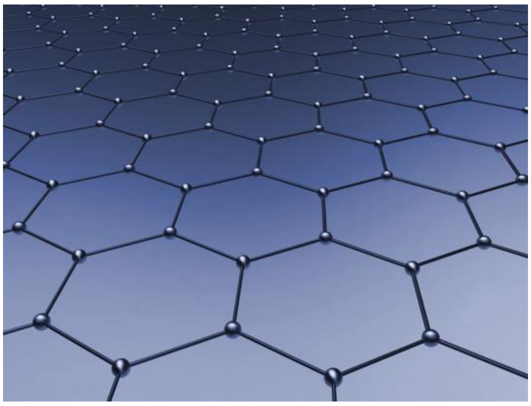


Fig. 1. Hexagonal lattice structure of graphene made of carbon atoms

EXPERIMENTAL/THEORETICAL STUDY

Electrical modes are techniques derived from atomic force microscopy (AFM) used to measure local electric properties together with the topography of the sample at nanoscale. First measurements of the contact potential difference between different materials using AFM were reported by Nonnenmacher, O'Boyle and Wickramasinge in 1991 (4) shortly after invention of AFM (5). They simultaneously measured the topography and contact potential difference of gold, platinum and palladium surfaces. Electrical modes require a conductive cantilever and an electrical contact between the sample and sample table must be established. No special sample preparation is necessary and the measurements can be performed under ambient conditions.

The Kelvin probe force microscopy (KPFM) and electrostatic force microscopy (EFM) record the long-range electrostatic forces and provide information about electric properties such as contact potential difference, electric surface potential, work function and electric charge distribution.

Both EFM and KPFM can operate in two different ways, known as single pass and two pass techniques. Single pass technique records the topography and the surface potential concurrently, while the traditional two pass techniques requires two scanning passes. Figure 2 schematically shows the principle of the two pass technique.

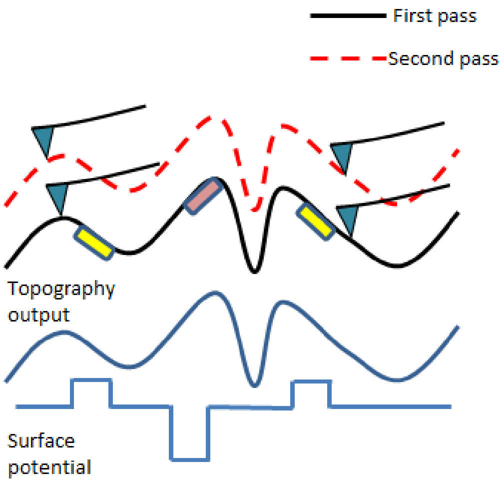


Fig. 2. Two pass technique AFM.

The two-pass technique, implemented in Tosca atomic force microscope, uses the first pass to record the surface line profile by tapping mode. Then the cantilever is lifted to a predefined distance above the surface, the so-called lift offset, and follows the recorded surface line profile to measure the signal of long-range electrostatic forces in the second pass. This two-pass technique effectively minimizes crosstalk from topographic effects.

Tapping mode is a form of dynamic imaging. In general, the dynamic imaging mode oscillates the cantilever near its resonance frequency. The interaction forces (either mechanical, van der Walls, electric or magnetic) between the probe and sample produce a frequency shift of the resonance frequency, amplitude and phase between excitation and oscillation of the cantilever. This variation in the cantilever oscillation behavior provides information about the probe-sample interaction forces. The changes can be monitored either by amplitude modulation (AM) or frequency modulation (FM) techniques and used to reconstruct height and other physical properties. Tapping mode uses the AM detection technique, where the cantilever oscillation amplitude is kept constant via a feedback loop.

Electrostatic Force Microscope (EFM)

Electrostatic force microscopy maps the local electric potential and charge distribution by probing the electrostatic force between the biased tip and the sample.

During the lift pass an external bias voltage is applied between the cantilever tip and the sample while the cantilever continues to oscillate at the tapping frequency. As the cantilever tip scans over a surface, its oscillation is affected by the electrostatic forces. Measured amplitude and phase shifts reveal the electric potential distribution on the sample surface. EFM provides qualitative information about electric field variations and is used for electrical failure analysis, detecting trapped charges, mapping electric polarization, etc.

Kelvin Probe Force Microscope (KPFM)

While the EFM mode is used mainly for the qualitative measurements of surface potential, the KPFM mode provides the quantitative mapping of the contact potential difference:

$$V_{CPD} = \frac{\varphi_{tip} - \varphi_S}{e} \qquad (1)$$

where e is the elementary charge and φ_{tip} and φ_S are the work functions of the probe and sample, respectively. The work function is the minimum energy needed to remove an electron from a solid surface.

In contrast to EFM, the cantilever in lift pass of KPFM is no longer excited mechanically but electrically by applying an external voltage V_{ext} between the tip and the sample as

$$V_{ext} = V_{DC} + V_{AC} \sin(\omega_{mod} t) \qquad (2)$$

where and ω_{mod} is the modulation frequency, and V_{DC} and V_{AC} are amplitudes of DC and AC components, respectively. The modulation frequency is (usually) selected to be equal to the resonant frequency of the AFM cantilever. The electrostatic force between the sample and the tip is given as

$$F_e(Z) = -\frac{1}{2}\,(\Delta V)^2\,\frac{dC}{dz} \qquad (3)$$

where C denotes the capacitance between the tip and the sample, z is the tip-sample distance and $\Delta V = V_{ext} - V_{CPD}$ is the potential difference between the tip and the sample. The feedback electronics, based on AM detection mechanism, adjusts the bias voltage V_{DC} until the oscillation amplitude at the modulation frequency ω_{mod} is nullified, thus $V_{DC} = V_{CPD}$.

Further, the work function of the sample φ_S can be calculated using Eq. (1) when the tip work function φ_{tip} is known. The work function influences many surface phenomena (adsorption, charge transfer, photoelectric activity, catalytic activity, etc.) and is a critical parameter in solid-state electronics. Therefore, KPFM is often used to study metallic and semi-conducting nanostructures in a variety of devices from biosensors to solar cells (7).

The exfoliated graphene flakes deposited on a silicon wafer were investigated by Tosca atomic force microscope under ambient conditions. An acoustic enclosure and active vibration table isolated the AFM device from surrounding vibrations. The measurements were performed using a standard Arrow-EFM silicon AFM cantilever with platinum iridium coating. The cantilever has a typical resonance frequency of 75 kHz and a force constant of 2.8 N/m.

Graphene flakes of various shapes and surface areas were deposited on a silicon wafer. The flake thickness ranges from one graphene layer to some hundreds of nm. Figure 3 shows a microscopic image of the deposited multilayer graphene flakes.

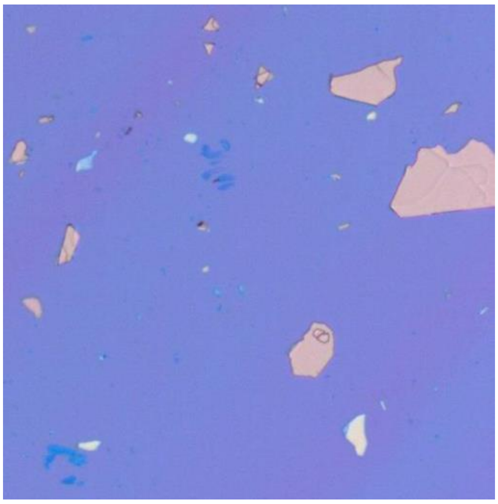


Fig. 3. Optical image (1000 x 1000 μm) of the exfoliated graphene flakes on a silicon wafer.

RESULTS AND DISCUSSION

The AFM investigation of graphene flakes was performed on a 15 x 15 μm area with a resolution of 750 x 750 pixels and a scan rate of 0.5 line/second. Both electric modes, EFM and KPFM, were employed at the same position.

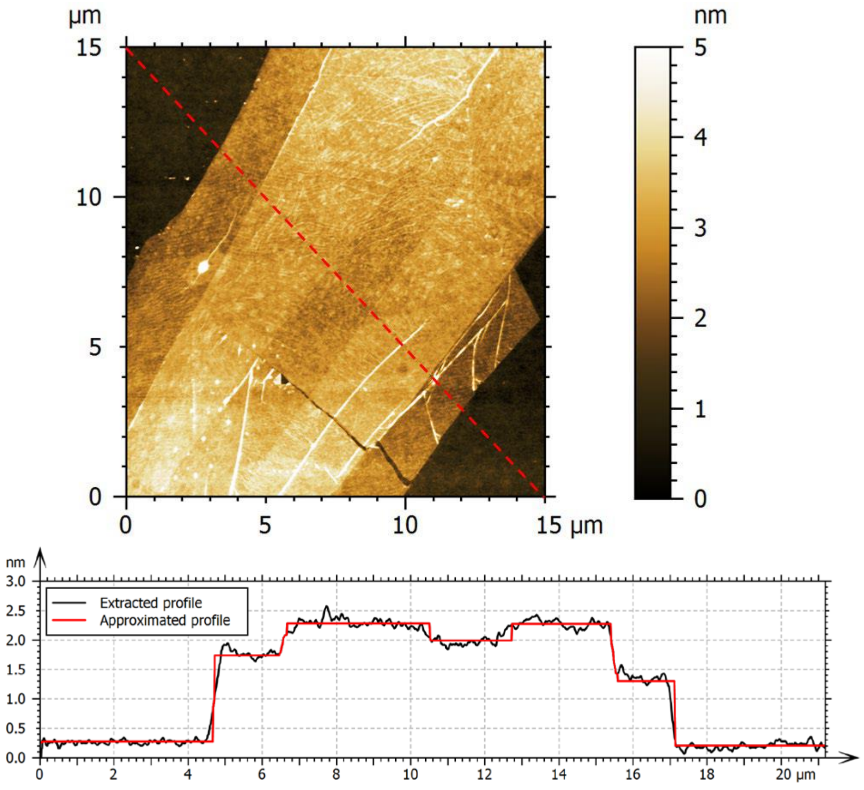
Topography

The topography data was recorded during the first pass of the two-pass technique by tapping mode. The same topographic results were obtained by both methods, EFM and KPFM. In addition to the height profile, the tapping mode also provides the phase contrast of the surface, which is related to the mechanical interaction between the tip and the sample and thus represents the distribution of the mechanical properties of the surface. Hence, the phase contrast data allows distinguishing regions with different mechanical properties.

Figure 4 shows the results of tapping mode investigations of the 15 x 15 μm scan area. The topographic image shows elevated graphene structures on a flat silicon wafer. Overall, the graphene flake has a very smooth surface with some residues, presumably coming from the graphene synthesis process.

Figure 4 (middle) shows the 2D height profile extracted from the topography data presented in Figure 4 (top). The investigated graphene flake is composed of about 3 to 6 layers of graphene and reach up to around 2 nm in height. The height difference of a single graphene layer, which is about 0.345 nm, is clearly visible.

The phase contrast image, Figure 4 (bottom), clearly displays a two phase separated structures. From the knowledge about material properties of graphene and silicon it may be concluded that the brighter regions correspond to the stiffer graphene flakes and darker regions to silicon wafer. As expected, the phase contrast does not depend on the thickness of the graphene flakes.



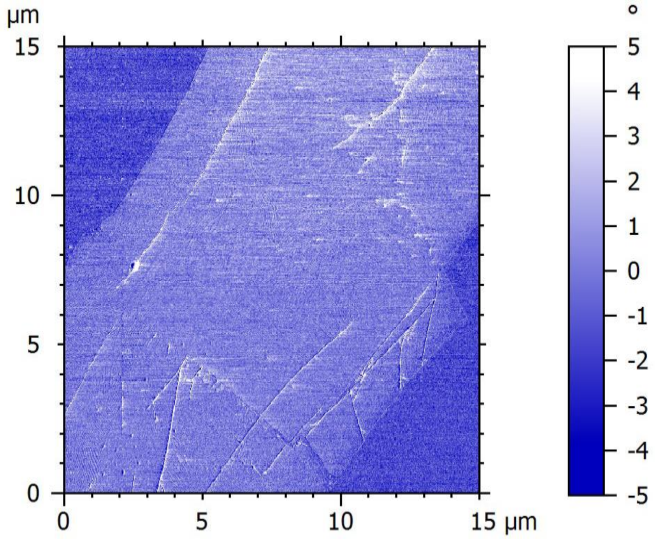


Fig. 4. Results of the tapping mode investigations of graphene flakes. The height image (top), where the red line indicates the position of the extracted 2D height profile shown in the middle. Phase contrast image (bottom) indicates two mechanically different materials.

EFM results

EFM method provides qualitative information about electric field variations by applying an external bias voltage, in the range from -10 V to 10 V, between the tip and the sample.

Figure 5 presents the results of EFM mode measurements performed at the lift offset of 15 nm and DC bias voltage set to 8 V.

A 3D superposition of the EMF phase signal and the topography is shown in Figure 5 (top). A highly contrasted, stepwise phase shift at silicon-graphene transition can be observed. On the surface of a single graphene flakes practically no phase shift between different height levels of graphene layers can be observed. These findings are confirmed by analysing the extracted 2D EFM phase profile shown in Figure 5 (bottom).

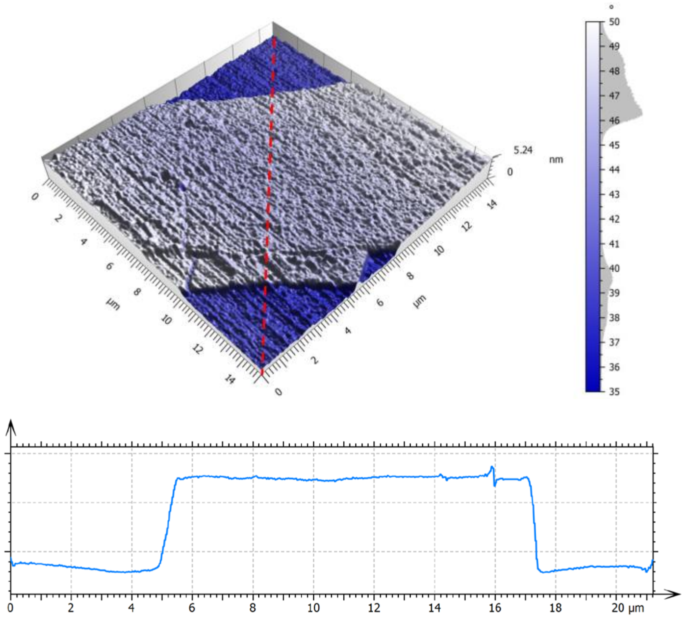


Figure 5. Results of EFM mode investigations of graphene flakes. Superposition of 3D topography and EFM phase signal (top), where the red line indicates the position of the extracted 2D phase profile shown below. A sharp, stepwise phase shift at the silicon-graphene transition can be observed.

KPFM results

The KPFM signal was obtained in the lift pass with the lift offset of 15 nm and AC excitation amplitude to 9 V. The results of KPFM investigations are presented in Figure 6.

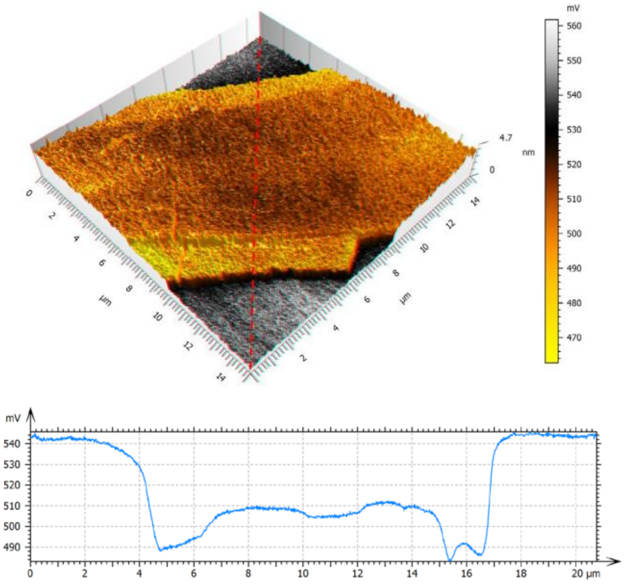


Figure 6. Results of KPFM mode investigations of graphene flake. Superposition of 3D topography and contact potential distribution (top), where the red line indicates the position of the extracted 2D profile shown below. The electric potential difference between graphene and silicon depends on the flake thickness and lies between 30 and 60 mV.

Figure 6 (top) presents a 3D superposition of the contact potential distribution and the topography, while Figure 6 (bottom) shows the extracted 2D profile of the contact potential difference.

By analyzing the KPFM results, similar conclusion can be made as in the case of EFM investigation. The data obtained by KPFM confirms the findings of EFM method and adds the quantitative aspects. KPFM resolves the contact potential difference on sub-nm scale to precisely distinguish graphene layers of around 0.35 nm.

The electric potential difference between the graphene flakes and silicon wafer is between 30 and 60 mV and shows to depend on the graphene flake thickness. Higher contact potential difference corresponds to the thinner graphene flakes and vice versa. This is in agreement with literature (7) where it has been shown that increasing the number of layer of graphene can eventually shield completely the substrate potential.

CONCLUSION

This short section should summaries the work based on the facts in evidence and point out the main areas covered in the writing. It may also contain the future directions of the work.

The electric modes of the atomic force microscope Tosca were used to image nanoscale topographic and electrostatic properties of multilayer graphene flakes. The topography measurements were able to resolve single layers of graphene in the composition of the examined graphene flake. The electrostatic force microscopy (EFM) provides qualitative information about local electric potential by detecting the electrostatic force between the biased tip and the sample.

In contrast, the Kelvin probe force microscopy (KPFM) provides quantitative information about contact potential difference by using additional feedback control. Further, KPFM was able to precisely distinguish single graphene layers on a sub-nanometer scale due to the contact potential difference. The contact potential difference is related to the material work function which can be determined when the tip work function is known.

Acknowledgments: The author would like to express his gratitude to colleagues in NSP team in Graz (Austria) for their invaluable help.

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Surface zeta potential analysis of novel graphene oxide coatings for membranes

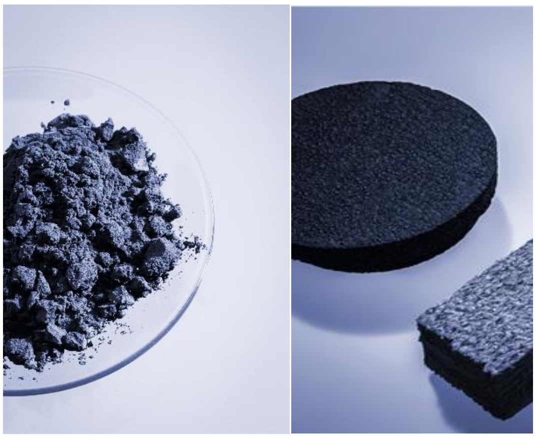
T. Luxbacher[1], R. Kohl[1]

[1]Anton Paar GmbH, Austria

pc-application@anton-paar.com

Relevant for: graphene, graphene oxide, membranes, surface charge, zeta potential

2D materials such as graphene and graphene oxide introduce new pathways for the development of novel materials with superior properties. Nanocomposite coatings containing graphene oxide are investigated for their capability to enhance the performance of membranes for water treatment applications. We use the analysis of the zeta potential to confirm the presence of graphene oxide at the surface of such nanocomposite membrane coatings, which is a requirement for their activity and interaction with the aqueous environment.



INTRODUCTION

The discovery of graphene has opened completely new technological pathways in material science. Nowadays graphene and its derivates can be found in a large number of applications due to their outstanding properties. Such applications can be found in the fields of electronics and batteries but also in biomedical applications including drug delivery, biosensing, bioimaging, and scaffolds for tissue engineering.

The hydrophilic nature of graphene oxide makes this graphene derivative also attractive for the advancement of membrane technology. Graphene oxide is expected to increase the water permeability, to suppress membrane fouling, and to introduce a reinforcement of polymer membranes.

We report on the modification of a polymer membrane by a graphene oxide-polyelectrolyte composite coating and its effect on the charging behaviour at the membrane-water interface. A second example considers the formation of a ceramic membrane composed of graphene oxide and aluminium oxide.

The characterization of the surface and interfacial charge of polymer and ceramic membranes employs the measurement of the streaming potential for the calculation of the surface zeta potential.

Graphene oxide composite membranes

Graphene with its two-dimensional planar structure increases the mechanical strength, the thermal stability and the electrical properties of composites, and has thus received attention as a suitable candidate for the development of membranes for water treatment processes. In contrast to graphene, graphene oxide (GO) exhibits an increased hydrophilicity due to the oxygen-containing functional groups (C=O, C–OH). The latter lead to a better dispersion of GO in water and other polar solvents thereby maintaining the superior properties of graphene. The amphiphilic character of GO makes it compatible with polymers, which are used as the base material in the low-cost membrane production process, and enhances the water permeability and the antifouling properties of membranes Additionally, graphene and GO are low-cost alternatives to carbon nanotubes (CNT) or carbon nanofibers (CNF).

These properties make GO a potential modifier for polymer but also ceramic membranes for wastewater treatment processes.

Membranes and the zeta potential

The zeta potential is a solid-water interfacial parameter and representative for the material surface charge. The zeta potential helps to predict electrostatic interactions between material surfaces and solutes in a surrounding aqueous solution. Furthermore, the analysis of the zeta potential is a sensitive method to detect changes in the surface chemistry. In the field of membrane applications, the zeta potential contributes to the rejection of solutes and to membrane fouling. With the help of zeta potential analysis, it is possible to determine the coverage of the membrane surface by a thin-film coating and the stability of such coating.

EXPERIMENTAL

Two types of nanocomposite coatings containing graphene oxide where prepared as potential candidates for novel membrane materials. A multilayer coating with alternating layers of GO and the cationic polyelectrolyte polyethylenimine (PEI) was deposited on a flat sheet polymer ultrafiltration membrane. A second nanocomposite coatings was of ceramic nature and composed of GO and nanocrystalline aluminium oxide (Al_2O_3).

The instrument SurPASS 3 was used for the zeta potential analyses of flat sheet membranes with different GO nanocomposite coatings. Small sample pieces of 20 mm x 10 mm were adhered to the sample holders of the Adjustable Gap Cell with the active coating facing upwards (Figure 1).

Streaming potential measurements were performed with an aqueous 0.001 mol/l KCl solution used as the background electrolyte. The pH dependence of the zeta potential was recorded automatically with the integrated dosing unit to determine the isoelectric point (IEP, pH value where the zeta potential is 0 mV and a charge reversal takes place) of each GO coating.

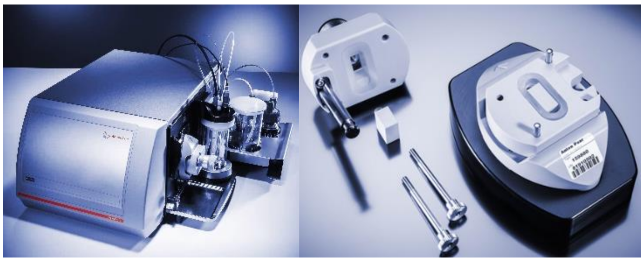


Figure 1: Left: SurPASS 3 equipped with dosing unit. Right: Adjustable Gap Cell with sample holders with a cross-section of 20 x 10 mm^2.

RESULTS

GO coating on a polymer membrane

The combination of GO nanoparticles with the cationic polyelectrolyte poly(ethylene imine), PEI, was used to improve the rejection of a polyacrylontrile membrane (PAN) for ultrafiltration (UF) thereby maintaining the high water permeability [1]. Figure 2 shows the pH dependence of the zeta potential for such a PAN UF membrane with a GO│PEI coating deposited by a layer-by-layer method. Above the IEP at pH 3.3, the coating is negatively charged with the zeta potential approaching $\zeta = –40$ mV at neutral and alkaline pH.

In order to interpret the observed zeta potential as a representative for the surface charge of the GO│PEI coating and to understand the composition of this coating, Figure 2 also displays the zeta potential for a pristine PAN membrane, for the PAN membrane coated solely with PEI, and for a membrane coated with a stand-alone film of GO.

We find significant differences in the zeta potential for each of these membrane samples. Although the zeta potential and therefore the electrokinetic charge density σ_{ek} of the pristine and GO│PEI-coated PAN membranes are similar at pH 8-9 ($\zeta \sim$ -40 mV, $\sigma_{ek} \sim$ -0.34 $\mu C/cm^2$), the pH dependences and the isoelectric points give evidence for the modification of PAN by the GO│PEI coating.

The sole coating of the PAN membrane by the cationic polyelectrolyte PEI shifts the IEP from pH 4.6 for the pristine PAN membrane to pH 8.9. The seconday amine groups of PEI get protonated at low and medium pH. Due to their strongly basic character, rather highly alkaline conditions are required to suppress this protonation. At pH 8.9 of the bulk aqueous solution we find an electroneutral condition at the membrane-water interface and thus the IEP for the PEI-coated PAN membrane. Since the IEP for the GO│PEI-coated membrane is shifted to even lower pH compared to the IEP of PAN, we conclude that PEI obviously does not contribute to the charge at the outermost surface of the GO│PEI coating.

A comparison of the zeta potential for the GO│PEI-coated PAN membrane with the zeta potential for a stand-alone membrane of graphene oxide reveals the apparent nature of the GO│PEI coating, which is dominated by the presence of GO. Obviously the layer-by-layer coating achieves a well separated stack of polymer (PEI) and GO layers with GO determining the terminal layer.

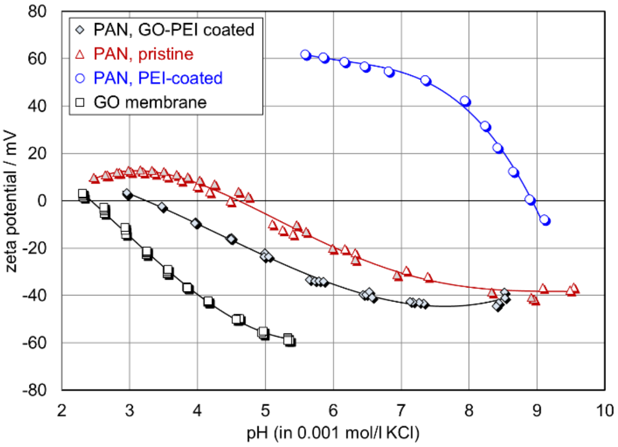


Figure 2: pH dependence of the zeta potential for a pristine polyacrylonitrile (PAN) membrane for UF, for the PAN membrane coated with a multilayer of GO and PEI, for a PEI- coated PAN membrane, and for a GO membrane.

GO /Al_2O_3 composite membrane

In another example we use the same zeta potential results for a membrane coated with a stand-alone porous film of GO shown in Figure 2 to explain the composition of a composite layer of GO and Al_2O_3. Figure 3 shows the pH dependence of the zeta potential for such a GO|Al_2O_3 coating and compares this result with the zeta potential of the GO coated membrane and with the zeta potential obtained for a single-channel tubular alumina ceramic membrane for microfiltration (MF). While all measurements of the streaming potential for flat membrane surfaces employ the Adjustable Gap Cell, the zeta potential analysis of the microporous ceramic membrane utilizes the measuring cell for tubular membranes [2].

The IEP 5.2 of the GO│Al_2O_3 composite layer is found in between the IEP 2.4 for the GO stand-alone film and the IEP 6.5 for the alumina MF membrane. To conclude on the composition of the GO│Al_2O_3 layer, we first elucidate the zeta potential of the Al_2O_3 ceramic membrane. For this purpose, we add another dependence of the zeta potential on the pH of the aqueous KCl solution, which was obtained for a sample plug of corundum powder. The difference in the IEPs of $\Delta pH = 1.5$ for corundum powder and the alumina membrane may be explained by an ageing effect for the membrane surface. Note that the surface of Al_2O_3 exhibits a rather complex behaviour and the IEP may range from pH 4 for single-crystalline sapphire [3] to pH > 9 for Al_2O_3 nanoparticles in dispersion [4].

For the interpretation of the composition of the outermost surface of the GO│Al_2O_3 layer, we refer to the IEP 8 of corundum powder and to the IEP 2.4 of the purely GO membrane. Based on the comparison of these materials and their IEPs, we conclude on a homogeneous composition of the layer with a contribution of 50% by GO and 50% by Al_2O_3.

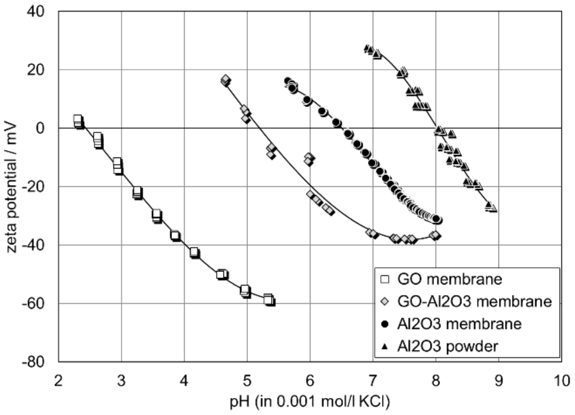


Figure 3: pH dependence of the zeta potential for a GO|Al_2O_3 nanocomposite coating, a stand-alone film of GO on a membrane, and two samples of Al_2O_3 (a single-channel tubular ceramic MF membrane and corundum powder).

SUMMARY

New generation 2D materials such as graphene oxide offer a high potential for the development of novel materials with advanced properties. The tunability of GO makes it a promising candidate for the modification of membranes for water treatment to improve the water transport and to suppress membrane fouling. The transfer of knowledge about the correlation of the surface zeta potential with salt rejection and the fouling propensity of (primarily) polymer membranes, which was developed in the past decades, to the characterization of novel coatings containing GO is straightforward. This report illustrates results for graphene oxide and GO-containing surface coatings for both polymeric and ceramic films. The IEPs summarized in Table 1 demonstrate the variability of surfaces prepared by using GO as a composite material. A comparison of the IEP of the composite material with the IEPs of the individual components enables an estimation of the surface composition. The derived contribution of GO of the composite layers to the zeta potential is also shown in Table 1.

Table 1: Isoelectric points of materials discussed in this report and surface contribution of GO to composite layers

Sample	IEP	GO contribution
GO membrane	2.4	100 %
PAN membrane	4.6	n/a
GO│PEI LbL-coated PAN	3.3	85 %
PEI-coated PAN	8.9	n/a
GO-Al_2O_3 composite	5.2	50 %
Al_2O_3 MF membrane	6.5	n/a
Corundum powder	8	n/a

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Boosted research of nanomaterials by XPS and HPXPS

Lukasz Walczak

Science & Research Division, PREVAC sp. z o.o., Rogow, Poland

l.walczak@prevac.pl

Many important processes such as energy conversion, electrochemical, corrosion and biological processes take place at solid-gas and solid-liquid interfaces [1-3]. X-ray photoelectron spectroscopy (XPS) is the most powerful technique for understanding of these processes at the atomic level and it is the key to improving the performance of novel battery generation or renewable energy sources such as solar, wind or hydropower energy conversion devices. We would like to promote the latest equipment, technology solutions and innovations for the photoelectron spectroscopies field. Here it will be presented an innovative and compact spectrometers for the routine X-ray photoemission spectroscopy and ambient pressure photoemission spectroscopy for study of important phenomena in the current research. The design, construction, and technical parameters of new analyser will be presented. We will report the research results of XPS measurements conducted on the photovoltaic [4], catalytical [5] or bio-materials. Moreover, it will be demonstrated the analyser operation in ambient pressure. Also the results of interaction of oxygen and surface alloy will be presented, in order to permit complete characterization for the different pressure and temperature conditions.

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Infrared (IR) Chemical Identification at the Nanoscale – When AFM meets IR –

Miriam Unger and Hartmut Stadler

Bruker Nano Surfaces and Metrology, 76187 Karlsruhe, Germany

Miriam.Unger@bruker.com

This presentation will overview atomic force microscope based infrared spectroscopy (Photothermal AFM-IR). The technique is based on the combination of a tunable infrared laser with an atomic force microscope that can locally map and measure thermal expansion of nanoscale regions of a sample resulting from the absorption of infrared radiation. The photothermal AFM-IR technique uses the tip of an AFM as a nanoscale detector of absorption of IR radiation. Therefore, AFM-IR can be used to obtain IR absorption spectra and chemical imaging with resolution as fine as the AFM tip radius, >100X smaller than spatial resolution limits of conventional infrared spectroscopy. One of the key benefits is that AFM-IR directly correlates to FT-IR transmission spectroscopy which makes it possible to use existing IR libraries for chemical identification at the nanoscale.

In the presentation we will introduce the underlying technology and highlight numerous applications ranging from polymer characterization, life and material sciences.

Nanomechanical Characterization of Battery Materials

Jaroslav Lukeš

Bruker Nano Surface and Metrology, Prague, Czech Republic

jaroslav.lukes@bruker.com

The storage and portability of energy is important. Currently, lithium ion is the most commonly used technology for rechargeable batteries but, nickel-metal and lead-acid are also utilized. However, better performance in terms of both cost and weight can be realized by moving to solid state batteries. Failure analysis allows scientists to construct longer lasting, better batteries.

Typical failure modes of batteries can be mechanical, such as thermal or electrical failure. Understanding the mechanical properties, at the length scales of interest, is imperative to the manufacture of safe, reliable, and cost-effective batteries.

We will use nanoindentation and particle compression to investigate the mechanical behavior of several battery materials. These include materials for conventional lithium-ion batteries, such as coated anodes and nickel manganese cobalt cathode particles as well as their solid-state counterparts.

Correlative Probe and Electron Microscopy using AFM-in-SEM

Veronika Hegrova[1*], Zdenek Novacek[1], Michal Pavera[1], Jan Neuman[1]

[1]NenoVision s.r.o., Purkynova 649/127, 61200, Brno, Czech Republic

conference@nenovision.com

Scanning electron microscopy (SEM) and atomic force microscopy (AFM) are two of the most used, complementary techniques for surface analysis at the nanoscale. Combining them by integrating a compact AFM into SEM brings novel possibilities for true correlative microscopy and advanced multi-modal sample characterization that would be often unfeasible using each imaging modality separately.

Correlative Probe and Electron Microscopy (CPEM) represents a hardware correlative technology, enabling simultaneous acquisition of SEM and AFM data, and their seamless correlation into one 3D image. The strength lies in combination of AFM modes (3D topography, electrical, mechanical, and magnetic measurements) and SEM capabilities (fast imaging with wide resolution range, chemical analysis, surface modification, etc.). This technique can be applied using LiteScope 2.0, produced by NenoVision, and opens door for very complex investigation in a variety of fields such as Material science, Nanotechnology, Semiconductors, Life science and other areas of research and industry.

Above mentioned advantages can be demonstrated on LiNiO2 cathode material used in rechargeable batteries. Since the powdered cathode material (Figure 1) is prone to immediate oxidation upon air exposure, in-situ AFM-in-SEM approach is necessary. The SEM combined with EDX technique provided fast navigation of the AFM probe on the sample, information of elemental composition and material contrast. The AFM LiteScope was used to measure the sample topography and conductive mapping to characterize the changes in the cathode after charge/discharge cycling. Lastly, the correlated CPEM image combines AFM topography with SEM material contrast and provides unmistakable data interpretation.

In conclusion, the AFM-in-SEM strategy benefits from the complementarity of both techniques alongside significant savings both in time and resources. Also, it opens completely new possibilities for advanced data correlation and measurements, in the field of material science, nanostructures, semiconductors or life-science.

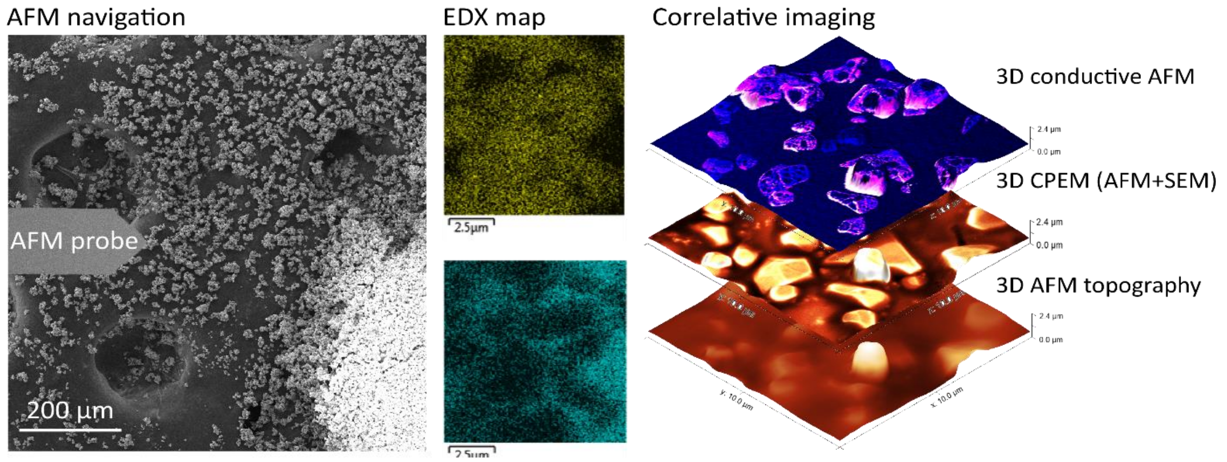

Figure 1: Complex, correlative analysis of LiNiO2 cathode powder: SEM provided fast AFM navigation to the region of interest, elemental analysis (EDX) and material contrast. AFM provided 3D topography and conductivity mapping. The 3D CPEM view merged AFM topography and SEM signal.

Combining ion beam and electron beam techniques to better understand LiB materials

Robert Steffen

Hitachi High-Tech Europe GmbH, Germany

robert.steffen.rs@hitachi-hightech.com

Scanning Electron Microscopy (SEM) and Scanning Transmission Electron Microscopy (S-TEM) are frequently used techniques for the structural and chemical analysis of battery materials, both in R&D and in failure analysis. Preparing and analyzing these devices and materials, however, is not always straightforward and it can be difficult to attain a true understanding of the structure and properties without careful preparation. This presentation briefly describes workflows and instrumentation which can significantly improve the understanding of battery materials - enabling researchers to improve battery performance and lifetime. Specifically, it describes how for SEM analysis broad Ar+ ion milling can be better used than focused ion beam milling to generate high quality cross-sections through LiB components including raw powder material, electrodes, separators or complete devices. It also describes techniques for subsequent imaging and compositional analysis, including workflows for handling of oxidation sensitive materials across BIB, FIB, SEM, STEM.

Scanning NV Magnetometry with ProteusQ

Marc Chaigneau

HORIBA Scientific/SPECION, s.r.o.

marc.chaigneau@horiba.com

The nitrogen-vacancy (NV) center is an atomic-scale defect in diamond. It hosts an electronic spin that can be initialized and detected optically, making it an exceptional system for quantum sensing of magnetic phenomena at room temperature. A particularly powerful approach is to incorporate the NV center in a scanning probe microscope (SPM), which enables a unique combination of field sensitivity with nanometric spatial resolution.

During this talk, we will present the Qnami ProteusQ, the first commercial SPM making use of NV quantum technology. Specifics of this quantum microscope, which enable quantitative and non-perturbative analysis of surface magnetic fields, will be discussed. We will demonstrate the performance of this new characterization tool through magnetometry on antiferromagnetic systems and ultra-thin ferromagnets.

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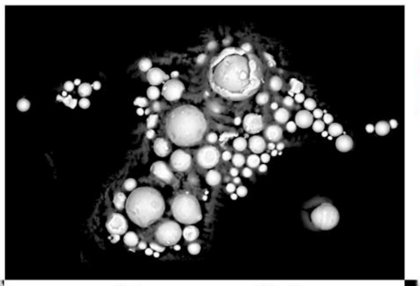
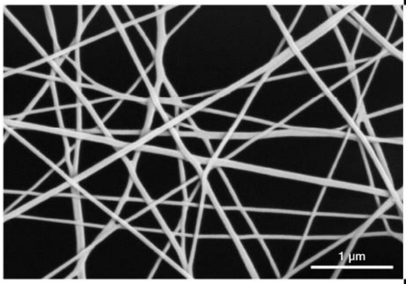




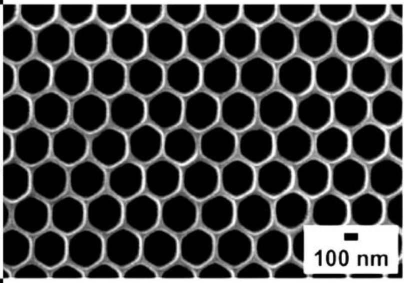
Nanoparticles

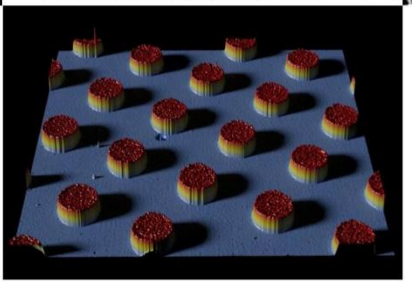
Nanofibers

ANAMET
ANALYTICAL & MEASURING & TESTING

Nanopores

Nanostructures



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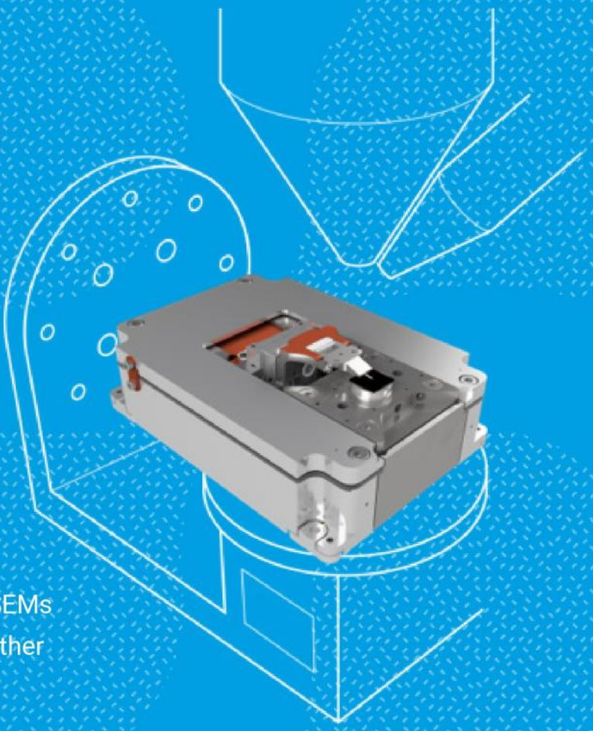
AFM for SEM LiteScope™

Next level of imaging

LiteScope™ is a unique Atomic Force Microscope (AFM) designed for „plug & play“ integration into the Scanning Electron Microscopes (SEMs).

Thanks to the unique technology of Correlative Probe and Electron Microscopy, shortly CPEM, it allows for nanometer precise in-flight AFM and SEM data correlation.



- Fast, plug and play integration into SEMs
- Compatible with FIB, GIS, EDX and other standard SEM accessories
- Highly customizable
- Can be used as a stand-alone AFM as well

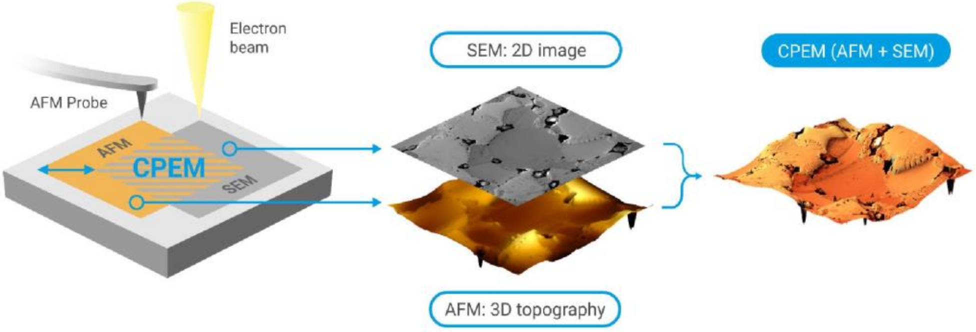
LiteScope 2.0

Product values

- Complex and correlative sample analysis
- Results of in-situ analysis are not influenced by external conditions
- Precise localization of the region of interest

CPEM advantages

- Simultaneous acquisition of AFM and SEM data
- Absolute correlative precision
- Multimodal correlation of multiple AFM and SEM images
- Extremely time efficient with respect to other correlative techniques



NenoVision

Key benefits

Enhancement of SEM capabilities

- AFM in SEM enhances the capabilities of both techniques, enabling complex sample analysis of electrical, mechanical and magnetic properties inside SEM.

Ultimate precision of correlative imaging

- Unique Correlative Probe and Electron Microscopy (CPEM) technology enables simultaneous acquisition and correlation of the chosen SEM and AFM channels.

Fast & easy localization of region of interest

- SEM helps to quickly localize the region of interes and to precisely navigate the AFM tip.

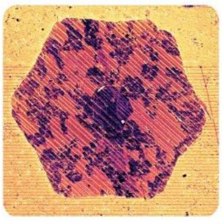
No risk of sample contamination

- In-situ conditions inside the SEM ensure sample analysis at the same time, in the same place and under the same conditions.

Extension of 2D SEM image into 3D

- SEM material contrast is enhanced by the information about sub-nanometer 3D topography and roughness.

Application areas

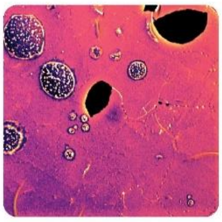


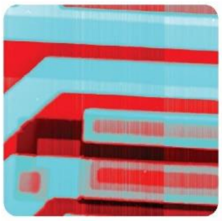
Material Science

- 1D / 2D materials
- Steel & metal alloys
- Batteries
- Ceramics
- Polymers & Composites

Nanostructures

- Modified surfaces FIB/GIS
- Quantum dots
- Nanostructured films
- Nano-patterning
- Nanowires



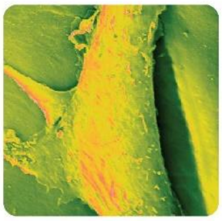


Semiconductors

- Integrated circuits
- Solar cells
- MEMS / NEMS
- Failure analyses
- Dopant visualization
- Current leakage localization

Life Science

- Cell biology
- Marine biology
- Protein technology



Technical specification:

- **Dimension XYZ:**
  118 mm x 84 mm x 37.5-48.4 mm
- **Total weight:** 460 g
- **Maximal scanned sample area XYZ:**
  21 mm x 11 mm x 8 mm
- **Scan range in open loop XYZ:**
  100 µm x 100 µm x 20 µm (±10%)
- **Scan range in closed loop XYZ:**
  80 µm x 80 µm x 16 µm
- **Resolution XYZ up to:**
  0.2 nm x 0.2 nm x 0.04 nm
- **Maximum sample height:** 8 mm
- **Maximum sample weight:** 100 g

Measurement modes

- **Imaging modes:**
  Topography and surface roughness
- **Mechanical modes:**
  Energy dissipation (tapping mode), FMM (contact mode), F-z curves, nanoindentation
- **Electrical modes:**
  C-AFM, C-CPEM, KPFM, PFM, I-V spectroscopy, STM
- **Magnetic modes:** MFM

NenoVision s.r.o.
Purkyňova 649/127
612 00 Brno, Czech Republic

info@nenovision.com
+420 605 287 732

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- **LEIS for quantitative measurement of top atomic layer**
- **High-vacuum SPM (scanning probe)**

KRATOS ANALYTICAL Ltd., UK

- **ESCA (XPS) – X ray – photoelectron microanalysis and imaging**

RIBER, France

- **MBE (molecular beam epitaxy) equipment**
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- **Particles distribution from 10 nm - 5000 µm by SLS**
- **DLS analysis of particles sizes, zeta-potential and molecular weight measurements 0,3 nm - 10 µm**
- **Surface and bulk elemental analysis by GD-OES and MS (Glow Discharge-Optical Emission Spectroscopy and Mass Spectroscopy), N/O/H and C/S analyzers**
- **Raman microscopy and micro luminescence measurement**
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- **Archimedes – particles metrological system**
- **Viscosizer – automatized ultra-low volume Taylor dispersion analysis**

HITACHI High-Technologies Corp., Japan

- **UHR-FESEM – scanning electron microscopes with ultra-high resolution 0,4 nm**
- **HR-TEM transmission electron microscopes**
- **Table-top microscopes with EDX analysis for educational and industry applications**

Leica Mikrosystems, Austria

- **Sample preparation techniques for SEM/TEM, microtomes**
- **Cryogenic sample preparation and high-pressure methods**
- **Samples coating**
- **CLEM – correlative light and electron microscopy**

SÜSS MicroTec AG, Germany

- **Precise photolithographic tools**
- **Bonding systems for microstructural elements**
- **Coating and deposition equipment for photoresists and hot plates**
- **Cleaning systems for chemical processes (MEMS) and semiconductor substrates, 3D semiconductor structures**

SPECION, s.r.o.
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Nanotechnology centre, CEET is one of the University centres concentrating on research and education in the field of technologies and nanomaterials. Their basic and application research is heading for many areas of life, recently focusing on environmental, energy and health problematics, where nanomaterials and nanotechnologies can help with additional values. Members of the centre have long term partnership with key nanotechnology industry and numerous national and foreign research institutions, being involved in world class research of excellent projects. Nanotechnology centre is open for students and young scientists giving an opportunity to study in the Nanotechnology program and to work in the international environment.