Nano and Advanced Materials NAMF 2013 Book of Abstracts

Nano and Advanced Materials NAMF 2013: Book of Abstracts

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Welcome

1

Programme

Monday, 16 September

Registration

Monday morning, 16 September, 10:00

Warsaw University of Technology: Building of Mathematics

NANOFORCE INFO DAY: Nano-particles characterisation at CePT

Monday morning, 16 September, 11:00

Chair: Witold Lojkowski

11:00

Oral

Nanometrology as a tool for nanotechnology problems solving

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Some examples will be given on the corelation between nanostructure and properties of nanoparticles, based on experiences of the Laboratory of Nanostructures for Photonics and Nanomedicine, Institute of High Pressure Physics, PAS, Center for Preclinical Research and Technologies (CePT).

For the avaiable equipment, look at http://w3.unipress.waw.pl/nano/index.php?id=aparatura

11:10

Polish Research Projects

Iinvestigation of nanoparticles by DSC-TG methods accompanied by chemical analysis of emitted gas

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Simultaneous Thermogravimetry (TG) and Differential Scanning Calorimeter (DSC) coupled with quadruple mass spectrometer provide a lot of useful data in nanomaterials investigations.

In laboratory practice is very important to keep a high tests precision, reproducibility and low measurement uncertainty, as well as use established standards. In this work, we present thermal behavior of different nanopowders such as ZnO nano-particle characterized by different morphology and nano-ZrO $_2$ used as the optical, oxygen sensor. Involving the mass spectrometer to thermal stability measurements helps to understand nature of tested nanoparticles. In case of both nano-oxides we noticed decomposition of adsorbed water in the temperature range 25-200°C and release of $\rm CO_2$ in temperature range 200-500°C. Such kind of measurements are very useful to obtain good quality of nanomaterial or to explain different behavior of nanopowders comparing to their bulk counterparts.

Netzsch STA (DSC-TG) and QMS was purchased with support of the project "Centre for Preclinical Research and Technology" CePT which was partly financed by European Union European Regional Development Fund in the frame of the Operating Program Innovative Economy, in years 2007-2013, Priority 2, Infrastructure of sphere R+D, activity 2.2

Iwona Malka gratefully acknowledges the *financial support* from the Foundation for Polish Science (FNP START 2013).

11.25

Oral

Method of Stability Evaluation for Nanodiamond Suspension

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Nanodiamond powders found broad application in the form of suspensions in medicine and biology. It is known, that because of the nanodiamond powders essential aggregation it is impossible to create stable suspensions. Therefore the stability evaluation of suspensions is an actual task.

In V.Bakul Institute for Superhard Materials NAS of Ukraine the method and technique of stability evaluation for suspensions of nanodiamonds is developed on the basis of comparasing their optical density with concentration of the suspensions. Determination of suspensions optical density was fulfilled by means of a photometric measuring. The photometric method is based on selective absorption of electromagnetic radiations buy the sectors of the suspensions with various density. The concentration of nanodiamond particles in a suspension was calculated depending on the density of nanodiamond particles, that was determined on the portable density meter of DMA 35 N as function of time. It is shown that optical density of 1% of water suspension during 2 hours diminished from 3,835E to 1,534E. So the concentration of nanodiamond particles changed from 0,37 to 0,15%

By means of photometric method kinetics of sedimentation has been studied for 1,0 % of the water suspension of nanodiamond powders (Specification ACYД 99) depending on time and temperature. It is shown that with the time increasing there are reduction of optical density and decreasing of the above mentioned nanodiamond particles concentration in the water suspension. When heating the suspension an optical density (absorbancy) goes down and speed of aggregating of nanodiamond particles increases, reducing stability of nanodiamond powder suspension.

11:40

Polish Research Projects

Characterization of nanomaterials according to EU recommendations, on the example of ZnO nanoparticles

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After more than twenty years of basic and applied research, nanomaterials and nanotechnologies started to be widely used in commercial products. In the Consumer Products Inventory there are currently listed

more than 1300 nano-goods, produced by near 600 companies which are located in 30 countries [1]. One can find nanomaterials in electronic, cosmetics, fabrics, automotive or medical products. Despite such widespread usage, until recently there was not current definition of nanomaterials in the framework of REACH. Only at the end of 2011. European Commission adopted the Recommendation on the definition [2], according to which "Nanomaterial" means: "A natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50 % or more of the particles in the number size distribution, one or more external dimensions is in the size range 1 nm - 100 nm". Such definition implies indispensable parameters describing nanomaterials, namely nanoparticles size and their size distribution. However, product requirements or health effects, usually determine other parameters such as: Specific Surface Area, particle shape, surface chemistry. Experience has shown, that characteristic of nanoparticles is a very complex issue due to the fact that a large number of different measurement techniques are available. That's why general standards in the nanoparticle characterization area were not developed till now. Within the International Organisation for Standardisation (ISO) there are only standards relating to the specific nanoforms, and particular measurement techniques [3, 4]. More general standard is planned to be published only this year [5]. Therefore, nanomaterials are usually characterized according to guides recommended by European Commission [6, 7, 8]. This practice is also used in Laboratory of Nanostructures of Institute of High Pressure Physics. In this article characterization procedure and adequate characteristic parameters are presented on the example of ZnO nanopowder, as a representative nanoparticle material (see Table 1).

Table 1. Characteristic procedure and obtained results on the example of ZnO nanopowder

1. Measured para- meter	2. Measurement tech- nique	3. Result
Morphology visualiz- ation	Scanning Electron Microscopy	Agglomerates of single-crystal nanoparticles
Phase composition	X-Ray Diffraction Analysis	Hexagonal ZnO
Specific Surface Area	BET	42 m ² /g
Density	Helium Pycnometry	5.1 g/cm ³
Average particle (agglomerate) size	Dispersive Light Scuttering	~ 85 nm
	Nanoparticle Tracing Analysis	~ 90 nm
Zeta-potential	Electrophoretic Light Scattering	34.7 mV

Acknowledgements

We would like to thanks to the NANOFORCE project: "Nanotechnology for Chemical Enterprises- how to link scientific knowledge to the business in the Central Europe space", implemented through the Central Europe Program co-financed by ERDF.

FORCE

LITERATURE

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- [2] "Commission Recommendations of 18 October 2011 on the definition of nanomaterial" (2011/696/EU)"
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11:55 Invited oral

Crystallite size determination from diffraction data: a doit-yourself tutorial.

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A method of determination of the size distribution of nanocrystals from X-Ray diffraction data will be presented. A brief introduction into experimental techniques will be given and the essential theoretical issues will be explained. A practical demonstration of the data analysis with an application of freely available software will be carried out. Limitations of the method and possible pitfalls will be discussed. Some particularly peculiar and challenging cases of data analysis will be presented.

12:15 Invited oral

Reliable XRD characterization in production or highthroughput laboratory regime

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In industrial or project-oriented research laboratories, X-Ray Diffraction (XRD) is often seen as "more art then science" technique due to

- wide variation between results from XRD vs. well-established methods (SEM/TEM/DLS),
- · large margin for interpretation, disputes and ambiguities,
- relatively high costs.

Indeed, within nanomaterials research area, there are legitimate reasons to think so, as nanocrystals violate assumptions of a number of established diffraction theorems driving them towards unstableness or even singularities. Experienced XRD experts are trained to deal with the matter, but industrial or high-throughput research laboratories require

unconditional reliability, reproducibility and specific accuracy (error bars) of the analysis.

Diffraction theories suitable for nanocrystals, i.e. got rid of infinite crystal size assumptions, are woefully complex, however. In recognition of the problem, a mild-complex diffraction theory for nanomaterials wrapped in a user-friendly web-based service has been attempted.

It is aimed to provide browser-based tool that performs microstructural analysis of nanocrystalline materials by nearly automatic processing of powder XRD pattern. Employed diffraction theory takes into account finite crystallite sizes, distribution of sizes (Grain Size Distribution), provides reliable error bars and authoritative status information (quality/quantity of input data in/sufficient for the processing).

LUNCH

Monday afternoon, 16 September, 12:35

NANOFORCE Info Day: nano-regulations, law, toxicity, nanometrology

Monday afternoon, 16 September, 13:40 *Chair: Anna Swiderska-Sroda*

13.40

Polish Research Projects

Nanoforce project, presentation of goals and achivements

Joanna Sobczyk

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Nanoforce is one of the European projects which deals with the nanosafety problem.

Thanks to OECD initiatieves and funds from European Commission, was possible realization of this project investigations. Comparisions of results and achivements of all similar projects and research which recently are performed in the 7-th Frame Program, give the possibility to have wider overview on nanotechnology responsible use.

The first step in the project it was research on current legal regulations about nanotechnology and finding out what is the situation at the moment in Central Europe countries. After completing all tasks in the project we will work out on the list of recommendations for future development of nanotechnology. Also we will try to draw the future development Road Map in time horizon of next four years.

One of the main part of the project is devoted to detection of unique nanomaterials properties by using state of the art methods of characterization. For three chosen nanomaterials nano-Ag, nano-ZnO and nanoTiO2, were made very detailed characterization with using crossimpact of the methods. To get the answer about toxicity and eco-toxicity of this nanomaterials were made some toxicological tests. Next, the Safety Data Sheets were elaborated as well as Exposure Scenarios. Report from that part of the project should include carefully described list of performed experiments, results and conclusions.

The third part is dedicated to build up financial background for future development of nanotechnological SEMs. That's for in the project was developed Nanodeals Generator to place ideas which could be finansed in the future. Detailed description about Nanotechnology Industry and companies in Central Europe were worked out as well as description of future perspectives for development. New established Technological Parks in Poland could be best place to host new SEMs.

Multidisciplinary skills and multitask work should be involved to fulfill all necesary activities in the project. Moreover, many teams like ours should add thair competencies and knowledge to answer all the open questions concerning nanotechnology.

13:50

Invited oral

Nanomaterials' EU legislative activities - situation at 16 September 2013

<u>Paulina Porębska-Sęktas</u>, Andrzej Krześlak, Marcela Palczewska-Tulińska

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1. INTRODUCTION

The usage of nanomaterials and nanotechnology in many sectors of industry is becoming more common. Therefore, the total quantity of nanomaterials placing on the market is increasing rapidly year by year. The industry is attracted by the wide range of benefits and possibilities offered by these materials.[1] However, because of lack of definite answer whether the use of nanomaterials is safe or not, in 2008, the European Commission decided to launch the first regulatory review of the EU legislation concerning nanomaterials.

2. REGULATORY REVIEW IN THE AREA OF NANOMATERIALS

2.1. Definition

As a result of the first review, in 2011, new definition of nanomaterial has been introduced by the European Commission. It is based solely on the size of the constituent particles of a material, without regard to hazard or risk [2]. The definition engendered a wide discussion among stakeholders and will be reviewed in 2014. European Commission plans to implement the new definition in the EU legislation, however, in justified cases sector specific solutions may also still apply [1].

2.2. Regulation No 1907/2006 (REACH)

Nanomaterials are covered by the provisions of the Regulation No 1907/2006 (REACH) concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals. Therefore nanomaterials that are produced or imported outside the Community in quantities over 1 tonne anually per relevant producer/importer must be registered. Registrants decide whether their nanomaterial is a form of a bulk substance (then nano form is included in a dossier for the bulk substance) or a separate nanosubstance (then it is registered on its own). In opinion of some NGOs, REACH in current state is not able to provide sufficient control over these materials and changes in the legal text and annexes or even a separate legislation are needed [3]. The European Commission has published public consultation on potential changes in REACH Regulation to better cover substances in nanoscale. Respondents have 6 options to consider, for example: no policy actions, changes in certain annex provisions, guidance updates or introducing revised or additional endpoints. The consultation runs until 13 September 2013. [4]

2.3. Regulatory initiatives on national level

In opinion of several Member States current regulations are not sufficient to provide safety of nanomaterials. France has published a decree on the annual declaration on substances at nanoscale. It requires companies, private and public research laboratories to declare the quantities and uses of substances produced, distributed or imported in France at nanoscale. The declaration is mandatory for substances produced, imported or distributed during the previous year in a quantity greater than 100 g and must be submitted before the 1st May each year. [5].

Similar decree regarding placing on the market substances in nanoscale is planning to be published in Belgium. Draft decree has been already submitted to the Commission. The declaration will have to include: identification of the declarer, identification of the substance, quantity of substance manufactured at the nanoscale placed on the market during the reporting period, uses of the substance and identity of the professional users. [6]

Denmark has launched a public consultation, related to a draft executive order for a national register of products and mixtures containing nanomaterials. The order is expected to come into force by 1 January 2014. [7]

There is also a group of Member States that suggest introducing European Registry of such products rather than national ones. As the result of these claims European Commission has issued a call for tender to help it prepare an impact assessment for a nano registry. The information required to be gathered includes: costs and administrative burden of such registry, possible effects on competiveness and potential benefits. [8]

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- [5]Decree no. 2012-232 of 17 February 2012 on the annual declaration on substances at nanoscale in application of article R. 523-4 of the Environment code;
- [6]Draft Royal decree regarding the placement on the market of substances manufactured at the nanoscale;
- [7] V. Zainzinger, Denmark consults on nanomaterial product register, ChemicalWatch,10 July 2013;
- [8]EU Commission tenders nano-registry impact assessment, ChemicalWatch, 27 June 2013.

14:10

Invited oral

Regulatory Aspects of Nanomaterials

Krzysztof Maruszewski

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The potential benefits of nanotechnology and nanomaterials for society are well known. However, the large scale use of particulate nanomaterials raises several issues regarding possible detrimental effects on human health and the environment. This applies to particulate nanomaterials already produced and used in large quantities, as well as the huge range of novel nanomaterials presently under development for different applications. These safety issues need to be addressed before any significant problems arise. For this reason a clear regulatory framework is needed to address them, without stifling innovative research, and without discouraging industry from developing and introducing new processes and consumer products based on nanomaterials. Such a framework should also reassure consumers about nanotechnology developments and foster the acceptance of nanotechnology-based products. EU legislation applies in principle to nanotechnology and

nanomaterials, and in recent years some specific provisions for nanomaterials have additionally been introduced into legislation, e.g., for cosmetic products, biocides, food labelling, and also for materials in contact with foods. Any nano-specific provisions must take into account that our current understanding of physiological responses to engineered nanomaterials is far from comprehensive. Improved nano-specific risk assessment and management methods have to be developed and incorporated into a good regulatory framework, based on validated testing methods. Further research and reliable scientific data is urgently needed in this respect. Regarding labelling requirements, analytical methods for their implementation, based on the Commission recommendation for a regulatory definition of nanomaterials, also need to be developed and validated. This presentation provides a brief overview of recent developments as summarized in the Communication from the Commission in its Second Regulatory Review on Nanomaterials[1]. It addresses the major scientific challenges regarding implementation of nanospecific regulations, especially with respect to determining whether a material falls into the category of 'nanomaterial' or not. It also touches on the challenging problem of detection, quantification and characterisation of nanomaterials in complex matrices, for which methods may be required in both industrial and official control laboratories. The presentation also discusses quality assurance tools such as reference materials and methods, including related databases, and emphasizes the role of the OECD Working Party on Manufactured Nanomaterials (WPMN), and the standardization activities of ISO and CEN, which help to foster a globally harmonised approach to the assessment of nanomaterials.

[1] EU Commission Communication COM (2012) 572 final

14.40

Invited oral

International, European and national standardization activity, review of publications and developed projects in nanotechnologies

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The dynamic development of nanotechnologies in recent years makes it necessary to normalize the many issues related to nanotechnologies and nanosciences. In the current international (ISO), European (CEN) and national (PKN) standardization organizations, new organizational structures to carry out the standardization activities in the field of nanotechnologies and related issues. This presentation discusses the history, scope of activities and cooperation with other organizations and major achievements of technical committees ISO/TC 229 and CEN/TC 352 and national committees (KT 207 and KT 314).

15:10

Invited oral

Nanomaterials characterization as a tool to high valueadded products

<u>Luis Rino</u>¹, João Gomes², Teresa Monteiro¹, Florinda M. Costa¹, A. J. Neves¹, Maria R. Soares¹, Joana Rodrigues¹, Cláudio Nico¹

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Novel nanomaterials and new nanotechnology applications are cornerstones of innovative high value-added products in several industrial fields. From a technological point a view, there is a strong demand for techniques to fabricate and measure the properties of these nanomaterials and related devices. In parallel, at a more fundamental level, a higher comprehension of the materials physical properties at the nanoscale level is needed.

Properties of these novel materials depend on a myriad of parameters. As an example optical properties will depend on such as feature size, shape, surface characteristics, interaction with the surrounding environment, etc. Characterization of nanomaterials employs expensive high-tech equipment combined with multidisciplinary teams of experts that cannot usually be accommodated in a Small to Medium-sized Enterprise budget.

This talk will address the advantages for the industry, mainly SMEs to partnership with established academic laboratories in order to have access to top research capabilities with state-of-the-art instrumentation and multidisciplinary specialists to help developing or improving their products, so that an essential value added step is incorporated, at affordable costs.

Representative material characterization techniques will be presented alongside with several successful examples of collaborations between our research group and several companies.

COFFEE and POSTERS

Monday afternoon, 16 September, 15:40

NANO-FORCE info day: nano-regulations, law, toxicity

Monday afternoon, 16 September, 16:10 *Chair: Joanna Sobczyk*

16:30

Oral

Legal aspects of nanotechnology

Marcin R. Jurewicz

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Nanotechnology regulations should guarantee the public an opportunity to obtain the benefits of nanotechnology applications with a high degree of compliance with safety, health and environmental protection. The provisions of the European Union law in relation to nanomaterials are arranged according to: products, chemicals, protection of workers and environmental protection. The legislation directly referred to nanotechnology was introduced for cosmetics, biocides and food so far. In other areas where nanomaterials are profited the provisions for their equivalent macro are used.

A duty to extensive data transmission by companies placing nanomaterials on the market in the evaluation procedure, entering the Community lists of authorized substances, granting permits allows authorities to verify the properties of nanomaterials, enhancing their safety and gradually reducing the gaps in scientific knowledge. Furthermore, having such detailed information helps companies to commercial use of nanomaterials. A duty to place components in the form of nanomaterials on the packaging of the product in the list of ingredients and followed them by the word 'nano' serves to strengthen the position of consumers on the market, giving them the possibility of a conscious, deliberate deciding on the purchase of the product.

16:50 Invited oral

Potential exposure to nano-objects in the work environment

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Knowledge about the risks arising from exposure to nano-objects is still limited, mainly due to the small number of studies carried out in real exposure of workers to nano-objects and the lack of uniform rules to interpret the obtained results in order to assess exposure [Brouwer D and et al. Harmonization of measurement strategies for exposure to manufactured nano-objects; Report of a Workshop. Ann. Occup. Hyg. 2012;56(1):1–9. DOI 10.1093/annhyg/mer099]. German institutes [Tiered approach to an exposure measurement and assessment of nanoscale aerosols released from engineered nanomaterials in workplace operations (2011), https://www.vci.de/Downloads/Nanomaterials%20in%20Workplace%20Operations.pdf.] proposed a three-tiered approach to estimate the potential exposure to nano-objects and their aggregates and agglomerates, namely: Tier 1 - collecting information about workplace conditions for ruling out (conventional risk assessment) or confirming (go to the Tier 2) possibility of presence of nanoaerosol in the workplace air, Tier 2 - performing of measurements with easy to used device, and if total number concentration extending reference value for investigated nanoaerosol or is considerably higher compeer to "no activity", measurements should be taken for determination potential exposure to nanoaerosol according to Tier 3, if not risk assessment should be carried out on the basis of data received, Tier 3 - potential exposure to nanoaerosol must be conducted with use of new knowledge and of measuring devices, as condensation particle counter (CPC), scanning mobility particle sizer spectrometers (SMPS) and nanoparticle surface area monitor (NSAM). Simultaneously samples of nanoaerosol should be taking for their further analysis with TEM-EDS or SEM-EDS. The aim of the performed studies was to investigate of potential exposure to nano-objects during processes of production and handling of different kind of nanomaterials. Measurements were done with devices for real-time measurements of concentrations and size distributions of particles with SMPS, condensation particles counter (P-Trak)) nanoparticle aerosol monitor (Aero-Trak) and electrical low pressure impactor (ELPI+). For confirmation of presence of nano-object in the work environment air samples were taking with nanometer aerosol sampler (NAS) for future analysis with electron microscope (TEM or SEM) and energy dispersive X-ray spectrometer (EDS). For most investigated processes it was confirmed that nano-objects were released to the work environment.

17:10

Invited oral

Toxicity testing of nanomaterials

Maciej M. Stępnik

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Over recent decades, nanomaterials are increasingly produced as the result of the rapid development in nanotechnology. They are currently used in many industries as well as in medicine and pharmacology. As the result, an increasing concern has been raised over their potential impacts to human health. Efforts in standardizing methods to study the in vitro and in vivo safety of these materials are currently undertaken by various government agencies and research organizations. In this lecture the following issues are discussed: physico-chemical

properties of nanomaterials determining their biological effects, major reported adverse effects after the exposure to nanomaterials and different aspects of toxicological risk assessment of these materials.

17:30

Polish Research Projects

Nano PUZZLES Project: Modelling properties, toxicity and environmental behaviour of engineered nanoparticles

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Nanotechnology is rapidly expanding. However, some types of engineered nanoparticles can be toxic for living organisms and exhibit negative impact on the environment. Thus, the design of new nanoparticles must be accompanied by a rigorous risk analysis. Following the recommendations by the EU REACH system and regarding ethical aspects, the risk assessment procedures should be performed with possible reduction of living animals' use. One of the most promising options is the application of computational techniques. In addition to the reduction of animal use, such a solution enables significantly reducing costs of the required risk assessment.

The main objective of the NanoPUZZLES project is to develop, within three years, a package of computational algorithms for the comprehensive modelling of the relationships between the structure, properties, molecular interactions and toxicity of selected classes of engineered nanoparticles (NPs). The package (i) will serve as a proof-of-theconcept that the risk related to NPs can be comprehensively assessed with use of computational techniques and (ii) will define a basis for development of further modelling techniques for a large variety of nanoparticles.

The project will focus on two groups of compounds: (i) inorganic engineered nanoparticles (metal nanooxides) and (ii) carbon nanoparticles (carbon nanotubes (single-walled and multi-walled), fullerenes and fullerene derivatives). That choice was dictated by the wide application of these nanoparticles in everyday household products, and by the fact that these compounds are commercially available in the market whch eliminates the necessity for their synthesis (reduction of costs).

Computational algorithms will be developed within four work packages related to the following thematic areas:

- · Quality assessment of physicochemical and toxicological data available for nanomaterials and data exploration (Nano DATA),
- Development of novel descriptors for nanoparticles' structure (NanoDESC),
- Simulating interactions of nanoparticles with biological systems (NanoINTER),
- Quantitative and qualitative structure-activity relationship modelling, grouping and read across (NanoQSAR).

Application of the methods developed within the four thematic areas will allow for predicting toxicity and the behaviour of novel nanoparticles from their structure and/or physicochemical properties without the necessity of performing extensive empirical testing. Moreover, it will result in a framework being established to categorise nanoparticles according to the potential for exposure, as well as physicochemical, structural and toxicological properties (based on available empirical data and computationally predicted results). This, in the longer perspective, should lead to designing and engineering nanomaterials that are of low risk for human and the environment.

COCKTAIL

Monday afternoon, 16 September, 17:50

NANOFORCE Info Day Poster session

Monday evening, 16 September, 18:00 Posters presented during breaks

Poster

18:00 NF1

Legal aspects of nanotechnology in environmental protection

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Legal problems:

- there is no regulation controlling the release of nanomaterials manufactured into the environment in European or international environmental regulatory laws, European Commission is finalizing the review assessing the adequacy of EU legislative framework for products of nanotechnologies,
- -EU is currently considering how to apply current environmental regulatory laws (On 3 October 2012, the Commission adopted the Communication on the Second Regulatory Review on Nanomaterials) analysis of the current european environmental law (integrated pollution prevention and control, major-accidents, air quality, soil, water, waste, industrial emissions, environmental liability), adequate implementation may require adaptations to the legislation as well as the development of new procedures,
- -The European Commission is responsible for proposing legislation as well as the administration and enforcement of enacted legislation. "Precautionary principle" forms the basis for all environmental directives that are under consideration or have been issued by the EC,

-approaches to regulation – whether and how to regulate nanotechnology? soft law or hard law mechanisms? – soft law mechanisms serves to assist in the implementation of existing hard law.

18:00 Poster NF2

Methods for testing dustiness of nanomaterials

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Intensive growth of the production and use of nanomaterials in industry obliges to carry out activities aimed at developing methods for prediction of exposure to nanomaterials, depending on the process used. One such method is the study of the dustiness of nanomaterials generated (at constant conditions), and then test the physical parameters and the time of staying the nano-objects in the air. In 2006 was developed standard EN 15051 "Workplace atmospheres - Measurement of the dustiness of bulk materials - Requirements and reference methods",

which provides general guidelines for testing dustiness of materials occurring in workplaces, and is applicable to industrial dust emissions in various manufacturing processes. In the standard EN 15051 are presented two standard methods (gravimetric methods), differ in the aerosol generating means, namely: 1) method A - the method of the rotating drum, 2) method B - the method of continuous drop of the material. The results obtained using the method A and / or B are used to determine the dustiness of materials for the respective particle size fractions (important because of their effects on health), namely dustiness for inhaled, thoracic and respirable fractions when data obtained by the rotating drum method and the dustiness for inhalable and respirable fractions for data obtained by the continuous drop of the material. Dustiness determined with gravimetric method can be one of the nanomaterials parameter but more appropriate for evaluation of workers exposure to nanomaterials are parameters which characterized of nanoobjects emission, as number concentration and size distribution. The INRS has developed a new method of testing dustiness of nanomaterials. In this method for generation of nanomaterials shaker was used and the method is called "vortex shaker". The tests may be carried out in two options, namely version allows performed tests:1) in real time (for respirable fraction) using the CPC and the ELPI and possibly in the collection of samples for microscopic analysis, 2) with gravimetric method using measurement filters. Method of testing dustiness with method VS was modified in CIOP-PIB for determination of dustiness of nanomaterials in real time with CPC, ELPI+, SMPS and AERO-TRAK. Test procedure for gravimetric method is the same as in INRS method. Currently are carried out standardization works (CEN) in the field of "Workplace atmospheres - Measurement of dustiness of bulk nanomaterials". Participants of the project are: INRS (leader), CIOP-PIB, HSL, IGF, NRCWE and TNO.

18:00	Poster	NF3
10.00	1 05101	1117.5

Polish Nanotechnology Platform - a discussion group at Linked-In

Witold Łojkowski

Polish Academy of Sciences, Institute of High Pressure Physics (UNIPRESS), Sokolowska 29/37, Warszawa 01-142, Poland Instytut Wysokich Ciśnień PAN (IWC), Sokołowska 29/37, Warszawa 01-142, Poland

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The Linked-in Group Polish Nanotechnology Platform is presented. This is a discussions and sharing information group in the cloud. Its possibilities are shown and researchers as well as companies are encouraged to join it.

10.00	Th	3.757.4
18:00	Poster	NF4

Zastosowanie nanotechnologii w stomatologii

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Wprowadzenie

Nanotechnologia, jako stosunkowo młoda dziedzina nauki, jest częścią niezwykle dynamicznie rozwijającego się życia naukowego. Opierając się na przełomowej pracy i przemyśleniach R. Feynmana, udało się stworzyć nowy wymiar nauki, bazujący na wszystkich dotychczasowych odkryciach z dziedzin takich jak: biotechnologia, biologia, chemia,

fizyka molekularna i wielu innych. W wyniku badań naukowcy odkryli i stworzyli nanocząsteczki i nanomateriały, dzięki którym możliwe jest udoskonalenie już istniejących materii i zjawisk, jak również - co najważniejsze - tworzenie zupełnie nowych, o niezwykłych właściwościach i co za tym idzie - nieprawdopodobnych możliwościach.

Wychodząc naprzeciw potrzebom medycznym, nanotechnologia znalazła zastosowanie również w stomatologii. Początki zastosowań w tej dziedzinie datuje się na lata 70. XX wieku. Wtedy to po raz pierwszy do materiału kompozytowego do wypełnień dodano nanocząsteczki tlenku krzemu. Od tego momentu rozpoczęła się rewolucja na rynku dentystycznym, zaczęto eksperymentować z nanocząsteczkami i nanomateriałami w celu uzyskania coraz lepszych wypełnień oraz podjęto próby ulepszania istniejących już metod, skierowanych w stronę odbudowy zębów oraz leczenia kanałowego.

Przegląd produktów dostępnych na rynku

Przełomowym produktem, który jako pierwszy pojawił się na rynku, okazał się Filtek Supreme Universal Restorative - nanokompozyt do odbudowy zębów przednich i bocznych, wyprodukowany przez firmę 3M ESPE w 2002 roku. Do dnia dzisiejszego firma cieszy się uznaniem oraz wprowadza na rynek nowe, udoskonalone produkty, które wykorzystują nanotechnologię w walce o zdrowy uśmiech.

Przykładowe produktu firmy 3M ESPE z nanotechnologią:

N a z w a produktu	Opis
Ketac TM N100	światłoutwardzalny nano-jonomerowy materiał do wypełnień, wykazujący zwiększoną odporność na ścieranie jak również wysoki poziom uwalniania fluoru.
Filtek TM Z550	tzw. wszechstronna hybryda, uniwersalny materiał kompozytowy do wypełnień. Charakteryzuje się dużą wytrzymałością na zginanie oraz rozciąganie, odpornością na ścieranie jak również ulepszonym utrzymaniem połysku.
Lava TM Ultimate CAD/CAM	nanoceramika kompozytowa RNC, wyróżniająca się wysokim stopniem wydajności, wyjątkową wytrzymałością i odpornością na zginanie (co zapobiega odpryskom warstwy licowej) oraz zapewnia trwały połysk i walory estetyczne.

Oprócz firmy 3M ESPE na polskim rynku działa prężnie rozwijająca się firma Dental Nanotechnology. W swojej ofercie proponuje przełomowe produkty bazujące na nanotechnologii.

Wybrane produkty Dental Nanotechnology



preparat do końcowego płukania kanałów korzeniowych, charakteryzujący się skutecznym i trwałym działaniem antybakteryjnym, oparty na bazie nanocząsteczek srebra wzbogacony o złoto.

Nanocare Gold



preparat eliminujący przyczyny próchnicy wtórnej, oparty na bazie nanocząsteczek złota i srebra o silnych właściwościach bakteriostatycznych i przeciwgrzybiczych, neutralnych chemicznie.Preparat nie przebarwia wypełnień oraz zwiększa ich trwałość. Stosuje się pod wypełnienia stomatologiczne i protezy zębowe stałe.

Wyżej wymienione produkty nie są jedynymi, które wykorzystują niezwykłe właściwości nanotechnologii. Pozostałe wybrane produkty to:

Nazwa firmy	Produkt	Opis
Zhermapol	Silikony addycyjne HD+	w paście podstawowej zastosowano nanowypełniacze o średniej wielkości ziarna około 200 nm, zaś w paście katalizującej platynę. Produkt wykazuje lepszą charakterystykę utwardzania, wysoką hydrofilowość, pamięć materiałową oraz bardzo małą zmienność liniową wymiarów.
BlanX	BlanX BioRepair Whitening	pasta do zębów z nanocząsteczkami microRepair - remineralizują szkliwo, odbudowują mikrouszkodzenia szkli- wa.
Dentsply Polska	Ceram - X	uniwersalny, światłoutwardzalny system materiałów kompozytowych z nanoceramicznym rodzajem wypełniacza, zapewnia idealne wrażenia estetyczne.
Dentsply Polska	Prime & Bond NT	oparty na nanotechnologii światłout- wardzalny system wiążący, przezn- aczony do trwałego łączenia materiałów kompozytowych oraz kompomerowych ze szkliwem i zębiną oraz metalem i ceramiką.
UC3M we współpracy z CEOSA - Euroortodon- cia	aparatach orto-	zastosowanie nanocząsteczek tlenku glinu na polisulfonie - w wyniku czego otrzymuje się zwiększoną odporność na ścieranie i poprawę sztywności aparatu.

Podsumowanie

Biorąc pod uwagę różnorodność i mnogość możliwości użycia nanopreparatów można stwierdzić, że w ostatnich latach nastąpił dynamiczny wzrost zainteresowania zastosowaniem nanotechnologii w stomatologii. Głównymi zaletami i osiągnięciami produktów z nanotechnologia sa:

- zwiekszona trwałość i wytrzymałość;
- właściwości bakteriostatyczne i przeciwgrzybiczne;
- · neutralność chemiczna;
- odporność na ścieranie i zginanie;
- wysoka polerowalność;
- remineralizacja szkliwa i zębiny;
- możliwość działania w skali nano.

Dzięki badaniom naukowców i nieustającym próbom udoskonalania produktów z użyciem nanotechnologii, zabiegi stomatologiczne mogą być dla pacjenta zarówno bezbolesne jak również dawać długotrwałe efekty leczenia.

Zastosowanie nanotechnologii w dentystyce to stomatologia przyszłości.

18:00 Poster NF5

Nanotechnologie w gospodarce województwa podlaskiego

Joanicjusz Nazarko¹, Katarzyna Dębkowska¹, Joanna Ejdys¹, Alicja Gudanowska¹, Anna Kononiuk¹, <u>Elżbieta Krawczyk-Dembicka</u>¹, Krzysztof J. Kurzydlowski², Dorota Leończuk¹, Malgorzata Lewandowska², Witold Łojkowski^{1,3,4}, Andrzej Magruk¹, Anna Olszewska¹, Adam W. Skorek¹, Urszula Widelska¹

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Projekt "Foresight technologiczny <<NT FOR Podlaskie 2020>> Regionalna strategia rozwoju nanotechnologii" ukierunkowany był na opracowanie podlaskiej strategii rozwoju nanotechnologii do roku 2020. Jednym z głównych celów projektu było wyznaczenie nanotechnologii kluczowych dla rozwoju województwa podlaskiego. Cel ten został zrealizowany poprzez prace Panelu Kluczowych Technologii (PKT).

Istotnym aspektem przygotowania prac panelu PKT był dobór szerokiego spektrum ekspertów ze świata nauki, biznesu oraz administracji i polityki. Wśród wybranych specjalistów znalazły się zarówno osoby pracujące nad tworzeniem, jak i nad wdrażaniem nanotechnologii oraz osoby ze sfery polityki regionalnej. Eksperci reprezentowali różne regiony Polski, ale w związku ze specyfiką projektu wymagane było ich zaznajomienie z charakterystyką ekonomiczno-społeczną województwa podlaskiego. Do zadań ekspertów należało opracowanie wstępnego katalogu technologii kandydujących z obszaru nano, a następnie ich ocena według kryteriów wykonalności i atrakcyjności w celu wyłonienia technologii kluczowych. Kolejnym etapem była priorytetyzacja technologii kluczowych poprzez ekspercką ocenę ich gotowości technologicznej.

Praca panelu eksperckiego PKT pozwoliła na wyodrębnienie propozycji tych nanotechnologii, które w najwyższym stopniu mogą przyczynić się do dynamicznego i zrównoważonego rozwoju społeczno-gospodarczego Podlasia.

Gałęzie przemysłu województwa podlaskiego, biorąc pod uwagę poziom zastosowania nanotechnologii, można podzielić na trzy kategorie:

- wykorzystujące nanotechnologie: przemysł maszynowy, tekstylny i odzieżowy, meblarski, budowlany, rolno-spożywczy i medyczny;
- planujące wykorzystać nanotechnologie: produkcja wyrobów gumowych i z tworzyw sztucznych;
- niezainteresowane wykorzystaniem nanotechnologii: przemysł drzewny i metalowy.

Nanotechnologie w opinii badanych przedsiębiorstw są znaczącym czynnikiem sprawczym, decydującym o rozwoju potencjału technologicznego przedsiębiorstw. Z drugiej jednak strony, dało się też za-

uważyć jednokierunkowe postrzeganie nanotechnologii tylko w kategoriach technologicznych. W mniejszym stopniu dostrzega się ten rodzaj usprawnień jako czynnik popytotwórczy, wpływający na poziom zaspokojenia potrzeb rynków docelowych.

Większą skłonność do wykorzystywania nanotechnologii mają przedsiębiorstwa, które posiadają już ten rodzaj usprawnień. Nanotechnologie w opinii większości badanych firm je wykorzystujących mają bezpośrednie przełożenie na osiągane efekty produkcyjne i rynkowe.

Podlaskie firmy dostrzegają pozytywny wpływ nanotechnologii na rozwój potencjału ekonomicznego. Korzystny charakter oddziaływania nanotechnologii jest jednak oceniany na poziomie dużej ogólności.

Wiedza na temat nanotechnologii jako czynnika decydującego o rozwoju potencjału technologicznego jest w dużym stopniu ograniczona. Przedsiębiorcy często w ocenie zjawiska posługują się uogólnieniami. Należy dostrzec bezpośrednie zależności pomiędzy potencjałem wiedzy a zastosowaniem nanotechnologii. Po pierwsze, właściwie zgromadzona wiedza zmniejsza ryzyko porażki rynkowej. Po drugie, nanotechnologie są zjawiskiem stosunkowo nowym, wymagają więc wiedzy ciągłej i aktualnej. Luka informacyjna jest szansą dla uczelni i instytutów badawczych, co może być ważnym czynnikiem powodującym zacieśnianie związków pomiędzy nauką i praktyką gospodarczą.

Przedsiębiorstwa planujące wdrożenie nanotechnologii wykazują postawę raczej zachowawczą. Wynika ona przede wszystkim z relacji, jakie występują pomiędzy firmami na poziomie sektorów. Nie wymuszają one zmian oraz nie są bezpośrednią przyczyną przedsięwzięć o charakterze proinnowacyjnym. Planowanie zastosowania nanotechnologii w przyszłości pokazuje świadomość przedsiębiorstw w zakresie korzyści wynikających z zastosowania nanotechnologii, jednak, biorąc pod uwagę sytuację w sektorze, decyzje o ich zastosowaniu są odkładane na przyszłość. Przeszkodą we wdrażaniu nanotechnologii w podlaskich przedsiębiorstwach są: bariery technologiczne, ekonomiczne, organizacyjne, społeczne oraz mentalne i świadomościowe.

Nanotechnologie mogą być jednym z istotnych czynników podniesienia produktywności i konkurencyjności regionu. Z zastosowaniem nanotechnologii wiążą się korzyści kooperacyjne, innowacyjne, społeczne, wizerunkowe oraz strategiczne. Uzyskane wyniki badań stanowią istotne źródło wiedzy o uwarunkowaniach rozwoju gospodarczego regionu w warunkach globalnego rozwoju technologicznego i powinny być ważną przesłanką w kształtowaniu polityki innowacyjnej województwa podlaskiego, wyrażającej się między innymi w podejmowaniu decyzji o wspieraniu przedsięwzięć związanych z opracowywaniem i wdrażaniem produkcji i usług opartych na nanoinnowacjach.

Efekty realizacji projektu są dostępne na stronie www.nt-fp2020.pb.edu.pl.

Projekt "Foresight technologiczny <<NT FOR Podlaskie 2020>> Regionalna strategia rozwoju nanotechnologii" był współfinansowany ze środków Europejskiego Funduszu Rozwoju Regionalnego oraz ze środków budżetu państwa w ramach Programu Operacyjnego Innowacyjna Gospodarka.

18:00 Poster NF6

Działalność naukowo-badawcza w zakresie nanotechnologii zidentyfikowana w projekcie "Foresight technologiczny <<NT FOR Podlaskie 2020>> Regionalna strategia rozwoju nanotechnologii"

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Projekt "Foresight technologiczny <<NT FOR Podlaskie 2020>> Regionalna strategia rozwoju nanotechnologii" był realizowany na Wydziale Zarządzania Politechniki Białostockiej w okresie kwiecień 2009 - czerwiec 2013. W tym czasie przeprowadzono szereg badań, których wyniki pozwoliły na wyłonienie katalogu kierunków badań w obszarze nanotechnologii, które w najwyższym stopniu przyczynią się do zrównoważonego rozwoju społeczno-gospodarczego województwa podlaskiego. Efektem końcowym prac realizowanych przez panel badań naukowych w zakresie nanotechnologii na rzecz rozwoju Podlasia jest wykaz priorytetowych kierunków badań, czyli takich, których poziom trudności gwarantuje relatywnie wysokie prawdopodobieństwo sukcesu rynkowego, polegającego na wdrożeniu rozwiązań do praktyki gospodarczej.

Proces badawczy uwzględniał realizację następujących zadań badawczych:

- Zadanie 1. Opracowanie wstępnego katalogu kierunków badań
- Zadanie 2. Opracowanie kryteriów oceny kierunków badań
- Zadanie 3. Wyłonienie kluczowych kierunków badań
- Zadanie 4. Opracowanie założeń priorytetyzacji kierunków badań
- Zadanie 5. Priorytetyzacja kierunków badań

Zadanie 6. Sporządzenie katalogu priorytetowych kierunków badań Podstawową techniką badawczą wykorzystywaną na potrzeby prac panelu były badania ankietowe realizowane wśród 27 członków panelu ekspertów. W tym celu wykorzystanych zostało 6 kwestionariuszy badawczych, które po wypełnieniu eksperci przesyłali drogą elektroniczną. Uzyskiwane w trakcie poszczególnych etapów wyniki uporządkował Kluczowy Zespół Badawczy.

Ostatecznie na wykazie kandydujących kierunków badań w obszarze nauk podstawowych znalazło się 90 kierunków badań, a w zakresie stosowanych 93 kierunki badań, z następujących obszarów: a) nauki podstawowe:

- nanonametrialy,
- · biotechnologia,
- nanomedycyna,
- przemysł maszynowy,
- przemysł drzewny,
- nauki społeczne;

b) nauki stosowane:

- przemysł drzewny,
- budownictwo,
- drogownictwo,
- przemysł włókienniczy,
- · nanomedycyna,
- · bionanotechnologia,
- rolnictwo i przemysł spożywczy,
- technologie informatyczne, ICT,
- energia.

Do obszarów o potencjalnie najszerszych badaniach naukowych w obszarze nauk podstawowych eksperci zaliczyli nanomateriały.

Do obszarów o potencjalnych najszerszych badaniach naukowych w obszarze nauk stosowanych eksperci zaliczyli:

- · nanomedycynę;
- technologie informatyczne;
- budownictwo;
- bionanotechnologie.

Na podstawie studiów literaturowych zidnetyfikowano dwie grupy kryteriów wykorzystywanych na potrzeby oceny kierunków badań, analogiczne do kryteriów oceny technologii:

- 1) kryteria odnoszące się do wykonalności;
- 2) kryteria odnoszące się do atrakcyjności.

W grupie kryteriów odnoszących się do atrakcyjności określono:

- innowacyjność realizowanych kierunków badań, których brakuje w Polsce lub ich poziom jest słaby, a są potrzebne ze względu na własne i sasiednie rynki zbytu:
- prawdopodobieństwo nawiązania międzynarodowej naukowej współpracy (rozszerzenie międzynarodowej współpracy obejmującej wzajemną wymianę naukowców, studentów i kadry zarządzającej w dziedzinach kluczowych dla rozwoju regionu);
- możliwości komercjalizacji wyników badań;
- wpływ kierunków badań na dalszy rozwój infrastruktury B+R w regionie;
- potencjalny wpływ prowadzonych badań na rozwój nowych branż przemysłu w regionie;
- potencjalny wpływ prowadzonych badań na rozwój branż przemysłu występujących w regionie.

W grupie kryteriów odnoszących się do wykonalności określono:

- możliwości finansowania kierunków badań ze źródeł wewnętrznych i zewnętrznych;
- ambitność i unikalność kierunku badań na skalę Polski, która pozwoli na uzyskanie odpowiednio dużych pieniędzy na rozwój regionu;
- istniejące zasoby kadrowe niezbędne do prowadzenia badań w regionie;
- istniejący i przewidywany rozwój infrastruktury badawczej (w tym Białostockiego Parku Naukowo-Technologicznego, uczelni) na rozwój przysztych badań;
- istniejący stopień zaawansowania prowadzonych badań naukowych. Wyniki przeprowadzonej oceny kandydujących kierunków badań oparte na zestawie kryteriów odnoszących się do atrakcyjności oraz wykonalności, pozwoliły na zawężenie zestawu do 16 kierunków w grupie nauk podstawowych oraz 16 kierunków w grupie nauk stosowanych.

Realizowana w dalszej kolejności przez zespół projektowy priorytetyzacja kierunków badań została przeprowadzono z wykorzystaniem metody opartej na wskaźniku poziomu trudności badań. Za priorytetowe kierunki badań uznano te kierunki, w których poddawany ocenie poziom trudności badań gwarantuje relatywnie wysokie prawdopodobieństwo osiągnięcia zakładanych celów badawczych. Docelowo takie cechy charakteryzuje poziom 3, w którym poziom trudności badań naukowych oceniany jest jako wysoki, ale związany z prawdopodobieństwem sukcesu na poziomie 80%.

W wyniku przeprowadzonej eksperckiej oceny wyłoniono siedem priorytetowych podstawowych kierunków badań oraz pięć priorytetowych stosowanych kierunków badań:

a) nauki podstawowe:

- zastosowanie nanocząstek magnetycznych w separacji makromolekuł i drobnoustrojów,
- opracowanie metod wytworzenia nanostrukturalnych materiałów dla medycyny regeneracyjnej,
- badania nad opracowaniem skutecznych środków bakteriobójczych i grzybobójczych nie oddziałujących negatywnie na środowisko,
- opracowanie metod wytwarzania nanocząstek o zadanych właściwościach funkcjonalnych do aplikacji biomedycznych,
- · nanotechnologie antybakteryjne,

- środki antyseptyczne, implanty z inteligentnych materiałów; b) nauki stosowane:
- nanomodyfikacje implantów,
- resorbowalne implanty,
- nanomateriały i nanotechnologie dla regeneracji tkanek i kości,
- biomateriały dla inżynierii tkankowej (scaffolds),
- · trwałe implanty.

W celu uzupełnienia poziomu wiedzy o kierunkach badań podstawowych i stosowanych, dalszy proces badawczy skonsultowano z ekspertami z danej dziedziny, których poproszono o przedstawienie aktualnego stanu wiedzy w zakresie danego kierunku badawczego i perspektyw jego rozwoju.

Eksperci, wśród istotnych korzyści wynikających z wdrożenia wyników badań przeprowadzonych w ramach wyżej wymienionych priorytetowvch kierunków, wymienili wzrost znaczenia jednostki naukowej oraz zacieśnienie współpracy pomiędzy sferą nauki i biznesu. Jako bariery rozwoju priorytetowych kierunków badań wskazano brak odpowiedniego wyposażenia laboratorium brak lub też małą liczbę specjalistów czy też niewystarczające kwalifikacje personelu oraz konieczność poniesienia wysokich nakładów finansowych na prowadzenie badań. Istotnym etapem badania była również ekspercka ocena szans na rozwój danego priorytetowego kierunku badań w województwie podlaskim w perspektywie roku 2020, w kontekście stanu czynników wybranych podczas dotychczasowych prac projektowych jako osie scenariuszy. Wyniki przeprowadzonego badania wskazują, że w województwie podlaskim w perspektywie 2020 roku, w warunkach zaistnienia efektywnych regionalnych sieci współpracy podmiotów biznes, nauka, administracja oraz wysokiego potencjału badawczo-rozwojowego dla nanotechnologii bardzo wysokie szanse rozwoju mają kierunki badań związane z środkami antyseptycznymi, implantami z inteligentnych materiałów oraz nanomodyfikacjami implantów. Niezbędne do rozwoju tych kierunków jest prowadzenie prac między innymi związanych z wdrożeniem implantów w medycynie oraz zidentyfikowaniem zależności pomiędzy mikrostrukturą a właściwościami implantów. Rozwój wyżej wymiennych kierunków w perspektywie 2020 roku jest warunkowany także współpracą z wiodącymi laboratoriami posiadającymi aparaturę stosowaną do nanotechnologii (lab on chip, "microroboty") oraz posiadaniem urządzeń do charakterystyki struktury i topografii warstw.

Zdaniem ekspertów przy założeniu efektywnych regionalnych sieci współpracy podmiotów biznes, nauka, administracja oraz niskiego potencjału badawczo-rozwojowego dla nanotechnologii wysokie szanse rozwoju w województwie podlaskim w perspektywie 2020 roku mają jedynie dwa priorytetowe kierunki badań związanych z nanomodyfikacjami implantów oraz resorbowalnymi implantami.

W województwie podlaskim, w perspektywie 2020 roku, przy założeniu nieefektywnych regionalnych sieci współpracy podmiotów biznes, nauka, administracja oraz wysokiego potencjału badawczo-rozwo-jowego dla nanotechnologii, wysokie szanse rozwoju ma tylko jeden kierunek badań – związany z środkami antyseptycznymi, implantami z inteligentnych materiałów.

Rozwój wyżej wymienionych kierunków badań warunkują takie kwalifikacje jak: (i) wiedza w zakresie fizyki, biochemii i biologii molekularnej; (ii) wiedza w zakresie modyfikacji procesu technologicznego w kontekście poprawy właściwości; (iii)umiejętność charakteryzowania struktury materiałów implantów i interpretacji wyników badań;(iv) umiejętności marketingowe; (v) znajomość rynku i regulacji prawnych.

Uzyskane wyniki prac panelu badawczego POB2 Badania naukowe w zakresie nanotechnologii na rzecz rozwoju Podlasia, zostały wykorzystane na potrzeby dalszych prac projektu "Foresight technologiczny<<NT FOR Podlaskie 2020>> Regionalna strategia rozwoju nanotechnologii", w szczególności do budowy strategii rozwoju nanotechnologii na terenie województwa podlaskiego.

Programme Programme

18:00 Poster NF7

Release of nanometer-sized particles during abrasion of construction material

<u>Urszula A. Mikołajczyk,</u> Stella Bujak-Pietrek, Irena Szadkowska-Stańczyk

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Background. During abrasion of construction materials containing nanoparticles, the latter can be released to the environment. Inhalation of these particles can cause symptoms of respiratory disease in workers. The aim of the present study was to assess the concentration, surface area and mass of particles with nanometer dimensions (nanoparticles) released during abrasion of mortar. Method. The measurements of the number, surface area and mass of nanoparticles were carried out in the laboratory of a construction plant before, during the application and abrasion of nano-mortars and afterwards. The concentration of <1,000 nm nanoparticles in terms of their number was determined using a condensation particle counter (CPC), the mass concentration was determined by a monitor of aerosol concentration in air (DustTrak) and the surface concentration of the nanoparticles possibly deposited in the alveolar (A) and tracheo-bronchial (TB) regions was determined by a nanoparticle monitor (AeroTrak). RESULTS. The mean number concentration of particles determined during abrasion was 8400.6 particles/cm3 of air, while the corresponding values determined before and after the examined process was 7881.6 and 6435.8 particles/cm3, respectively. The surface area of the <1000 nm particles potentially deposited in region A was 18.99 µm2/cm3 during grinding, while before and after the operation the corresponding values were 15.73 μm2/cm3 and 13.89 μm2/cm3, respectively. For the TB fraction, the corresponding values were 5.00 µm2/cm3 during, 3.65 µm2/cm3 before and 3.13 µm2/cm3 after the procedure, respectively. The mass concentration of the particles was 0.10 mg/m3, while the corresponding values before and after the operation were 0.10 mg/m3 and 0.09 mg/m3, respectively. CONCLUSIONS. The number of particles smaller than 1000 nm released in the process increased by 6,6% while particle surface area possibly deposited in the region A has increased by 21%, and in the region TB it has increased by 37%. The mass concentration of particles does not change during the examined process. This work has been supported by the grant of the Minister of Science and Higher Education No. 5052/B/PO1/2010/38 and 7PR/236215/2011

18:00 Poster NF8

Comparison of in vitro cytotoxicity of platinum nanoparticles and cisplatin against humans glioma cells line

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Background. Gliomas are the most aggressive and common primary tumors of central nervous system (CNS). Platinum based drugs are wildly use in medicine as a chemotherapeutic agents for the treatment of tumors. However, the poor penetration of CNS is characteristic for drugs containing platinum and the chemotherapy based on platinum salt can be ineffective against brain tumors. Recently the new biologic-

ally active platinum nanoparticles (NP-Pt) can be useful as a therapeutics in gliomas cancer therapy.

The aim of the study was to investigate and compare the toxicity of NP-Pt and cisplatin and their proapoptotic or necrotic properties in examination with glioma cells line (U87).

Materials & methods. NP-Pt were characterized by electron transmission microscopy (TEM). The NP-PT and cisplatin were incubated with glioma cells. The comparison the biological properties of NP-Pt and cispatin were evaluated through morphology, metabolic activity, viability, mortality and the type of cell death of U87 glioma cell line.

Results. NP-Pt and cisplatin are toxic to glioma cells and activated apoptosis in the U87 cell line. NP-Pt treatment activated programmed cell death without activating necrosis. The effectiveness of cisplatin and NP-Pt treatment against glioma cells were mostly at the same level. **Conclusions.** The results suggest not only genotoxic and cytotoxic effect caused by NP-Pt but also the cell cycle arrest and apoptosis in NP-Pt treated cells. The comparison between effectiveness of treatment cancer cells by NP-Pt and cisplatin showed promising results for future studies. The results indicate that properties of NP-Pt might be utilized for brain cancer therapy

CEPT Poster session

Monday evening, 16 September, 19:00 Posters presented during breaks

19:00 Poster CePT1

[CEPT] Studies of nanoparticles size distribution

<u>Aleksandra E. Kędzierska</u>^{1,2}, Jacek Wojnarowicz¹, Agnieszka Opalińska^{1,2}, Tadeusz Chudoba¹, Witold Łojkowski¹

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Research at the Laboratory of Nanostructures for Photonics and Nanomedicine, IHPP PAS team is focused on synthesis and characterization of nanomaterials and their possible applications in medicine, optics, cosmetics and other industrial fields.

One of the most important issues of nanomaterials characterization is size of their agglomerates or particles in different dispersants. One of use method is Nano Tracking Analysis (NTA), from NANOSIGHT. Principle of measuring is based on detection and live visualization of a single nanoparticle diffusion in colloidal suspension. Measured particle size range from 10 nm to 2 μ m.

Other, also popular technique is Dynamic Light Scattering (DLS). In our Laboratory, we use Zetasizer NanoSeries, from MALVERN company. This measuring technique calculate size of particle from changing intensity of laser light, which is scattered at different intensities by particle movement (Brownian motions). Measured particle size range from 1 nm to $10~\mu m$.

Advantages of possibility to measure particles/agglomerates distribution with both of methods and use of standardized procedures (PN/EN ISO 17025) are dipper and better characterization of colloidal suspensions, with focus on their polydispersity, resulting in reliability of the result.

19:00 Poster CePT2

[CEPT] MSS synthesis of highly biocompatible nano-HAP with properties depending on the synthesis time.

<u>Sylwia Kuśnieruk</u>¹, Dariusz Smoleń², Jacek Wojnarowicz³, Aleksandra Kędzierska, Elżbieta Pietrzykowska¹, Tadeusz Chudoba³, Witold Łojkowski^{1,3}

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Area of bone regrowing materials has dynamically developed in recent years, yet the most popular approach to bone regeneration is still the use of autografts and allografts. Though, alternative methods are gaining momentum as researchers have embarked on a quest for more biocompatible, cell-friendly materials that could replace autografts. One of them is a Hydroxyapatite (Hap), a bioactive ceramics that is used in the form of paste and granules to fill small bone defects. For large bone gaps regrowing there are still some challenges that need to be addressed, such as low regrowth rate, poor mechanical properties, high inflammatory risk and low resorption rate. Therefore the main objective of the current regeneration medicine projects is to develop the technology for bioactive scaffold with improved shape control, better mechanical properties, bioactivity and resorbability. Aforementioned goal can be achieved through production of nonstoichiometric nanoparticles of hydroxyapatite with grain size lower than 10nm and shape close to the natural Hap, which will be used as a material for bioactive, mechanically strong scaffolds. Such nanoparticles due to their calcium deficiency and high surface to volume ratio may achieve necessary solubility level and increased osteoblasts adhesion.

The Institute of High Pressure Physics of the Polish Academy of Science (IHPP) is an expert in synthesis of doped nanoparticles with narrow size distribution, at relatively low temperatures by using Microwave Solvothermal Synthesis (MSS) technology. The MSS technology permits synthesis of nanoparticles with precise control of reaction time, temperature and pressure.

By leveraging unique MSS technology for nanoparticles synthesis, IHPP is able to synthesize innovative HAp nanoparticles using the standard reaction between calcium hydroxide and phosphoric acid. The reaction is carried out in water solution in time lower than 5 minutes. The specific surface area is almost $240 \text{m}^2/\text{g}$ the average grain size lower than 10nm with shape in the form of platelets mimicking the natural bone particles. 28 days of degradation test conducted according to norm ISO 10993-14 indicated material solubility equal $20 \text{mg/dm}^3 [1, 2]$.

- 1. Smolen D, Chudoba T, Malka I, Kedzierska A, Lojkowski W, Swieszkowski W, Kurzydlowski KJ, Kolodziejczyk-Mierzynska M, Lewandowska-Szumiel M. *Highly biocompatible, nanocrystalline hydroxyapatite synthesized in a solvothermal process driven by high energy density microwave radiation.* International Journal of Nanomedicine 2013, 1-16.
- 2. Smolen D, Chudoba T, Gierlotka S, Kedzierska A, Lojkowski W, Sobczak K, Swieszkowski W, Kurzydlowski KJ. *Hydroxyapatite nanopowder synthesis with a programmed resorption rate*. Journal of Nanomaterials 2012, 1-9.

19:00 Poster CePT3

[CEPT] Metoda ultradźwiękowego pokrywania polimerów nanocząsteczkami

<u>Sylwia Dąbrowska</u>¹, Wojciech Fidziukiewicz¹, Tadeusz Chudoba², Jacek Wojnarowicz², Aleksandra E. Kędzierska², Sylwia Kuśnieruk², Witold Łojkowski²

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Nanotechnologia jest jedną z nowszych dziedzintechnologii, wykorzystywanych we współczesnym przemyśle tworzyw sztucznych. Już niewielki dodatek cząsteczek w skali nano powoduje zmianę i poprawę właściwości, w porównaniu do tych samych materiałów w skali makro. W celu poprawy właściwości polimerów wykorzystywanych do produkcji opakowań, autorzy we współpracy z Laboratorium Nanostruktur dla Fotoniki i Nanomedycyny IWC PAN, przeprowadzili szereg badań odnośnie ultradźwiękowego pokrywania polimerów nanocząsteczkami (ZnO, HAp) .

Dodatki cząsteczek w skali nano do polimerów, powodują, m.in. zwiększenie wytrzymałości mechanicznej, hydrofobowość lub hydrofilowość materiału, czy zwiększenie fotostabilności na promieniowanie UV. By uzyskać możliwie największy stopień homogeniczności zawiesin użytych do pokrywania polimerów były one poddawane działaniem ultradźwięków.Do syntezy nanocząstek wykorzystano technologię solwotermalnej syntezy mikrofalowej (MMS), która pozwala na kontrolowanie parametrów syntezy, np. czasu reakcji, temperatury czy ciśnienia. Dzięki temu możliwe jest otrzymanie materiałów o wysokim stopniu czystości i homogeniczności w bardzo krótkim czasie.

Autorzy wykonali szereg zawiesin ZnO i HAp o różnych stężeniach, które po homogenizacji ultradźwiękowej we wcześniej ustalonych warunkach, zostały poddane badaniom rozkładu wielkości cząstek (DSL). Uzyskano i zaobserwowano nano-warstwy proszków na powierzchni polimerów, przeprowadzono obserwację struktur nano-warstw za pomocą mikroskopii elektronowej (SEM). Materiały zostały również przebadane pod kątem zdolności absorpcyjnej dla światła UV, ale również zwilżalności.

19:00 Poster CePT4

[CEPT] Nanohydroxyapatite-polylactide composites for regenerative implants

<u>Elżbieta Pietrzykowska</u>¹, Aleksandra E. Kędzierska¹, Dariusz Smolen, Tadeusz Chudoba¹, Sylwia Kuśnieruk¹, Janis Ločs, Witold Łojkowski^{1,2}

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Regenerative implants are anengineering solution which enables and gives chance for full reconstruction and recovery of patients. In orthopaedics, biomaterials with appropriate mechanical properties and surface nanotopography are in great demand. There is still much room for research on materials which enhance cell attachment, cell growth

and tissue formation. One of the most widely used biomaterials in bone tissue engineering substitutes is polymer- polylactic acid (PLA) and ceramic -hydroxyapatite (HAP). These two materials complement each other. Polymer materials exhibit biodegradable properties but they often lack desired mechanical properties. HAP ceramic on the other hand is biocompatible and osteoconductive, but pure the ceramic is brittle. The challenge is to combine those two materials and exploit the properties of both in order to obtain implants with desired mechanical and biological properties and avoid inflammatory reactions.

Our work aims to develop consolidation technology of composite nanohydroxyapatite (GoHAP, synthesised in our laboratory) with PLA The aim of our work is to improve and prepare the related technology and render it available for application in industry. Our goal is to obtain homogeneous composite with good connection between polymer and nanograin size GoHAP. The paper shows mechanical properties of consolidated composite (GoHAP with PLA polymer). Developed in our Institute, high pressure consolidation technology permits to obtain materials with mechanical properties close to the natural bone. The process is carried out in extreme pressure 1GPa and temperature under 200°C. Thanks to this, the GoHAP nanopowder structure and biological properties are preserved in the composite. The mechanical properties of this nanoceramic were investigated and the compressive strength reaches a value above 130 MPa.

19:00 Poster CePT5

[CEPT] Studies of solubility of nanoparticles and stability of their suspension

<u>Jacek Wojnarowicz</u>, Sylwia Kuśnieruk, Aleksandra Kędzierska, Elżbieta Pietrzykowska, Agnieszka Opalińska, Tadeusz Chudoba, Iwona E. Malka, Anna Swiderska - Sroda, Witold Łojkowski

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In the era of nanotechnology in the 21st century nanomaterials are increasingly used in the daily life of man. Main products of daily use such as cosmetics, detergents, paints, varnishes and electronics have in their composition nanomaterials / nanoparticles. Following the fear of uncontrolled penetration and accumulation of nanomaterials in the environment. Laboratory of Nanostructures for Photonic and Nanomedicine carries out research the fate of the stability and solubility of zinc oxide nanoparticles in suspension of natural waters. The results show that nano zinc oxide and, cobalt doped zinc oxide is agglomerating after a few minutes of its insert into the natural water. Nanoparticles of zinc oxide is more soluble than micro zinc oxide, the main factor responsible for this situation is the particle size and surface area. There are significant differences in the solubility of Zn depending on the type of water.

Study of the fate of ZnO nanoparticles in natural waters indicate the ZnO particles transform into other types of nanomaterials and increase the its size by recrystallization.

19:00 Poster CePT6

[CEPT] Solvothermal synthesis and characterization of nanomaterials in the Laboratory of Nanostructures for Photonics and Nanomedicine, Center of Bio-Nanomaterials

Sylwia Kuśnieruk, <u>Jacek Wojnarowicz</u>, Aleksandra Kędzierska, Elżbieta Pietrzykowska, Agnieszka Opalińska, Tadeusz Chudoba, Iwona E. Malka, Jan Mizeracki, Adam M. Presz, Witold Łojkowski

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Expertise of Laboratory of Nanostructures for Photonic and Nanomedicine IWC PAN lies in synthesis and comprehensive research on nanomaterials. Synthesis of nanomaterials requires the use of state of the art, advanced reactors. For more than 10 years the Laboratory of Nanostructures for Photonic and Nanomedicine IWC PAN has been developing solvothermal technologies of nanoparticles production. The process is based on the MSS Microwave Solvothermal Synthesis. The precursors of the reaction (solutions, suspensions) are enclosed in a pressure vessel and as a result of heating with the microwave energy, the temperature increases above the boiling point. The MSS process allows to prevent contamination of synthesis, by sealing the reaction vessel, which is made of chemically inert material, so an ultrapure product is obtained. The mixing effect occurs in a microwave reactor, so that the obtained product is homogeneous. MSS technology innovation is the possibility to control the size of crystallite nanoparticles in a narrow distribution of size. Thanks to this technology, we are able to obtain nanoparticles in the range from 9 to 100 nm in ultra-short synthesis time. We can obtain power density in liquid reaching up to 10 W/ml. We specialize in the production of: HAp, ZnO and ZrO2 nanopowders.

We have constructed innovative reactors MSS-1 and MSS-2 and our reactors were awarded a gold medal at:

- 1) MSS-2 International Fair in Poznan, Innovation Technology Machines Poland. 2011
- 2) MSS-1 International Exhibition in St. Petersburg in 2009. MMS-2 technical parameters:
- Maximum working pressure to 10 MPa
- Temperature up to 260°C
- Capacity 470 cm3
- Microwave Heating at 2450 Mhz
- Microwave power up to 3 kW
- Chamber material PTFE and ceramic Al2O3

Research directions of the Laboratory of Nanostructures for Photonic and Nanomedicine IWC PAN are focused on the characteristics of nanomaterials and the possibility of their application in medicine, optics, optoelectronics pharmacy and cosmetics.

The correct characterization of nanometric-materials is necessary in order to determine the properties of materials. Research in these areas requires advanced, sophisticated measuring equipment. With co-financing from the European project CePT (Centre for Preclinical Research and Technology) laboratory has purchased devices such as:

- particle size distribution and zeta potential analyzer with an automatic titrator MPT-2 model Zetasizer Nano-ZS, Malvern
- particle size distribution analyzer model NS500, NanoSight
- ultra high resolution scanning electron microscope, model ULTRA plus, Zeiss with X-ray spectroscopy analyzer model Quantax 400, Bruker
- thermal stability analyzer TGA/DSC model STA 449 E1 Jupiter, Netzsch coupled with mass spectrometry analyzer model Gas Analitycal

System QMS 403C, Netzsch and FTIR spectroscope analyzer System TENSOR27, Bruker

- viscometer analyser model DV-II+Pro, BROOKFIELD
- bubble pressure analyser model BPA-1P, Sinterface
- particle dispersion analyzer model Turbiscan Lab, Formulaction
- Opti Max^{TM} synthesis workstation for chemical and process development, Mettler-Toledo
- multi-parameter Instrument model ProLab 2000, SI Analytics, pH and conductivity measurement

In addition, the laboratory has such a measuring devices as:

- x-ray powder diffractometer model X'Pert PRO, Panalytical, and model D8, Bruker
- gas displacement density analyzer AccuPyc II 1340 Pycnometer, Micromeritics and AccuPyc 1330 Pycnometer, Micromeritics
- surface area analyzer, model Gemini 2360, Micromeritics Laboratory of Nanostructures for Photonic and Nanomedicine IWC PAN deals with the characteristics of nanomaterials obtained by our team, as well as provides research services such as analysis of density, surface area, morphology, phase composition, thermogravity of nanomaterials, surface tension, viscosity, stability and the size of nanoparticles in colloidal solutions and suspensions. We cordially invite you to collaborate with us in the area of nanoparticles synthesis and functionalization, their characterization, and applications in nanomedicine.

19:00 Poster CePT7

[CEPT] Solvothermal synthesis nano zinc oxide in microwave reactor MSS2

<u>Jacek Wojnarowicz</u>, Tadeusz Chudoba, Witold Łojkowski, Sylwia Kuśnieruk, Elżbieta Pietrzykowska, Aleksandra Kędzierska, Agnieszka Opalińska

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Laboratory Nanostructures for Photonics and Nanomedicine IWC PAN is an expert in the synthesis of doped nanoparticles with small size distribution using microwave technology solvothermal synthesis (MSS). MSS technology allows precise control parameters of nanoparticle synthesis such as reaction time, temperature and pressure. We obtain the zinc oxide particle size distribution in solvothermal synthesis which can be expressed as follows:

 $(CH_3CO_2)_2Zn \rightarrow ZnO$

Synthesis of nano-zinc oxide doped with cobalt take place in solution of ethylene glycol during 12 minutes in microwave reactor MSS2. Specific surface doped ZnO is 10-50 m2/g and crystallites size are in a range from 20 to 150 nm.

Microwave, pressure chemical reactor MSS2 is used to carry out solvoand hydrothermal microwavesynthesis, wherein is obtained nanopowders with specific particle size and morphology. Used solutions in the reactor overtake a global level and allows to obtain ultrapure nanoparticles in the production and experimental scale. Developed high-pressure seals provide insert of substrates, running processes, released products in track made from chemically inert materials.

Tuesday, 17 September

REGISTRATION

Tuesday morning, 17 September, 8:30

NANOFORCE INFO DAY: Funding and future of nanotechnology

Tuesday morning, 17 September, 9:00

Chair: Helmut Schmid

9:00

Invited oral

Nano Velos - biotechnological start-up founded by GIZA Polish Ventures. How VC fund may turn your idea into the company with a global perspective

Dariusz Smoleń

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NanoVelos is a bio-medical start-up founded by GIZA Polish Ventures VC fund. Our technology is a universal drug delivery system based on polysaccharide nanoparticles. The encapsulated drug or substance is connected with our nanoparticles by the covalent bonding which allows us to produce stable particles able to circulate in vascular system even for few days and have a fully controlled drug release mechanism. Application of NanoVelos drug delivery system for known drugs improves therapy efficiency and reduce side effects. NanoVelos system is directed to drugs, cosmetics, dietary supplements and veterinary products.

From the beginning our team was focused on searching for practical solutions in the fields of medicine and pharmacy so that we chose VC funding support as the way to transfer our idea from laboratory to market products.

Giza Polish Ventures (GPV) Fund - our investor, is a Venture Capital fund with a professional back-up provided by the Israeli company Giza Venture Capital. The company was established in 1992 and currently manages funds worth over USD 600 million. Over the past 20 years on the market, Giza VC has made almost 100 investments in Israel, Europe, the United States and Asia and has achieved more than 35 successful exits. Giza Investment professionals contribute with wealth of expertise and experience in communications, semiconductors, information technologies, enterprise software, the life sciences and medical equipment, clean technologies, media, the Internet and entertainment. Thanks to its experienced team, Giza has become one of the leading Israeli VC groups and has led to the following winning investments: Zoran – digital entertainment, M-Systems (currently Sandisk), Libit (currently TI), Actimize (acquired by Nice), Danen Technology, BioLineRX and others. Moreover, Giza VC established an office in Singapore in 2001 and in recent years has been pursuing investment opportunities in Taiwan-based companies. Giza VC is also actively engaged in supporting the Israeli company portfolio in developing and partnering in Taiwan and South-East Asia.

GPV Fund mainly invests in early stage companies and technology start-ups in Poland. The aim of the Fund is to use the broad investment experience and international network of business contacts of the Giza VC company as well as expert knowledge of the Polish team on identifying local companies and teams with extraordinary international development potential.

The aim of presentation is to describe you through the NanoVelos story, how VC fund may turn your scientific idea into the company with a global perspective. It will present the challenges and opportunities from both sides perspective. Finally it will suggest you a few tips how to become a successful inventor in partnership with a global VC fund.

Programme Programme

9:20 Invited oral

NANOFORCE - promoting the successful exploitation of Nanotechnologies across Central Europe

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The involved 8 Areas, inhabited by more than 150 million people, realize a combined GDP of more than \in 3,000 bn, with more than 120,000 manufacturing companies operating in their territories. At national level, there are more than 200 universities and research centres. Overall, in the 8 regions of NANOFORCE Partners, there are more than 140,000 researchers which are engaged in the frontiers of the knowledge.

NANOFORCE is trying to create a common platform within 20 scientific & industrial parks and incubators, a new tool named "NanoDeals Generator" to support the creation of academic spin offs and industrial start ups, enlarging the availability of financial resources offered by more than 20 seed & early stage venture capital funds already active in the 8 Areas.

Through a proactive web source (see http://www.nanoforceproject.eu) entrepreneurs, researchers, experts and managers are solicited to newtork in order to discuss and advance "deal", offer their experiences, looking for young talents and search for promising initiatives such as Public & Private Partnerships.

Finally, at the end of the Project, a proposal to set up an Interregional Ventture Capital Fund will be defined and advanced to potentially interested investors and stakeholders in order to try increase the available "patient moneys" to financially back promising entepreunerial initiatives presented by young researchers in the 8 Areas.

9:40 Invited oral

Horizon 2020 - new programme, new approach?

Grażyna Omarska

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The aim of the presentation is to give a very compact general overwiew of Horizon 2020 - the new EU programme for research and innovation (2014-2020): the structure, budget and what new and what already-known elements one may expect there.

10:00 Invited oral

Prezentacja CEZAMAT

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10:20 Oral

Nanotechnology development strategy for Podlasie region

Joanna Ejdys, Joanicjusz Nazarko

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This paper presents the results of research relating to the Podlaskie Province and concerning the key nanotechnology and research directions that will contribute to sustainable socio-economic development of the region to the highest degree, and the potential usage of nanotechnology to the benefit of the existing and new industries. This survey was carried out within the framework of the Technology Foresight "NT FOR Podlaskie 2020» Regional strategy for the development of nanotechnology project. The project was financed by the Innovative Economy Operational Programme, Priority I. Research and development of new technologies, Activity 1.1. Support of scientific research for the development of knowledge-based economy, Activity 1.1.1. Research projects using foresight methods. The beneficiary of the project is the Bialystok University of Technology, and the Implementing Authority - Information Processing Centre (OPI). In line with the general methodology of the research project, the execution of the main objectives of the research was based on a panel of experts, whose task was to create, analyze and synthesize the knowledge relevant to the matter. The results of the individual panels were presented in the following monographs: · Conditions for the development of nanotechnology in the Podlaskie Province. Results of the STEEPVL and SWOT analyses, · Key nanotechnologies in the economy of the Podlaskie Province · Nano-science for the development of the Podlaskie Province Directions of development of nanotechnology in the Podlaskie Province. Maps. Routes. Trends, · The Podlasie nanotechnology development strategy up to the year 2020, According to the authors of the «NT FOR Podlaskie 2020» project, nano-innovation may be the key to the competitiveness of the Podlasie companies and the main driver of productivity growth of the region, while at the same time protecting the unique environmental values. Innovative nanotechnology is an opportunity to modernize the existing endogenous potential of the region through the application of these technologies to the existing industries in the region (with high potential for its use). Implementation of the nanotechnology solutions can also influence the process of diversification of the regional specialization and build a new one. The results of the project are available at the project website www.ntfp2020.pb.du.pl.

10:40 Invited oral

New Research Initiatives in Poland

Krzysztof J. Kurzydłowski

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COFFEE and POSTERS

Tuesday morning, 17 September, 11:00

NANOFORCE INFO DAY: Advanced Nanotechnolgies 1

Tuesday morning, 17 September, 11:20 *Chair: Joanna Ejdys*

11:20

Polish Research Projects

Industrial perspectives of nanometals

Malgorzata Lewandowska

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11:40

Oral

Nano-Titanium for medical applications

<u>Grigory Dyakonov</u>¹, Irina Semenova², Alexander Polyakov, Ruslan Z. Valiev²

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Formation of ultrafine-grained structure in metallic materials using severe plastic deformation techniques leads to modification of their properties and makes potential areas of their application much broader. A considerable progress in recent years has been achieved in the sphere of fabrication of semi-products from nanostructured commercially pure titanium [1, 2]. High strength and fatigue endurance of nanostructured titanium makes this material very attractive for medicine. In this work, beside investigation of mechanical properties and microstructure parameters, a special attention was paid to biomedical studies and advantages of application of nanostructured titanium in medicine, particularly for dental implants. Formation of a nanostructure in titanium Grade 4 was conducted using the ECAP-Conform technique and subsequent drawing. Semi-products from nanostructured titanium Grade 4 had the ultimate tensile strength of 1300 MPa and fatigue endurance limit of 620 MPa at a load of 10⁷ cycles. Samples of nanostructured and coarse-grained titanium were subjected to surface modification using the mechanical polishing and acid etching. Study of surface topography after surface modification was performed. Corrosion properties of titanium in an artificial physiological solution were analysed with a use of a cyclic voltammetry technique. It was demonstrated that the surface topography and properties of an oxide layer of titanium influence the bio-compatibility parameters. And nanostructured titanium possesses enhanced bio-compatibility as compared to the coarse-grained one. Thus, a combination of high strength and fatigue properties as well as enhanced bio-compatibility of nanostructured titanium makes it an attractive material for medical implants.

References:

- 1. R.Z. Valiev, I.P. Semenova, V.V. Latysh, H. Rack, T.C. Lowe, J. Petruzelka, L. Dluhos, D. Hrusak, J. Sochova, Nanostructured titanium for biomedical applications, Adv. Eng. Mater., 10, No. 8 (2008), pp.B15-B17.
- 2. D.V. Gunderov, A.V. Polyakov, I.P. Semenova, G.I. Raab, A.A. Churakova, E.I. Gimaltdinova, I. Sabirov, J. Segurado, V.D. Sitdikov, I.V. Alexandrov, N.A. Enikeev, R.Z. Valiev. Evolution of microstruc-

ture, macrotexture and mechanical properties of commercially pure Ti during ECAP-conform processing and drawing. Mater Sci & Eng A 562 (2013) pp. 128–136

11:55

Invited oral

Nano-Silver as Interesting Material for Various Applications

Helmut Schmid

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Nano-silver has many beneficial properties that enable a high number of different applications.

- 1. The antimicrobial properties can be used for product applications in medicine, the construction and sanitary sector, coating of handles, controls and worktops up to antifouling coatings for ships to name just a few.
- 2. Excellent electrical conductivity predestinate to applications for conductor pathways and printed electronic circuits. This effect can be even further improved in combination with CNT's.
- 3. A high reflection rate in the range of thermal-IR is suitable for indoor as well as for outdoor applications. Heat insulation surfaces can help to save energy and improve indoor comfort. External applications help to avoid excessing heating of facades which leads to cool buildings.

The paper is focussed to product applications in the described areas, showing research and development activities of FhG-ICT and covers all steps from nano-particle-synthesis, stabilization, analysis and system-integration under special consideration of nano-safety throughout the entire product life-cycle.

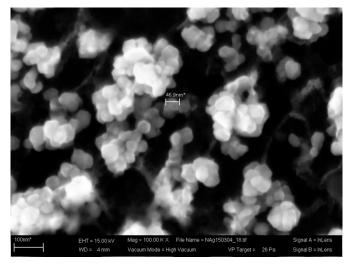


Figure 1: REM-record of nano-silver particles used for product functionalization.

12:15 Oral

Green Technology and Pilot Unit for Manufacturing of Ultra Clean Concentrated Colloids of Nano Metals

<u>Vitaliy V. Sadokhin</u>¹, Ludmila Kisterska¹, Olga B. Loginova¹, Jacek Wojnarowicz²

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An innovative green technology of plasma dispergation of various metals and one-step productive manufacturing cycle of nano metal concentrated colloids are described. Metals nano particles size and distribution control is fulfilled by managing of main parameters of localized plasma sputtering process. Physical model of metal nano dispergation by plasma jet and nano particles implantation into carrier liquids in vacuum is developed. The study of aggregation of nano metal particles in water solution of concentrated colloids on the glycerine bases as function of time is completed. The new productive multicycles unit for super concentrated nano metal colloids is created. The case of successful practical application of nano silver colloid in glycerine is described.

LUNCH

Tuesday afternoon, 17 September, 12:30

NANOFORCE INFO DAY: Advanced Nanotechnologies 2

Tuesday afternoon, 17 September, 13:30 *Chair: Ludmila Lipinska*

13:30

Invited oral

Coating anti-bacterial nanoparticles on flat and curved surfaces, and fighting resistant bacteria by employing the sonochemical method

Aharon Gedanken

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Sonochemistry is an excellent technique to coat functional nanomaterials on various substrates, and imparting new properties to the substrates.

After a short demonstration of coating NPs on ceramics and stainless steel, I'll present the coating of textiles such as polyester, cotton, nylon, and nonwoven. In all cases a homogeneous coating of NPs was achieved. Silver is known for generations as antibacterial, and indeed the Ag NPs have killed the gram-negative *E. Coli* (strain 1313) as well as the gram-positive *Staphylococus aureus* (strain 195) bacteria very efficiently¹. Lately, since the FDA shows less enthusiasm towards Ag we have moved to NPs of ZnO, CuO and MgO as antibacterial agents. They were coated on the above-mentioned fabrics and showed excellent antibacterial properties². The coated textiles were examined for the changes in the mechanical strength of the fabric. A special attention was dedicated to the question whether the NPs are leaching off the fabric when washed repeatedly. Lately the coated ZnO NPs on cotton

underwent 65 washing cycles at 92 0 C in water in a Hospital washing machine, no NPs were found in the washing solution and an the antibacterial behavior was maintained. Our vision is that all the textiles in the Hospitals of the future will coated by antibacterial NPs.

We have demonstrated recently that we can fabricate NPs of salts (NaCl, CuSO₄, KI, KBr) from their aqueous solution³. This work was continued and we have shown that in the same way we can produce organic nanoparticles (R6G, Anthracene, Polyaminopropyl Biguanide, Amylase, DNA, RNA) from their aqueous or from their solution in any other solvent. This study was continued to antibiotic compound such as gentamicin, tetracycline, ampicillin, and methicillin. We have prepared NPs of these four antibiotic materials from their aqueous solution. More importantly, these nanoparticles exhibit antibacterial activity, even against pathogens that are resistant to the antibiotic in its non-nanometric form.

13:50 Invited oral

Nano-inks for printing of electric circuits for microelectronic technology

<u>Andrzej J. Mościcki</u>¹, Anita K. Smolarek¹, Tomasz Fałat², Andrzej E. Kinart¹

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Actual microelectronic industry needs new and advanced materials and technologies for reason of miniaturization and very high reliability. Nano sized metals as silver or copper as a electrically conductive fillers gave possibility to develop new types of formulations called Nano Inks for packaging technologies and for making electrical circuits on rigid substrates and flexible polymer foils. The paper present information about possibility use of nano size silver for electronic packaging technologies.

Each kind of ink, needs different nano silvers is shown in Fig. 1 and Fig. 2.

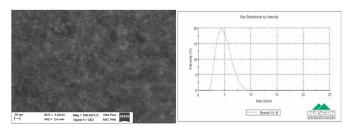


Figure 1. SEM picture of the smallest nanoAg and particle size distribution by "Malvern Zeta Sizer".

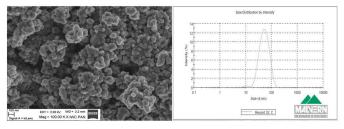


Figure 2. SEM picture nanoAg and particle size distribution by "Malvern Zeta Sizer".

As it is well known, nanosized silver does not exist without protection layer around each particles. When metallic nanoparticle inks are heated the dispersants first, and protective layer the second, should be effectively removed allowing the remaining active metallic nanoparticles to be successfully sintered. We have tested several types of nanosilvers with different coating shells. Basing on Ag with paraffin and polymer coating the conductive inks were tested in Amepox lab.

The base properties of the inks are shown in Table 1.

	Ink with 3-8 nm silver filler	Ink with 60 nm silver filler
Viscosity	7,5 – 10,5 mPas	5 – 12 mPas
Sizes of silver particles	3 -8 nm	50 - 60 nm
Percentage of silver filler	40 - 60%	20 - 30%
Surface tension value	28,5 – 32,5 mN/m	~ 35 mN/m
Specific gravity	$1,1-1,3 \text{ g/cm}^3$	$0.8 - 1.0 \text{ g/cm}^3$
Sintering temperature	230°C	up to 150°C
Electricaly resistivity	$(4-6) \times 10^{-6} \Omega \text{cm}$	5 x 10 ⁻⁶ Ωcm
Printing possibility	High temperature flexible substrates	Low temperature flexible substrates

In this paper, we report on the basic properties of different inks, conditions for making printed stuctures and other possibilities as microvias interconnections (Fig.3).

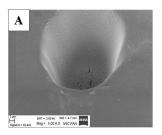




Figure 3. A – Interconnection through microvia-hole by using ink. B – Printed structures on polymer foil.

14:10 Invited oral

${\bf Superhard\ metal\ nanoparticles:\ a\ pathway\ to\ applications?}$

Eugen Rabkin¹, Seok-Woo Lee³, Julia R. Geer³, Dan Mordehai¹, Oleg Kovalenko¹, William D. Nix²

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We demonstrate that single crystal Au and Fe micro- and nanoparticles produced by solid state dewetting technique can attain a strength level approaching the theoretical shear strength of respective metals. This is related to the perfect atomic structure of the particles, their faceted morphology and scarcity of potential sites for dislocations nucleation. High level of strength was achieved both in nanoindentation experiments with sharp pyramidal diamond tip, and in microcompression tests with flat diamond punch. The possibility of purely elastic deformation of the particles with engineering strains as high as 10 % was demonstrated. Atomistic computer simulations of the deformation process reveal the mechanisms of nanoparticles plasticity. It was also

shown that irradiating the particles with Ga ions drastically reduce their strength.

We propose employing single crystalline metal nanoparticles as a reinforcing phase in protective coatings.

14:30 Invited oral

Application of the Ultra-Plus Zeiis FE-SEM in the study of nano and bio-nano structures

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14:50 Polish Research Projects

Scanning Electron Microscopy Investigations and Analysis of Nanostructures

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Several examples will be given on application of Field Emission Scanning Electron Microscopy in the studies of nanopartciles and nanostrcutres, including polymers. Application of BSE detector, inlens detector and other detectors will be ilustrated. Methods to optimise the image will be explained.

15:10 Invited oral

Applications of Helium and Neon Ion Microscopy; from Imaging Shale Rock to Machining Graphene

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Ion microscopy using helium or neon beams created from a gas field ion source (GFIS) shows great potential and flexibility for many imaging and nanofabrication applications. With helium or neon, sub-10 nm structures can be routinely fabricated in a variety of materials including sensitive materials such as graphene. Additionally, the beamsample interaction dynamics of helium/neon ion beams offer unique contrast and stunning surface detail at sub 0.5nm lateral resolution.

The helium ion beam introduces a unique opportunity for high precision patterning in graphene. High aspect ratio nanoribbons have been machined down to 5 nm without damage. Due to the nature of imaging with the helium ions, non-destructive imaging of graphene with excellent surface sensitivity can be achieved both before and after patterning. Helium and neon beams have also been used for a diverse range of other nanofabrication applications. Solid state nanopores for DNA sequencing devices with holes down to 3 nm in diameter and aspect ratios greater than 10:1 have been created in a variety of materials. In plasmonic applications, dislocation damage to surrounding structural elements is greatly reduced compared to gallium FIB when using helium or neon, thus allowing work on delicate and sensitive membranous materials. Plasmonic devices with nearly vertical sidewalls have been patterned in films demonstrating machining precision of better than 5 Direct write lithography using commercial resists HSQ and PMMA resulted in line features as narrow as 4 nm. Finally, the use of helium and neon ions beams for circuit edit applications is being

developed. Deposition of conducting and insulating materials creates features smaller than those obtained with gallium FIB and with better electrical properties due to the absence of gallium.

A gallery of helium ion microscopy imaging and nanofabrication results will be presented to showcase the capability and performance of this novel microscope.

COFFEE and POSTERS

Tuesday afternoon, 17 September, 15:40

NANOFORCE NANO-Deals: Advanced nanotechnologies 3

Tuesday afternoon, 17 September, 16:00 *Chair: Ludmila Kistersky*

16:00

Oral

Textile with silver nanoparticles.

Aneta Popławska¹, Ladislav Torcik²

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Predictions are that in the following years nanotechnology will be widely applicated in industrial processes, and that the amount of such products will greatly increase. NanoTrade, Ltd. was established in Olomouc in the year 2004. The company's mission is to commercialize innovative materials, products and processes based on nanotechnology, which are the result of research, technology and development of laboratories in the Czech Republic and abroad. NanoTrade, Ltd. is currently focusing on: collaboration with R&D institutions in the Czech Republic, Poland, Austria, Germany and Great Britain, testing and developing new materials, technologies and application processes in all levels, providing tests of certification in collaboration with certification authories, production of new nanomaterials, analysis, definition and projects preparation, definition of production program, marketing, consulting services, trading activity in nanotechnology products. Another kind of NanoTrade activity is creating clothes using nanofibres. Nowadays the increasing market competition and customers' conscious choices are causing the change of products and a neccesity of finding new solutions. Popularity of spending time actively created a need for using specific clothes and NanoTrade products seem to provide a good response to these requirements. This presentatnion shows a new collection of garments dedicated especially to people who do sports and workout at the gym. Collection made with Nanosilver® and Coolmax® fibres is a modern high performance sportswear with great cooling and antibacterial properties. The Coolmax fibre is very effective at conducting sweat away from the body without getting soaked wet. The moisture is transported to the surface where it rapidly evaporates, keeping the underlying skin dry. Nanosilver® yarn, the second component of the garment, prevents growth of bacteria and the resulting unpleasant odour. Silver has been proven to be effective against bacteria and moulds. This feature remains active for the lifetime of the garment. Fiber composition: 52% Coolmax®, 48% Nanosilver®

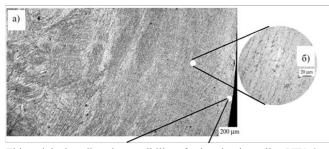
16:15 Oral

Rules of creating of the double layered structure (bedding) in deformation strengthening layer in the process of titanium alloys spherical products machining

<u>Dmytro A. Sergach</u>¹, Yurij A. Tsekhanov², Sergij Y. Sheykin¹, Dmitriy V. Karih²

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This article describes the possibility of using titanium alloy VT1-0 and VT6 in manufacturing spherical heads for human's hip joint. Low mechanical properties and increased tendency to setting with the majority of materials impede the application of these materials, so the production of titanium heads seems possible on condition of modifications of effective area by applying nitriding. It is known that the properties of the nitrided layer determined by the structure of the surface layer of titanium product. Shredding of the structure and strengthening of the surface layer of the product with CSPD before nitriding, leads to the increasing of the dislocation density and an expansion in the area of inter grain borders, which extends the depth of the diffusion layer and increases its hardness. We developed a spherical technological scheme of run-up pieces of VT1-0 and VT6 flat surfaces, which strengthens the surface layer of 2 or more times at a thickness of the surface layer of 1 mm or more. Experiments show that during processing, heating of the work piece surface up to 150 ° -200° takes place; combined with volume compression in the contact zone, this provides a high plasticity of the material and cause no defects. Plastic deformations in these conditions lead to the creation of finely dispersed structure and nanostructure in the surface layer, favourable to its further diffusion saturation with nitrogen. Recrystallization and consolidation of crushed grains do not occur

16:30

Polish Research Projects

Production of nanocrystalline structure in steels by heat treatment process

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The paper describes a method to produce a nanocrystalline structure in medium-alloyed, commercial steels by use of specially designed heat treatment process. Conventional heat treatment processes of steel consisting on quenching and tempering do not permit reducing the grain size below 300nm of phases formed. It was however shown, that a nanocrystalline structure consisting of grains with thickness below

100nm may be formed in steels with specific chemical composition as a result of isothermal bainitic transformation. To obtain a nanobainitic structure it is necessary to conduct heat treatment process in precisely defined conditions. These are defined on the basis of knowledge of the kinetics and critical points of phase transformation occurring in steel. The general scheme of the heat treatment consisted on austenitizing followed by quenching to the a temperature lying in the lower zone of bainite transformation and isothermal holding at this temperature for a time allowing completion the bainite transformation. Four kinds of commercial steels have been chosen for investigations: bearing steel, spring steel, hot work tool steel and structural steel. In order to establish the parameters of austempering heat treatment, the processes of phases transformation occurring in selected steels have been investigated by means of dilatometric measurements. The determined kinetics and critical points of phase transformations were used to establish the proper parameters of austempering treatment that would lead to a nanobainitic structure. The structure of steels after austempering treatment was studied by light microscopy and transmission electron microscopy. It was shown, the austempering leaded to the formation of carbide free nanobainitic structure in bearing steel, spring steel and hot work tool steel. This structure was composed of plates of bainitic ferrite with thickness below 100nm embedded in retained austenite matrix. The thickness of retained austenite films separating the plates of bainitic ferrite was below 50nm. The obtained structure can be thus considered as a nano-composite structure. The structural steel after austempering heat treatment exhibited a sub-micrometric structure of lower bainite containing about 11.3% of retained austenite, mostly in form of blocks. This microstructure results from insufficient carbon content 0.35% wt. in this steel. In order to increase the carbon content steel samples were subjected to the vacuum carburizing process. As a result the carbon content was doubled in the surface layer of structural steel. The austempering process leaded in this layer to a nano-composite structure similar to the structure of other investigated steels. The mechanical properties of steels with nano-composite structure were determined by hardness measurements, tensile tests and Charpy impact tests. It was shown, that nano-composite steels exhibited high value of strength parameters combined with relatively high ductility. Moreover it was shown, that the carburized layers in structural steels exhibited higher wear resistance after austempering treatment than after the conventional quenching and tempering process commonly applied in carburized steels.

The technological limitations associated with the production of steels with nanocrystalline structure have been described and the possibilities of eliminating these restrictions have been analysed.

The study was accomplished within the Structural Project "Nanocrystalline structure formation in steels using phase transformation" NANOSTAL (contract no. POIG 01.01.02-14-100/09). The project is co-financed by European Union from the European Regional Development Fund within Operational Programme Innovative Economy, 2007-2013.

16:45 Invited oral

Au/anionic clays nanoarchitectonics as novel photocatalysts for hydrogen generation from water, under solar irradiation

Mihaela Birsanu¹, Hermenegildo Garcia², Kiyoshi Okada³, Magda Puscasu¹, <u>Gabriela Carja</u>¹

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We present here nanoarchitectonics of nanoparticles of gold/LDH anionic clays as active plasmonic photocatalysts for H₂ production from water/methanol mixtures (80:20) by using solar light, at room temperature [1]. The fabrication of Au/LDHs, as self-assemblies, is based on the formation of nanosized gold on the surface of the anionic clay during the clay structural reconstruction in the aqueous solution of AuCl₃ or Au(O₂CCH₃)₃. No organic compound is involved either in the clay synthesis or in the formation and the organization of Au NPs. The derived mixed oxides are obtained from Au/LDHs by calcination at 750°C. All the catalysts exhibit strong light absorption at around 550 nm due to surface plasmon resonance (SPR) of nanosized gold. Results reveal that not only the size-dependent characteristics of Au nanoparticles (e.g.: $D_{Au},\,S_{Au})$ but also the composition of the LDH clay are important parameters for establishing the catalysts effectiveness for H₂ production under solar irradiation at room temperature. Au/clay nanoarchitectonics can open new opportunities for progress in the development of complex plasmonic nanostructures as solar-light driven photocatalysts for clean H₂ production.

[1] J. Mater. Chem A, accepted manuscript; DOI: 10.1039/C3TA11569K.

17:05 Polish Research Projects

Safe nanomaterials of spinel structure for lithium-ion secondary batteries

<u>Ludwika Lipińska</u>¹, Monika Michalska¹, Ryszard Diduszko¹, Bartosz Hamankiewicz², Michał Krajewski², Andrzej Czerwiński², Dominika Ziółkowska³, Krzysztof P. Korona³

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The market of lithium-ion batteries develops rapidly. These batteries are commonly used in portable electronic devices, such as: cell phones, notebooks, tablets, ipods, media players, as well as in hybrid electric vehicles (HEVs), plug-in hybrid electric vehicles (PHEVs), owing to its high energy density and working voltage, long lifecycle, small dimensions and weight. Especially, the last applications require large format batteries and the their safety becomes a key issue.

Lithium manganese oxide ($LiMn_2O_4$) of spinel structure has been extensively studied as a cathode material for Li-ion batteries. Application of $LiMn_2O_4$ has several advantages like: low cost, easy preparation, non-toxicity, high potential (4V vs. lithium metal), a satisfactory capa-

city, high-energy density, low self-discharge and high thermal and structural stability. In spite of these advantages, LiMn $_2$ O $_4$ suffers from a serious capacity fading during charge-discharge cycles, which is unacceptable in commercial applications. This problem can be caused by several factors: manganese dissolution, electrolyte decomposition at high potentials, the Jahn-Teller distortion at the state of a deep discharge and lattice instability. There are various strategies to improve structural stability of LiMn $_2$ O $_4$. One of them is a partial substitution of manganese ions by other divalent or trivalent metal elements e.g. Fe, Co, Ni, Al. Another way is to use nonstoichiometric lithium manganese spinel like – Li $_{1+x}$ Mn $_{2-x}$ O $_4$. Modifying their surface by ceramic coating is practiced, too. Recently, a new class of safe anode materials – lithium titanium oxide (Li $_4$ Ti $_5$ O $_1$ 2) of the spinel structure is investigated. This compound is very suitable for LiMn $_2$ O $_4$ used as cathode.

We succeeded in obtaining nanocrystalline compounds of spinel structure by modified sol-gel method. Examples spinels: $LiMn_2O_4$ (stoichiometric, nonstoichiometric and substituted) [1] and $Li_4Ti_5O_{12}$ will be showed. The details of sol-gel synthesis will be presented at the Symposium. All mentioned above compounds are safe (resistant to uncontrolled oxidation) and environment friendly.

As-synthesized samples were characterized by X-ray powder diffraction (XRD), scanning electron microscopy (SEM) and simultaneous thermal analysis (differential scanning calorimetry DSC and thermogravimetry TGA), Raman spectroscopy. Also the electrochemical properties of synthesized powders were examined. All obtained materials were nanostructured, single phase and exhibited good specific capacity. Some of them showed improved cycle-ability. Application of nanocrystalline electrode materials has many additional advantages: high surface area, new active reactions, decrease the path length for Li ion transport, reduce the specific surface current rate, improved stability, enhanced specific capacity. The modified sol-gel synthesis turned out to be a very effective way for production of the electrode materials for lithium ion batteries.

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17:20 Oral

New photonic composite materials for aircraft applications

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In the past decade, advanced composite materials have been widely used in a variety of load-bearing structures such as rotor blades, aircraft fuselage and wing structures. The unique properties of composite materials such as their high strength-to-weight ratio, high creep resistance, high tensile strength at elevated temperatures and high toughness have been attracting increasing interest in numerous automotive, aerospace, and sports applications.

Composite structures are frequently subjected to external excitations over a variety of vibration frequency ranges. Such a dynamic interference may cause the structures to suffer from fatigue damage and/or catastrophic failures. A typical composite material fails in a sequence of transverse micro-cracking, delamination and fiber failure. Non-destructive evaluation (NDE) techniques have been developed to detect internal or invisible damage. To the traditional NDE techniques belong: ultrasonic scan, an eddy current method, an X radiography, an acoustic emission method, and passive thermography. The NDE techniques are effective in detecting damages in materials and structures, but it is difficult to use them in operation due to the size and weight of the devices. Therefore, there is a strong interest in development of smart composite structures with integrated optical fiber sensors which would allow in-situ monitoring of both the manufacturing process and the service life. Compared to the traditional NDE techniques, fiber-optic sensors offer unique capabilities as: monitoring the manufacturing process of the composite parts, performing non-destructive testing once fabrication is complete, and enabling health monitoring and a structural control. Due to their minimal weight, small size, high bandwidth, high sensitivity, immunity to electromagnetic interference, possibility to operate in a hazardous environment and in the presence of electric currents fiber-optic sensors offer significant performance advantages over traditional sensors.

Composite structures are made of two or more components. This feature allows for introducing optical fiber sensors into the composite material. Typical optical fiber sensors are based on highly birefringent fibers where the output signal is a periodic function of the external strain or on fiber Bragg gratings where the output signal is a linear function of the external strain but this type of the fiber needs an additional equipment to encode the output signal. Due to a variety of sensing fibers and different responses to external perturbations there are a lot of possibilities to construct fiber-optic strain gauges precisely adjusted to particular needs and applications.

In our research many types of the fiber optic sensors embedded in the composite materials have been investigated. We observed that interactions between composite material and optical fibers during manufacturing process are very significant. Lamination process can dramatically change strain sensitivity of the highly birefringent fibers. By using soft coatings of the optical fibers we protect them against stress introduce to the composite materials during manufacturing process. Additionally this research allows us to propose the new hybrid fiber optics sensing system which can measure separately temperature and strain.

COFFEE and POSTERS

Tuesday afternoon, 17 September, 17:35

NANO-INFO DAY POSTER SESSION

Tuesday evening, 17 September, 18:00 Posters presented during breaks

18:00 Poster P1

Studies on the inhibition mechanism of the human coronavirus NL63 replication by polycations

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Human coronavirus NL63 (HCoV NL63) is generally classified as a common cold virus. It may cause severe upper and lower respiratory tract diseases. Thus, there is a need to develop effective methods of preventing and treating these infections.

The aim of this study was to synthesize and explore anticoronaviral activity of polymer-based compounds. Four polycations, i.e. N-(2-hydroxypropyl)-3-trimethylammonium chitosan chloride (HTCC), hydrophobically-modified HTCC (HM-HTCC), O-(2-hydroxypropyl)-3-trimethylammonium poly(vinyl alcohol) chloride (HTPVA) and poly(allylamine hydrochloride) (PAH) were investigated. In order to evaluate the inhibitory activity of the tested polymers in vitro study using LLC MK2 cells and ex vivo study using human airway epithelium (HAE) cultures were performed. The cytopathic effect (CPE) was correlated with a quantitative RT PCR based assay. The cytotoxicity was examined by an XTT assay and a Neutral Red assay.

The results show that cationically modified polymers (HTCC, HM-HTCC, PAH and HTPV) are effective inhibitors of human coronavirus NL63 replication. Evaluated polymers show relatively low cytotoxicity. What is more, their specificity is high. None of the compounds inhibited replication of other human viruses (influenza A, hMPV, adenoviruses, enteroviruses, and human herpes virus type 1).

Mechanism of antiviral activity is complicated. Size and charge of polymers suggest that they are not actively transported into the cell. Analysis of interaction between HTCC polymer and the recombinant ectodomain of the S protein showed binding, resulting in the formation of protein-polymer complexes.

Acknowledgements

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18:00 Poster P2

Study of newly developed materials for use in endodontics regarding to its biocompatibility and biofunctionality

<u>Joanna Karaś</u>¹, Andrzej Olszyna², Lidia Ciołek¹, Ewa Zaczyńska³, Anna Czarny³, Piotr Jadczyk⁴, Barbara Umińska-Wasiluk⁴, Jerzy Garcarek⁵, Bogusława Żywicka⁵

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Have been developed and manufactured biomaterials belonging to family based formulations Portland cement, as in the case of ProRoot MTA or MTA Angelus, while easy to be activated powders with suitable fluids to produce homogenous pastes.

It were developed and manufactured materials with contrast radiology, optimal consistency and good workability, adequate mechanical strength for use in endodontic treatment.

Also preferably be assessed fact that aqueous extracts of these biomaterials exhibit a pH of 10.2 and rising to 11.52 after 24 h. Developed and manufactured materials release significant quantities of calcium ions which should result in a biological environment stimulation of reparative processes pulp and repair processes in the bone tissue surrounding the root of the tooth. Based on this results may be assumed that developed materials should provide good root end filling and pulp capping.

Cytotoxicity developed cements for use in endodontics was performed by indirect contact with the monolayer cell culture L929 fibroblasts. It was no toxic effect of developed materials.

Also biomaterials showed no genotoxic potential against strains of *Salmonella typhimurium TA 98 and Salmonella typhimurium TA 100* with or without metabolic activation in rat liver microsomal fraction S9 in the Ames test.

The developed biomaterials have antibacterial activity against the strains of *Streptococcus mutans*, *Streptococcus sanguinis*, *Escherichia coli*, *Staphylococcus aureus*, *Pseudomonas aeruginosa*.

Based on the results of the *in vivo* evaluation stated that the obtained biomaterials were characterized high biocompatibility tested with the muscle tissue of rats and bone tissue of rabbits. Moreover they initiated in the soft tissue cementogenesis process.

Developed biocompatible biomaterials can be used in dentistry for endodontic treatment.

18:00 Poster P3

Introduction of nanocosmetics into assortment of PPH Kosmed Zbigniew Leżański

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PPH Kosmed as the small/medium-sized enterprise studied and tried to bring into the market nanocosmetics to enhance efficiency and

competitiveness of their products. Use of nanocosmetics is restricted because of potential toxicity of nanoparticles released during their application, as the released nanoparticles can penetrate into the skin. Therefore, nanocosmetics currently selected for the use do not release nanoparticles that can penetrate into the skin. Consequently, their health effects are evaluated basing on the analysis to characterize their activity on the skin surface. However, the introduction of new or modified nanocosmetics requires additional studies towards activity of nanoparticles which could penetrate deep into the skin.

Moreover, the restrictions on the use of nanocosmetics is due to their potential impact on the environment. Previous studies take account of the action of the released nanoparticles as active species and do not include the deactivation of the species under the environmental conditions

It can be seen that the green/sustainable chemistry requirement fulfil conditions required for the placing of the new or modified nanocosmetics on the market.

Preparations currently used for the nanocosmetics correspond to the nanoparticles of transition metals and their oxides such as Ag, Au, TiO2, ZnO, Fe2O3 as well as relevant complexes structured to enhance processability or useful properties of the nanocomposites. Nowadays, efficient nanocosmetics were introduced including fullerene c60 and their derivatives. The fullerene act as antioxidants deactivating reactive oxygen species generated by ultraviolet radiation. Successful structuring of nanocosmetic results in improved performance and competitiveness, but require effective method of preparation of the nanoparticle suspensions as well as the additional studies throughout the above mentioned health- and environmental effects. For example, the above presented oxides reduce the effect of ultraviolet radiation selectively, depending on their nanoparticle sizes and the size distribution and, moreover, operate as stable components. Similarly, efficient are nanocosmetics containing fullerenes or derivative thereof. On the contrary, the effects of conventional cosmetics containing organic UV filters are neither so selective nor stable. On the other hand, the nanocosmetic preparation requires effective deagglomeration and dispersing that are hard to perform because of fast agglomeration of the active nanoparticles while the conventional homogenization proceeds smoothly.

Consequently, the objective of this work was the selection and testing of the efficient deagglomeration and dispersing of the nanoparticles which can be introduced into the R&D cycle of nanocosmetics.

As a result, continuous synthesizing of silver nanoparticles was successfully studied using modular microreactor of the Institute of Microtechnology in Mainz, Germany.

Generally, the modular microreactors coupled witch microstructured reactors allow study and development of chemical synthesis from the micro-scale to production scale, optionally via efficient deagglomeration and dispersing of reactants. A number of novel applications utilizing the micromixers can be seen to emerge in cosmetics. Efficient mixing of reactants and control of their concentrations result in optimised condition for the nanoparticle synthesis leading to relatively short nucleation times in the reaction mixture of high homogeneity. Therefore, microreactor systems produced narrower nanoparticle size distribution compared to the batch reactors of conventional size using similar ratios of reactants and experimental conditions. In the comparison, the use of microreactors reduce polydispersity of nanoparticulate products, and increase both inter-run reproducibility and throughput. Because, as mentioned above, the nanoparticles show size-dependent properties in the cosmetics, hence the accurate control of the particle size is essential here. Additionally, investigations dealing with the preparation of semi-solid pastes for cosmetic were undertaken at various industries. In a sum, the microreactors coupled with microstructured reactors with efficient mixing/dispersing module provide an excellent tool for synthesis of the useful nanoparticles. Generally, the coupled microreators that are fully automated and analytically equipped present

a powerful synthesizing and screening tool for flow chemistry with which to perform substrate selection and process condition optimization at intermediary stages. Therefore, use of these microreactors enable intensification of the R&D cycle of nanocosmetics according to the both green/sustainable chemistry and market requirements.

18:00 Poster P4

Control of hydroxyapatite/β-tricalcium phosphate ratio of Mg-substituted biphasic calcium phosphate

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Biphasic calcium phosphate (BCP) bioceramics composed of hydroxyapatite (HAp) and of β -tricalcium phosphate (β -TCP) in various ratios show a significant benefits compared to others calcium phosphate (CaP) materials, i.e., the ability to control the balance between the bioactivity and solubility in the living body, which guarantees the stability of bioceramics during the bone ingrown process. Incorporation of ions into the structure of CaP is important for the control of characteristics, the increase in bioactivity of material, and ion delivery able to act on bone diseases etc. It has been demonstrated that the incorporation of Mg sensibly affects apatite crystallization and its thermal stability, promoting the formation of β -TCP and thus forming BCP.

The aim of this work is to characterize Mg-substituted BCP prepared through modified wet chemical precipitation of $Ca(OH)_2/Mg(OH)_2$ and H_3PO_4 , using different approaches: A - control of phase ratio of HAp/β -TCP by changing concentration of Mg into synthesis products; B - control of phase ratio of HAp/β -TCP by altering the synthesis technological parameters. Thermal stability, phase and chemical composition, and morphology of obtained products were evaluated by differential thermal analysis (DTA), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FT-IR), field emission scanning electron microscopy/energy dispersive X-ray spectroscopy (FE-SEM/EDS).

FE-SEM/EDS mapping indicated that Mg was uniformly spread out in the CaP products. As evidenced by DTA and XRD the amount β -TCP phase is dependent on the Mg substitution level and Mg found to stabilize the β -TCP phase while sintering at higher temperatures.

It is possible to obtain Mg-substituted biphasic calcium phosphates with various and reproducible phase and chemical compositions by wet chemical precipitation method by precise control of composition of starting materials as well as synthesis technological parameters.

18:00 Poster P5

Biofunctionalized Gold Nanorods for Enhanced Stability and Mucin-1 Targeted Cancer Theragnosis

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Recent studies have highlighted the overexpression of mucin 1 (MUC1) in various epithelial carcinomas and its role in tumorigenesis. These mucins present a novel targeting opportunity for nanoparticle-mediated

photothermal cancer treatments due to their unique antenna-like extension. MUC1 antibodies were introduced into the albumin coating of polydopamine-primed gold nanorods (DP-NRs). As an analog of the mussel-mimetic adhesive catechol L-3,4-dihydroxyphenylalanine (DOPA), DP forms an adhesive platform for the deposition of albumin and MUC1 antibodies, achieving a surface that is stable, bioinert and biofunctional. Two-photon luminescence confocal and darkfield scattering imaging revealed targeting of MUC1-BSA-DP-NRs to MUC1+ MCF-7 breast cancer and SCC-15 squamous cell carcinoma cells lines. Treated cells were exposed to a laser encompassing the near-infrared AuNR surface plasmon and assessed for photothermal ablation. MUC1-BSA-DP-NRs substantially decreased cell viability in photoirradiated MCF-7 cell lines vs. MUC1- MDA-MB-231 breast cancer cells (p < 0.005). Agents exhibited no cytotoxicity in the absence of photothermal treatment. The facile integration of the coating may provide novel prospects for multimodal cancer therapy via albumin-based therapeutics such as Abraxane. Future work will probe the in vivo efficacy of these NRs and whether albumin's involvement in the gp60-mediated endothelial transcytosis pathway may be exploited to enhance intratumoral delivery.

18:00 Poster P6

Nanocomposite alginate fibres as components of composite materials for medicine

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During realisation of a developmental project entitled 'Manufacturing of biodegradable, hybrid composites based on lactide nanofibres and alginate nanocomposite fibres for medical applications', a technology of manufacturing of different types of alginate fibres was developed, including nanocomposite alginate fibres for medical applications. A series of alginate fibres were manufactured during the project that differed in structure of the material, type of the implemented nanoadditive and properties:

- · Fibres from calcium alginate characterised by high sorption properties, capable of absorbing exudation from wound.
- · Fibres from copper alginate characterised by the capability to generate negative static electric charge, which results in decreasing the feeling of pain.
- · Fibres from calcium alginate containing silver nano-additive characterised by high sorption properties and bacteriostatic properties.
- Fibres from calcium alginate containing Fe₃O₄ nano-additive used as an implantation material, they enable imaging of the material after its engrafting.
- · Fibres from calcium alginate containing hydroxyapatite nano-additive showing osteoconductive action due to the presence of a bioactive nano-additive.
- Fibres from sodium alginate containing silver nano-additive characterised by solubility in water and bacteriostatic properties.

On the basis of the manufactured alginate fibres, composite materials were obtained, intended for wound dressings or implantation, characterised by an oriented therapeutic action. In case of materials for wound dressings the following composites were manufactured which, depending on the raw material content, are characterised by differend pace of changing into gel form, differentiated sorption properties and showing antibacterial action:

- A composite intended for a general-purpose wound dressing, combining specific action of alginates, meaning supporting of the process of wound healing, with antibacterial action manufactured from fibres from copper alginate, calcium alginate and sodium alginate containing silver nano-additive.
- A composite intended for wound dressing for healing skin changes without exudation with antibacterial acion, showing capability to decrease a feeling of pain by a patient, manufactured from fibres from copper alginate and from fibres from calcium alginate containing silver nano-addivite.
- A composite intended for infected or hard-healing wounds in early stage of healing, manufactured from fibres from copper alginate and from fibres from sodium alginate containing silver nano-additive.

In case of materials for implantation, there was a composite manufactured from alginate fibres and nano-fibres from polylactide intended for healing perforations of bone tissue, characterised by the action of supporting the process of bone tissue regeneration and giving the possibility of its imaging after implantation.

The fibres and composites developed during realisation of the project were manufactured in the Department of Material and Commodity Sciences, and Textile Metrology of the Lodz University of Technology, equipped in specialised laboratories with apparatuses necessary for manufacturing and examining of innovative composite materials.

Acknowledgements:

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18:00 Poster P7

Plasmonic nanostructures for applications in SERS of bacteria

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Continuous threat of terrorist attack with chemical (CWA) or biological warfare agents (BWA) has been stimulating research on development of rapid and sensitive methods for their detection for many years [1]. The development of such methods for the detection of bioagents is also important for medical diagnostics of infectious diseases. Traditionally, PCR method has been used for bacterial identification. However, despite the recent advances, it still requires relatively long time of analysis [3,4]. Among other methods for detecting bioagents, spectroscopic methods, such as fluorescence or Raman spectroscopy, have been shown to be applicable for biological detection. Recently, one of the Raman spectroscopy techniques, Surface-Enhanced Raman Spectroscopy (SERS), based on enhancement of scattering on plasmonic

nanostructures have been shown as a great tool for spectroscopic detection of bacteria [2,4]. However, the key challenge is the development of sensitive and stable SERS substrates as well as methodology of SERS measurements in case of bacteria.

In this work we present results of studies on the surface enhanced Raman spectroscopy of Bacillus Atrophaeus (Bacillus globigii, BG) – simulant to Bacillus Anthracis [4]. The SERS spectra of BGs were obtained using various types of nanostructures – drop-deposited layers of gold, silver and $\rm SiO_2@Au/Ag$ core-shells nanoparticels and assembled monolayers of noble metal nanoparticles [5]. The example of SERS spectra of BGs is presented in Figure 1. Preliminary studies have shown that the signal enhancement is strongly dependent on type and morphology of SERS substrates, as well as the way sample for SERS measurement is prepared.



Figure 1. SERS spectrum of BGs obtained with use of silver nanoparticles.

The financial support from NCBiR under grant No. O N507 282540 and NCN under Grant No. 2011/03/D/ST5/06038 is greatly appreciated. References

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18:00 Poster P8

Intervertebral disc endoprosthesis – structure, tests, production technology and preparation for clinical applications

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Full title of the project: Intervertebral disc endoprosthesis – structure, tests, production technology and preparation for clinical applications

Name of the program: Development Project of the 10th Competition

Application year: 2009

Realization years: 2010-2013

The list of key parameters of the project:

1. Metal Forming Institute, Poznań;

2. Warsaw University of Technology; Faculty of Production Engineering; Institute of Mechanics and Printing;

The objective of the project includes preparation of the structure and production of clinical prototypes of an intervertebral disc endoprosthesis for clinical applications. The scope of works conducted includes identification of characteristic (anatomical) features of the muscle-skeleton segment of the spine in various individuals with the use of CT and MRI as well as preparation of methodology of parametrical designing of the intervertebral disc endoprostheses. A relevant trend of works under the realized project includes optimization of the intervertebral disc endoprosthesis production process and production of prototypes of an intervertebral disc implant for tests on animals and clinical evaluation.

During realization of the project they prepare technical documentation in the form of parametrical 3D models and 2D drawings as well as technological processes for production of physical models (one of the generative techniques) and production of prototypes with the use of CNC machines.

The project includes designing of two independent three-element structures of the lumbar intervertebral disc endoprostheses: IN-OP/LSP.1101 (Fig. 1) and PW-BSZ (fig. 2) [1, 2]. Owing to appropriate selection of geometrical parameters, each of the structures is to reflect operation of a natural disc. By complying with requirements relating both to reflection of movement in L4-L5 spine segment as well as correct connection with intervertebral bodies, they show anatomical functional properties (custom design). Moreover, use of mobile inserts ensures variability of position of the rotating axis during operation of the endoprostheses and, thus, better reflection of natural disc kinematics [1].

Positive verification of the selection of the geometrical and ergonomic features of endoprostheses and satisfactory results of the numerical strength analyses of the implant-bone couple allowed a commencement of production of technical prototypes. The prototypes were made of materials used successfully for implants – cobalt based alloy (Co28Cr6Mo), titanium based alloy (Ti6Al4V) and nickel-free austenite steel (316L) with the use of three various technologies (fig. 3). Endoprostheses of Co28Cr6Mo and Ti6Al4V alloys were made with the use of CNC machines. Endoprostheses components were made of Co28Cr6Mo and Ti6Al4V alloy powders with the use of Selective Laser Melting technology. 316L steel forgings were made with the use of gear forging method and, then, subject to CNC machining. The quality of friction surfaces after polishing was characterized by the $R_{\rm a}$ roughness below 0.05 μm .



Fig. 1. INOP/LSP.1101 endoprosthesis – components: a) anteverted, b) retroverted [3]

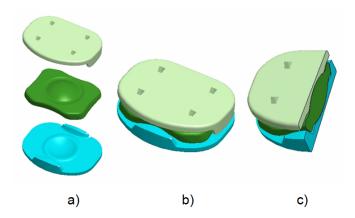


Fig. 2. PW-BSZ endoprosthesis: a) components, b) anteverted endoprosthesis, c) cross-section in the sagittal plane







Fig. 3. INOP/LSL.1101 endoprosthesis components made of various materials with the use of various technologies, the condition of surface after polishing: a) upper plate, CoCrMo alloy, CNC machining, b) insert, 316L steel, CNC machining following gear forging, c) bottom plate, Ti6Al4V alloy, SLM technology

An endoprosthesis implanted in the human organism should be characterized by biocompatibility and high durability in order to fulfill its functions properly. The durability of presently used endoprostheses is limited due to friction between the cooperating components. In order to verify the applied production technologies activities aimed at friction and wear tests of a selected endoprosthesis structure were undertaken. The verification included:

- determination of the influence of technology of endoprosthesis production upon the friction resistances,
- determination of wear as a function of number of cycles worked depending on the applied technology of production of metallic components,
- testing of the roughness changes of the cooperating endoprosthesis surfaces as a function of the number of cycles,
- microscopic analysis of friction surfaces and wear products resulting from friction.

The object of friction and wear tests constitutes six sets of INOP/LSP.1101 endoprostheses. Three of them were made of Co28Cr6Mo alloy through machining. Another three were made with the use of SLM technology and Co28Cr6Mo powder. Each of the endoprostheses worked on the simulator for 1 000 000 cycles. The friction loop was loaded with a force of maximum instantaneous value of 1500 N. The swiveling angle of the upper plate for bending was: +80, the swiveling angle for hyperextension was: -50. The one-sided angle of axial twisting was: \pm 30. The frequency of movement was 1.25 Hz. The tests were conducted in a distilled ultraclean water environment (fig. 4).



4. A view of the simulator for the testing of spine implants [4, 5]

Results of friction and wear tests showed that, regardless of technology used, the value of friction coefficient in the tribological system of the tested endoprostheses was similar and on average amounted to 0.278 (± 0.013) for CNC and 0.273 (± 0.019) for SLM. However, the wear processes in SLM were more intense. This is proved by the mass wear values. The average mass wear of the CNC endoprostheses amounted to 19.2 mg, whereas for SLM endoprostheses the mass wear decrease was 25.5 mg. Greater wear of the SLM components is reflected in the roughness of the friction surfaces. For example, the average roughness of the inserts made with the use of CNC method upon completion of the tribological tests was 1.07 μ m, whereas the Ra roughness of the SLM insert was characterized by the average roughness of 1.14 μ m.

The main objective of the Project is preparation of the structure and production of an intervertebral disc endoprosthesis prototypes used for clinical applications and complying with the license requirements of the European Union. It is highly probable that the project will contribute to the introduction of a new and competitive product into the market and formulation of a business policy based on identification of market and social demands as regards the improvement of human functions in the society, vocational and physical activities as well as economic aspects.

The area of advanced technologies clearly determines the horizontal policy, guidelines of the European Union and national strategic and research programs. One of the priorities of the National program for Scientific Research and Development Works is the 2nd Research area - HEALTH. The most serious health threat for the population in Poland includes civilization diseases such as, inter alia, primary as well as secondary degenerative lesions, i.e. those resulting from previous disorders of joint mechanics on the basis of childhood diseases or mobile organ injuries. An increase in the number of joint endoprostheses implanted is connected with progressive ageing of the population, as the percentage of degenerative lesions is the largest among persons above 65 years of age. Apart from ageing of the population, the main cause of most of the diseases of the bone and joint system are hazards resulting from the change in the human environment and unfavorable lifestyle trends. A rapid progress of knowledge makes it possible to get to know civilization diseases better, identify high risk groups, address intervention activities properly and detect diseases earlier, which improves the treatment effectiveness. A significant element of the project includes biotribological tests and acquaintance with the mechanism of mutual influence of individual elements of the endoprostheses.

The significance of the project lies in its innovative nature, which involves the fact that intervertebral disc implants constitute a new invasive method of treatment of spine diseases. The invasive method of treatment of spine diseases (an intervertebral disc endoprosthesis) has been applied quite recently. In Poland this method is rarely used despite high social demand for treatment of such types of diseases. This work allows creating fully functional implants. The prototypes will be subject to any tests (in accordance with the EU guidelines) that will specify, whether a given product complies with the requirements for medical devices.

The project contributes to a development of collaboration with a local manufacturers of implants and medical devices. The realization of the project will contribute to a development and potential implementation of the method of treatment of spine diseases on a local scale with the use of the solution proposed by INOP. A significant activity includes strengthening of the position of Polish research centers as regards works on designing of human joint endoprostheses, which will also be reflected in the inspiration for development of new patents. The realized project constitutes a platform for establishment of a close collaboration with manufacturers of endoprostheses and medical community.

The project will contribute to introduction of a new and competitive product into the market and formulation of a business policy based on identification of market and social demands as regards the improvement of human functions in the society and vocational and physical activities. Economic aspects are also significant, because, as the literature analysis has shown, due to the fact that spine diseases affect young people more and more often and eliminate them from active vocational life, the problem of spine diseases is not only a social issue, but also a general national economic problem. The main advantage for the Metal Forming Institute is the opportunity to prepare and implement the method of treatment of spine diseases on a local scale. The project also allow an improvement and extension of activities of the Institute towards the scientific research works on joint endoprostheses initiated in 2004.

A significant solution of the collaboration oriented at the use of results of the project in practice for a new medical device may also be international markets. This activity is oriented at the western markets and countries of the Eastern Europe. A significant orientation includes crisis-affected countries due to the economic aspect and the fact that the project will contribute to a more favorable economic balance of the proposed solution as compared to the presently used solutions.

The project results attract companies of the medical sector having a developed distribution network and scientific and research facilities (construction and technology back-up facilities and state-of-the-art laboratories) able to create and absorb innovations of the greatest growth potential and export capabilities. The companies are strictly connected with the medical community, scientific and research institutions and they actively participate in the implementation of the results of the realized projects. As regards the increasing competitiveness, the interested companies, above all, had to prepare personnel responsible for the realization of tasks in all stages of the management.

One of the forms of establishment of collaboration oriented at the use of the project results in economic practice or other activities is the promotion of the project. The promotion of technology is mainly addressed to enterprises of the orthopaedic sector to an interested group of practitioners. A significant aspect of the promotion of the realized works is the dynamically developing Polish market. INOP prepared a long-term program connected with the promotion of the project results. The promotion involves the use of the following: the Internet platform (website), promotional events (fairs, international and local conferences, seminars, conferences — congresses with panel discussions, target workshops, Delphi methods and scenarios), a newsletter, educational

and training materials (online, e-learning, study visits, debates) as well as a commencement of a debate relating to the project in light of the presented test results. The Center for Excellence of the Metal Forming Institute organizes workshops and trainings for representatives connected with the said issues of the project. The information about the project has been put on the Institute website. The tests results were promoted and propagated and collaboration with entrepreneurs was established during the International Fair in Poznan and at the booth of the INOP Center for Excellence.

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18:00 Poster P9

Label free diagnostic tool based on Whispering Gallery Modes

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The success in preparing monodisperse fluorescent microparticles with almost ideal spherical shape during the last 20 years allowed the observation of so called Whispering Gallery Modes (WGM) after excitation of the particles by light. Therefore, several publications and patents were published claiming the usage of such particles for the detection of biomolecules like DNA, RNA, antibodies or other Proteins by specific interaction with the functionalized particles surface. Due to changes in the surface refractive index by adsorption of such molecules, the WGM shifts very sensitive, allowing a quantitative analysis of such molecules. This analytical method is completely independent of premodification of the bio-molecules with fluorescent dyes or other markers.

The aim of this study is preparation and functionalization of microparticles for label free diagnostics by means of Whispering Gallery Modes.

A comparable method on planar surfaces the Surface Plasmon Resonance is already widely used by the BIACORE instruments. But due to the expensive instrument and the complicated preparation of special chips the costs per measurement are very high. Results of my investigation showed that WGM analysis could be remarkably lowered by using microparticles in microfluidic cells.

18:00 Poster P10

Synthesis and luminescence properties of Ln(III) doped $Gd_2(WO_4)$

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Trivalent lanthanide ions show interesting properties due to the optical transitions within 4fⁿ- electronic mainfold. In luminescent material field, phosphors based on lanthanide ions play an important role because of sharp absorption and emission lines [1]. Nowadays, the three basic emission colors: blue, green and red are usually obtained with rare-earth materials. Eu(III) and Pr(III) ions exhibit intense red emission, Tb(III) ions show intense green emission. However, Dy(III) ions are known as two primary color phosphors. Luminescence spectra of Dy(III) present two characteristic bands at 480 nm (blue) and 575 nm (yellow) [2].

In the search for new luminescent materials we suggest to use tungstate as a host for lanthanide ions because of their specific energetic structure and optical properties of the WO₄²⁻ group which absorbs UV radiation and emits visible light [3,4].

In order to enhance the Ln(III) emission the energy transfer process can be achieved by using $Gd_2(WO_4)_3$. The luminescence properties of the phosphors were analyzed by means of emission and excitation spectra, including results of luminescence lifetimes of Ln(III) ions. Additionally, we used hydrostatic pressure applied in a diamond anvil cell (DAC) to change the interaction of localized electrons with the lattice and to change the energy of the localized states of Ln(III) with respect to the band edges [2].

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18:00 Poster P11

Investigations of Raman Spectroscopy of C-Pd films for hydrogen sensor applications

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The work presents the results of Raman studies of carbon nanofilms containing Pd nanostructural films. The samples were obtained in two step method: physical (PVD) and physical/chemical deposition (PVD/CVD). Due to CVD modification of the PVD substrate films carbon nanofoam is formed. The analysis of Raman spectra confirms the presence of various carbon allotrope forms . For the PVD samples fullerite and nanographite grains are observed. For the CVD samples the amorphous carbon and/or graphite-like structure are observed.

C-Pd films containing Pd nanocrystals can be applied as active layers in proposed gas sensor applications

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18:00 Poster P12

Application of silica-metal core-shell nanostructures for Surface Enhanced Raman Spectroscopy

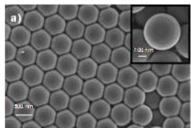
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Studies of the silica–noble metal core-shell particles are fascinating mainly because of their unique structures and interesting physicochemical properties, which make them attractive for a variety of applications [1,2]. These structures are of great interest in biological and biochemical studies, for instance, in cancer diagnostics or pathogens identification, spectroscopy, e.g., surface-enhanced Raman spectroscopy (SERS), optical signal modulation and photonics.

In this paper we report an effective recipe for preparation of core-shell nanostructures consisting of spherical siliceous cores with diameter *ca.* 500 nm and gold or silver external shell layer (Fig. 1). We found the optimal process conditions to obtain products with desired size and morphology. Optical properties (extinction) of obtained silica-gold and silica-silver nanostructures were examined in wavelength range from 200 to 800 nm. SERS activity of prepared nanostructures was tested using standard compound – p-mercaptoaniline and revealed enhancement of the order of 10^6 .



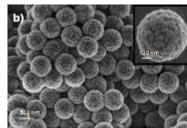


Fig. 1. Siliceous cores of nanostructures (a), silica-gold nanostructures (b) and silica-silver nanostructures (c)

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18:00 Poster P13

Development of spherical carbon structures for carbon dioxide capture

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In recent times a lot of attention was focued on nanoporous spherical carbon structures because of their unique properties [1]. Their exhibit thermal and chemical resistance, easy preparation, tunable pore structures and relative low cost. These advantages make them attractive for various applications in the fields of environmental protection (industrial capture of carbon dioxide), catalysis and energy storage [2].

In this work we have shown an effective recipe for preparation of monodisperse carbon spheres with diameters in the range 100-1000 nm and extended micropore structure. Synthesis of carbon spheres was based on the ammonia-catalyzed polymerization of resorcinol and formaldehyde in the ethanol-water phase [3]. The polymerization products upon subsequent heat-treatment for improving cross-linking of polymers and carbonization yielded carbon spheres. The KOH-activation process was performed in order to enhance microporosity carbon spheres. The obtained materials morphology was characterized by scanning electron microscope (ULTRA ZEISS, Germany). Adsorption properties were determined based on low-temperature nitrogen adsorption-desorption isotherms. The calculated value of the specific surface area was larger than 2500 m2/g and total pore volume was ca. 1,1 cm3/g. Carbon dioxide (CO₂) adsorpton isoterms were obtained at 0°C on ASAP 2020 system. The obtianed carbon spheres exhibited very high CO₂ uptake of 7,5 mmol/g. The interesting properties of fabricated micro-porous carbon spheres make them potentially useful in many practical applications.





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Acknowledgements

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18:00 Poster P14

Polystyrene coated gold nanoparticles developed for use as an optical sensors

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The core-shell polymer- metal nanoparticle hybrids have a great potential for designing a new advanced materials with unique properties combining the properties of metal core and polymer shell. One of the most important size-related phenomena observed for gold nanoparticles (AuNPs) is the surface plasmon resonance (SPR). The SPR frequency strongly depends on the size and shape of nanoparticles, dielectric properties of the surrounding chemical environment and inter-nanoparticle coupling interactions. [1] Additionally, appropriate modified AuNPs are excellent platform for Surface Enhanced Raman Spectroscopy (SERS). Thus AuNPs are promising to potential applications in highly sensitive chemical and biological sensors.[2] Simultaneously polymer shell in such materials improves stability and surface chemistry of core nanoparticles.[3] There are two major approach of synthesis of core-shell polymer hybrids: "grafting-from"[4] and "grafting-to"[5] methods combined with controlled/living polymerization: anionic polymerization [6], ATRP [7] and RAFT polymerization

We report a new method for the preparation of core-shell nanostructures based on the covalent attachment of polymer chains to the gold nanoparticles covered by TEMPO radicals [8] during nitroxide mediated polymerization of styrene. The obtained material demonstrates strong surface plasmon resonance (SPR), narrow size distribution and uniform dispersion of metal cores in polymer matrix. The obtained nanohybrids have been characterized by X-ray photoelectron spectroscopy (XPS), thermogravimetric and elemental analyses, UV-ViS, FTIR spectroscopy and TEM observation. SEC analyses showed that the polymer shell in the obtained structures is built from polymer chains with narrow molecular weight distribution (PDI<1.2).

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18:00 Poster P15

Improved Stöber synthesis of nanosized silica in O/W microemulsion

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Stöber method is widely employed to obtained silica particles of different size [1]. Originally, micrometer-sized particles have been obtained, but nanosized ones can be synthesized using microheterogeneous medium; microemulsions seem to be particularly useful for such purpose [2]. In the case of water-in-oil microemulsion, spatially constrained droplet of water act as template, determining size of nanoparticles formed. Usually, the reaction medium is composed of cyclohexane, alkylphenol ethoxylate, water, and tetraethoxysilane (TEOS) and the sol-gel process is induced by the addition of an aqueous ammonium hydroxide [3]. In some cases, medium-chain alkyl alcohol (pentanol or hexanol) was added as co-surfactant. During the process, partial expulsion of water from the microemulsion phase may occur, resulting in formation of second phase of bulk water which, causing bimodal size distribution [4].

Present contribution describes modification of standard synthetic process. Oil-in-water microemulsion was formed using heptane, 2-ethylhexanol, TWEEN®85 and TEOS. After some specified incubation time, ammonium hydroxide was added and thereaction mixture was stirred for 24 hours at room temperature. Prior to synthesis, pseudoternary diagram was created for oil-rich area and Winsor IV region was identified. These microemulsions were used for synthesis of silica particles. Resulting SiO₂ particles were characterized by dynamic light scattering, electrokinetic measurements, specific surface area measurements and scanning electron microscopy. Particles' diameter was ranging between 150 and 600 nm, usually monodisperse distribution was obtained. The specific surface area of nanoparticles was about 250–300 m²/g. Notably, productivity per unit volume of solution was 3 to 5 times higher than for previously reported procedures.

Our method can be extended, because polymeric or monomeric materials can be added to dispersed aqueous phase. In our studies, β -cyclodextrin and hydroxyethylcelluse have been used,, giving particles between 40 and 160 nm, wht the surface area larger than 300 m²/g.

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18:00 Poster P16

Theoretical explanation of unzipping process of WS₂ nanotubes

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The results of the theoretical analysis of experimental data of unzipping of multilayered WS₂ nanotubes by consequent intercalation of lithium atoms and organic molecules are presented. The extension of the tube was described in the framework of elastic shell theory with parameters evaluated from the *ab initio* calculations. In assumption that the driving force of attraction organic molecule is its interaction with the intercalated Li ions, the *ab initio* calculation of connection of organic molecule with Li⁺ was carried out. In addition, non-chemical interaction of dipole of organic molecule with array of negative point charges represented Li⁺ was taken into account. The comparison between energy gain from the interaction and elastic strain energy allows to evaluate the value of tube layers deformation after the organic molecules implantation. The *ab initio* molecular dynamics simulation confirmed the estimations and shows that stretched nanotube unzip into the WS₂ nanoribbon.

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18:00 Poster P17

Structural and electrochemical studies on LiMn₂O₄ cathode material for LIBs coated with ceramic oxides

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Spinel structured lithium manganese oxide (LiMn₂O₄) is one of the promising cathode material for transportation and large scale batteries due to its low cost and easy preparation, environmental friendliness, high discharge potential (4V vs. Li/Li⁺) and good safety compared with the lithium cobalt LiCoO₂ or lithium nickel LiNiO₂ oxides. Hovewer the LiMn₂O₄ electrodes at the 4V potential (vs. Li/Li⁺) region suffers from capacity fading during charge-discharge cycles, especially at elevated temperature (50-60°C), which limits the application in

commercial lithium-ion batteries. The capacity loss could be attributed to three main factors: (1) dissolution of manganese Mn^{2+} ions; (2) Jahn-Teller (J-T) distortion effect; (3) decomposition of organic solvents in electrolyte. Recently, several investigations have been proposed to resolve this complicated problem including metal doping at Mn-sites (Fe, Ni, Cr) and surface coating with metal or ceramic oxides. Finally, it should to improve the capacity retention and minimize the surface area of LiMn₂O₄ contacting with the electrolyte.

In our current work, we investigated and compared the electrochemical performance of $LiMn_2O_4$ coated by various ceramic oxides. The low temperature chemical synthesis (LTCS) was used to modify the surface grains of LMO.

Firstly, pure stoichiometric nanopowder of $LiMn_2O_4$ was prepared by modified sol-gel method using lithium and manganese salts. Secondly, we used LTCS for the surface modification by ceramic oxides (like i.e. La_2O_3 , CeO_2 , SiO_2) of LMO spinel.

X-ray powder diffraction (XRD) and Raman spectroscopy were used to characterize the crystal structures of all samples. The particle size and morphology were observed by: SEM, HRSEM, TEM. Also the electrochemical tests were performed.

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18:00 Poster P18

Synthesis of nano-Li $_4$ Ti $_5$ O $_{12}$ decorated by silver nanoparticles as an anode material for lithium ion batteries

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Lithium-titanium oxide ${\rm Li_4Ti_5O_{12}}$ of spinel structure is one of the promising negative electrode (anode) material to replace a costly and unsafe graphite. ${\rm Li_4Ti_5O_{12}}$ (LTO) is cheaper and more safe compared to graphite, due to high potential of 1.55 V versus ${\rm Li/Li^+}$ which prevent metallic lithium plating on negative electrode during overcharge. Owing to no structural change during lithiation and delithiation processes ("zero-strain" electrode) the material shows excellent cyclability. However, ${\rm Li_4Ti_5O_{12}}$ suffers from lower theoretical specific capacity compared to graphite, which is 175 mAh/g and its insulating character, which prevents using it in high current applications. The conductivity of LTO spinel can be greatly improved by various surface modifications, cation doping or preparing this material in the nanocrystalline form.

In this context, we have prepared nanocrystalline lithium-titanium oxide ($\mathrm{Li_4Ti_5O_{12}}$) and modified its surface using silver metalic nanoparticles. Also our studies demonstrate, that highly dispersed Ag nan-

oparticles on the surface of Li₄Ti₅O₁₂ grains, greatly improve their high-rate capability and cyclability.

All the obtained powders have been characterized by a numbers of methods: X-ray powder diffraction (XRD), Raman spectroscopy, X-ray Photoelectron Spectroscopy (XPS), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM). Also the electrochemical tests were performed.

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18:00 Poster P20

Synthesis of graphene-like structures in hybrid organicinorganic polymers

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Hybrid polymers represent a particular class of polymers, which main chains consist of organic and inorganic moieties. Due to such a feature, they have properties of the both components above. Hence stands the major premise of their research and of the development of hybrid polymer-based materials. Hybrid polymers are intensively studied and widely used in medicine, computing and analytical technique. They are also in use as drug carriers and metal particle-carrying substance, as well as membranes and semiconductors.

Polymers were synthesized on the basis of polyphenylenes and metal clusters and the polymerization mechanism has been proposed. Such materials have graphene-like cycles in their structures as confirmed by WAXS data. Inorganic clusters are located between grapheme layers. Clusters include stoichiometric and non-stoichiometric oxides as confirmed by X-ray photoelectron spectroscopy. There is a possibility to control characteristics of graphene-like structures adjusting temperature and pressure of polymerization. Properties of hybrid materials are dependent on degrees of transition of organic component of metal-complex oligomeric precursor into graphene-like state.

18:00 Poster P21

Studies the size of palladium nanoparticles in Pd-C films annealing at different temperatures.

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Methods of Transmission Electron Microscopy (TEM) were used to research carbon porous-palladium composites. The investigated composites were obtained in two different processes: Physical Vapour Deposition (PVD) and Chemical Vapour Deposition (CVD). The relation between an average size of palladium particle and the time and also temperature were carried out. The samples were annealed at different temperatures -Alpha from 500°C to 750°C and Alphain three different times that is: 5 min, 10 min and 30 min in argon atmosphere. Activation energies were calculated from the Arrhenius equation and obtained values for PVD and PVD/CVD samples is: 64.6 kJ/mol and 38.8 kJ/mol respectively. Determination of activation energy would be useful in controlling the growth of palladium grain. Significant differences in this values was caused that palladium particles obtained in the PVD/CVD process, were encapsulated in graphite. AlphaThose graphite planes are formed in temperatures above 600°C and blocks the growth of palladium particles.

ACKNOWLEDGEMENTS

This research was co-financed by the European Regional Development Fund within the Innovative Economy Operational Programme 2007-2013 ("Development of technology for a new generation of the hydrogen and hydrogen compounds sensor for applications in above normative conditions". No UDA-POIG. 01.03.01-14-071/08-06 and "Analytical high resolution transmission electron microscope for nanoscience, nanotechnology and spintronics" No POIG.02.01-00-14-032/08.

18:00 Poster P22

Synthesis and luminescent properties of Sr₂SiO₄:Eu²⁺ codoped with Ti⁴⁺ ions.

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In this contribution luminescence and luminescence kinetics of $\rm Sr_2SiO_4:Eu^{2+}$ and $\rm Sr_2Si_{0.95}Ti_{0.05}O_4:Eu^{2+}$ are studied. The samples were synthesized using solid state method. $\rm Sr_2SiO_4:Eu^{2+}$ and $\rm Sr_2Si_{0.95}Ti_{0.05}O_4:Eu^{2+}$ have strong luminescence which consists of two partially overlapping broad bands with maxima at 480 nm and 570 nm. The bands are attributed to the $4f^65d^1 \rightarrow 4f^7$ transitions in $\rm Eu^{2+}$ occupying in $\rm Sr_2SiO_4$ crystal lattice two different $\rm Sr^{2+}$ sites, respectively ten-coordinated SI and nine-coordinated SII. Contribution of these two bands to the total luminescence can be controlled by choosing excitation wavelength.

Besides described above two strong emission bands, which decay in microseconds range, Sr_2SiO_4 : Eu^{2^+} exhibits also luminescence that lasts at room temperature for few seconds after stopping the excitation. The persistent luminescence is relatively weak at ambient conditions but can be easily seen in dark with eye. In the case of the samples codoped with Ti^{4^+} the persistent luminescence is significantly enhanced. We have proved that besides the concentration of Eu^{2^+} and Ti^{4^+} also the form of the titanium precursor used in synthesis influences on the persistent luminescence phenomenon observed in $Sr_2Si_{0.95}Ti_{0.05}O_4$: Eu^{2^+} phosphor. For all investigated samples the persistent luminescence is dominated only by the one band with maximum at 570 nm, independently of the excitation wavelength. It leads to the conclusion, that traps responsible for long lasting luminescence of Sr_2SiO_4 : Eu^{2^+} observed

at room temperature, deactivate directly and only through Eu²⁺ ions occupying nine coordinated SII sites.

18:00 Poster P23

Current-voltage measurement system for investigation of nanocontacts

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The break-junction technique is widely used to create nanocontacts, giving a unique possibility to study the electron transport through a single or a few atoms or molecules connecting two macroscopic electrodes [1]. Metallic nanocontacts have attracted interest due to their potential applications in nanoelectronic circuits [2]. In the case of the gold nanocontacts, the quantized conductance behavior is observed even in air at room temperature. Also the breaking process of the nanocontacts is a current research topics [3]. The investigation of the mechanical breaking process of metal nanocontacts shows a very well ordered atomic narrowing for Ni, Fe and V nanocontacts [4]. The knowledge about the fundamental correlations between metastable atomic or molecular configurations is necessary for the designing of next generation electronic building blocks. Recently, a novel cross-correlation techniques was proposed to extract additional information from the conductance traces [5].

Another method for the characterization of metasable atomic configurations in nanocontacts is to determine a current-voltage characteristic. The time of I-V measurements must not go beyond the duration of the atomic configuration. We present a measurement system for capturing electrical signals from which to determine the current-voltage characteristic. Used in the system, a digital storage oscilloscope (DSO) allows a reduction of measurement time to microseconds. The proposed system allows users to select a value of electrical conductance of the breaking nanocontact for which the measurement is carried out, which is a big advantage over measurement systems proposed earlier. Users also can use for forming and breaking nanocontacts two types of actuators: piezoelectric and magnetostrictive. The first one is used for forming a nanocontact between the metal tip and the metal substrate, whereas the second one, for forming a nanocontact between the metal tip and the semiconductor substrate [6]. The short measurement time involves an impact of transition states occurring in the setup circuit, and consequently, a systematic error in the measurements. The right interpretation of the measurement data requires elimination of the systematic error through the application of a suitable correction procedure correction procedure, which the proposed system supports. [7].

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18:00 Poster P24

Obtained results of selective soldering using the developed fluxes.

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Most of components in medical applications, measurement and control and other highly reliable devices are mounted on printed circuit boards (PCB) with the use of standard techniques such as reflow soldering and wave, the leaded solder is used. Those products are within the exemption of directive RoHS [1]. It is so because of the reliability of lead-free solder joints was in doubt. Bulky components or temperature sensitive are mounted during additional steps such as automatic selective soldering with the use of dedicated fluxes for this technique. Hand soldering is often abandoned because of reliability is questionable and is dependent of operator [2]. This topic became more complex after 1-st of July 2011 when an amendment to RoHS directive was published known as RoHS2 [3]. In this directive medical equipment and measurement and control devices are not any more in the exemption, that's why have to be produced in lead-free technology.

The quality of solder joint [4] in lead-free selective soldering is very dependent on application of flux and its composition. As the time during soldering is short the enough heat has to be transferred to activate the surface, by chemical reaction of flux as well as make solder joint as such.

Practical aspects of the usage of the developed fluxes will be presented. The fluxes are designed to work with lead-free solders. Crucial aspects of obtaining reliable solder joint for demanding applications will be presented.

18:00 Poster P25

Electrodeposition of nanocrystalline silicon onto graphitebased materials from molten fluoride mixtures

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Crystalline silicon is one of the most perspective materials for terrestrial solar cells, and results of further investigations concerning its obtaining methods should be associated with its cost reduction.

Production of solar cell using LiF-KF-K₂SiF₆ fluoride melts is very attractive and perspective in terms of silicon obtaining with desired purity level.

Silicon coating deposition onto low-cost electrically conducting substrates such as steel, brass, nickel, tungsten, or graphite is very promising in terms of solar cells cost reduction. Unfortunately, for most of these substrates, silicides formation is typical at 1023 K. Therefore, graphite is the most perspective material due to its lowest reactivity.

The objective of the present study was to electrodeposit high purity silicon onto graphite substrate in the form of adherent coating and to study the deposits morphology dependence on the electrodeposition conditions.

Dense, good adherent silicon coating was deposited onto graphite base at potentials -0,75 \pm 0,05 V vs. Ag or Pt electrode from molten LiF-KF-K_2SiF_6 system at 1023 K. Silicon containing compound concentration was within 8-14 mol. % range. Cross-sections of sediments indicate nodular or dendritic growth up to several millimeters at the main part of adherent layer up to 1.0 mm thick. Polycrystalline silicon has columnar structure with grain size of about 250 nm. Impurities (Cu, Fe, and Ni) content did not exceed 0.005%. Specific resistance of deposit at room temperature was always higher than 1 $\Omega \cdot cm$.

The effect of electrodeposition parameters on the morphology of the silicon surface and the possibility to use polycrystalline silicon in solar cells were discussed.

18:00 Poster P27

Structure and properties of superhard cBN composites with a nanostructured binding

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The process for obtaining PCBN composites by the reaction sintering powders cBN with Al at pressures of 7.7 and 4.2 GPa with a preliminary dispersion of Al in the mixture at p = 2.5 GPa and T = 1300 K is designed in the ISM NASU. The problems of sintering nanopowders cBN are following: high cohesive activity, tendency to a phase transition cBN→hBN; difficulty infiltration due to the small size of the pores. cBN submicron powder advisable used in the mixture cBN-Al, in submicron cBNthe 15 % nanofraction located on the surface of larger particles. Nanosized particles before others interacted with Al. The reaction product AlN inherit the size and morphology of nanoparticles cBN. Instead of a continuous frame cBN, resulting in sintering micron, highly dispersed structure of phase cBN in the shell with AlN nanoscale grains (15-30 nm) is formed. The material has a high fracture toughness - 10.5 MPa·m¹¹² at a hardness 27 GPa.

Nanocomposites system cBN-Al-TiB $_2$ were obtained by the same principle, where a high modulus TiB $_2$ in the mixture acts as an activating, alloying and modifying additives. Here nanophases were the reaction products - solid solutions Ti $_x$ Al $_{I-x}$ B $_2$ (5-200 nm), Al $_x$ B $_y$ N $_z$ (\leq 100 nm) and on the boundary between cBN and Ti $_x$ Al $_{I-x}$ B $_2$ formed Ti $_6$ 8B $_1$ 0N $_2$ 2 (\leq 30 nm). Structure of composite with strain hardening frame cBN and high modulus nanostructured binding formed in the nanocomposite cBN-Al-TiB $_2$. The new composite has Knoop hardness 34-35 GPa and fracture toughness 6.5 MPa·m $^{1/2}$.

18:00 Poster P28

Examination of parameters of friction and wear for different mutual position of endoprosthesis elements

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Full title of the project: Examination of parameters of friction and wear for different mutual position of endoprosthesis elements

Name of the program: Development Project of the 10th Competition

Application year: 2009 Realization years: 2010-2013

The list of key parameters of the project: Metal Forming Institute, Poznan University of Medical Science

The advanced process of hip joint destruction in history of numerous diseases can be treated effectively with the use of hip replacement procedure. The procedure involves resection of the damaged elements of the joint (articular cartilage and subchondral bone) and replacement of the same with an artificial hip joint – endoprosthesis. The endoprosthesis is to assume functions of the damaged joint and release the patient from pain.

Presently, manufacturers of endoprostheses focus on production of implants for even younger and more active patients. In recent years we have observed growing interest in surface endoprostheses characterised by very little bone resection during implantation. The use of the "metal-on-metal" (MoM) couple with large diameters of the head and cup is characterized by an increased scope of mobility and reduces the probability of dislocation. The use of endoprostheses with MoM couple has a favorable influence upon the extension of the implant life as compared to a metal-on-polyethylene couple (MoP), ceramics-on-polyethylene (CoP). A significant problem still includes wear products generated resulting from the use of the endoprosthesis as well as metal ions released in the human body.

One of the relevant problems of contemporary hip replacement procedures involves the influence of the setting axis of endoprosthesis components on the friction resistances and their wear mechanisms. The works realized in the Metal Forming Institute aimed at determination of the angle values of mutual position of the "head-cup" couple, for which endoprosthesis functions are characterized by the most favorable tribological parameters. The determination of the optimum ranges of mutual position of endoprosthesis components will contribute to the improvement of computer navigation systems aiding implantation procedures of such types of endoprostheses.

The clinical part of the project included tests performed with the use of computed tomography (CT) of hip joints in a group of patients who have had a surface endoprosthesis implanted. CT tests aimed at a determination of average and extreme angle values of position of the endoprosthesis components in relation to the actual axis and rotation of the limb. The analysis of results allowed a determination of nine locations of the head-cup couple that were reconstructed during friction and wear tests. The implant stem angle of the head $(45^{\circ} + 90^{\circ})$ and cup inclination angle (45°) were assumed as fixed. However, the angle of antetorsion of the head $(-5^{\circ}, 10^{\circ} + 25^{\circ})$ and anteversion of the cup $(-10^{\circ}, +20^{\circ}, +30^{\circ})$ were subject to changes (fig. 1). In order to determine the concentration of Co^{2+} and Cr^{3+} ions, blood samples were collected from the tested patients.

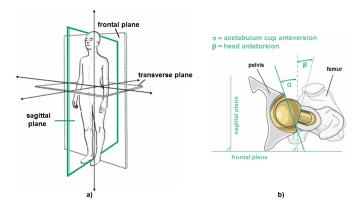


Fig. 1. The angle of anteversion of the cup and antetorsion of the head in relation to the system of anatomical planes: a) three basic anatomical planes [1], b) artificial hip joint (view from the top) [aofoundation.org]

Upon determination of the angle values of locations of endoprostheses components, friction and wear tests were performed with the use of a simulator of hip endoprostheses (Fig. 2), the structure of which was prepared in INOP. The structure was based upon the anatomical structure of the human hip joint. It enables a measurement of the friction resistances with a possible modification of geometry of mutual position of the head-cup couple. Moreover, the simulator was designed to enable sampling of wear products generated as a result of friction for the purposes of further analyses. Covering of the metal elements of the simulator with a fluoropolymer coating made enabled determining of the concentration of metal ions released into the lubricating fluid during the realization of the friction and wear tests.

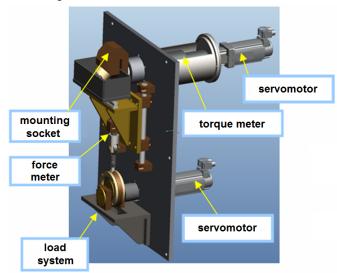


Fig. 2. A simulator of the hip joint SBT-01.1 [2]

The object of the friction and wear tests included nine sets of MoM type hip endoprostheses with Ø44 mm diameter of the cup and head. The endoprostheses were made of an alloy based on cobalt (Metasul, ISO 5832-12). Each of the endoprostheses worked for 1 000 000 cycles in the simulator, which corresponded to approx. one year of use in invivo conditions. The friction loop was loaded with a force of maximum instantaneous value of 1300 N. The range of bending/strengthening movements was 40° with the frequency of 1 Hz. The tests were conducted in the distilled ultraclean water environment. Each test was performed for different mutual position of the "head-cup" couple elements [3].

The friction coefficient value was calculated on the basis of measurements of the friction moment. All components of the tested endoprostheses were weighed and measured with respect to roughness before commencement and after completion of the friction and wear tests. The analyses of the concentration of $\mathrm{Co^{2+}}$ and $\mathrm{C^{r3+}}$ ions in the lubricating fluid were made with the use of the method of atomic absorption spectrometry.

The analysis of the obtained test results and definition of correlations between the friction coefficient, changes of roughness of friction surfaces, mass wear and concentration of metal ions in the lubricating fluid in relation to the geometry of mutual position of the head-cup couple contributed to the creation of a unique relative database. Apart from the possibilities of visualization of the data collected, the database was equipped with a tool, enabling forecasting of the values of friction resistances, wear and level of concentration of metal ions depending on the mutual position of the endoprosthesis components.

Further scientific and research works will include numerical simulations aimed at determining of the influence of the mutual position of the head-cup couple upon values of reduced stresses existing in the friction loop and at the boundary of the implant-bone couple.

The test results obtained so far confirmed the assumption that the angle of mutual position of hip endoprosthesis components has a significant influence upon the values of the friction resistances and, consequently, the intensity of the wear of endoprosthesis, which influences their life.

Depending on the change of position of the head in relation to the cup or the cup in relation to the head, an average value of friction coefficient (for results recorded every 100 000 cycles worked) changed from 0.139 to 0.231. The friction processes resulted in an increase of roughness of the friction surfaces and a decrease of mass of the endoprothesis elements cooperating with one another. The measurements showed that cups were characterized with a greater mass wear. The roughness parameter of friction surfaces upon completion of tribological tests was within the range of 0.51 μm to 0.85 μm for the heads and from 0.54 μm to 1.13 μm for the cups. The mass decrease fell within the range of 29.8 mg to 97.8 mg for the heads and from 179.1 mg to 316.6 mg [3]. The condition of friction surfaces upon the completion of the friction and wear tests is presented in Fig. 3.

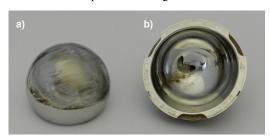


Fig. 3. An example of the condition of friction surfaces of the endoprosthesis upon completion of the friction and wear tests: a) head, b) acetabulum cup

The analysis of concentration of cobalt and chrome ions in the lubricating fluid showed that their concentrations were much too high than in the clinical samples. Depending on the mutual position of the head-cup couple elements, the concentration of Co^{2+} ions changed from 17.2 to 31.5 mg/dm³ and Cr^{3+} from 0.14 to 1.64 mg/dm³, whereas the clinical samples showed from 1.01 to 7.39 $\mu\text{g}/\text{dm}^3$ Co^{2+} ions and from 0.32 $\mu\text{g}/\text{dm}^3$ to 3.90 $\mu\text{g}/\text{dm}^3$ Cr^{3+} ions.

Figure 4 shows the results of the calculations of the friction coefficient, the Ra roughness measurements, mass wear and concentration of Co²⁺ and Cr³⁺ ions in the lubricating fluid for three randomly selected positions of the head in relation to the cup. The results show unambiguously that the change of the angle of the cup position (angle of anteversion:

-5°, +10°, +25°) in relation to the head (angle of antetorsion equaling 10°) has an influence upon the values of the friction resistances. Decreases or increases of the values of the angle of cup anteversion in relation to the initial position (20°) led to an increase of the value of the friction coefficient (Fig. 4a). Greater friction resistances led to the intensification of the wear processes, which was manifested in increases of the roughness (fig. 4b) and mass wear (fig. 4c) of the friction components. The greater intensity of the wear processes was also reflected in an increase of the concentration of metal ions in the lubricating fluid (fig. 4d).

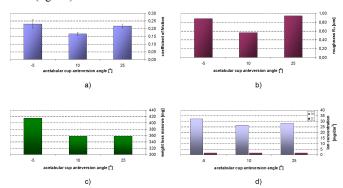


Fig. 4. Influence of the angle of cup anteversion upon: a) friction coefficient, b) roughness of friction surfaces, c) mass wear, d) concentration of ions in the lubricating fluid

Finding and defining of the correlations between the tested physical values depending on the mutual position of the head-cup couple elements constituted an inspiration for designing and delivery of a relative database. Next, a platform was developed as connected directly with the database. Apart from the functions involving graphic and user-friendly method of presentation of the collected clinical and laboratory test results, the platform was equipped with a special tool that enables forecasting the values of friction resistances, wear consequences as well as the levels of concentration of Co²⁺ and Cr³⁺ ions depending on the mutual position of the hip endoprosthesis elements. Owing to this unique tool, an orthopaedist will be able to make a correction in his suggested position of the "head-cup" couple elements at the very stage of preoperative procedures. It will be sufficient to introduce angles ranges of position and the tool will search for and suggest the optimum position (fig. 5).

A serious health threat for the population in Poland includes civilization diseases such as, inter alia, primary as well as secondary degenerative lesions, i.e. resulting from previous disorders of joint mechanics on the basis of childhood diseases or mobile organ injuries. An increase in the number of joint endoprostheses implanted is connected with progressive ageing of the population, as the percentage of degenerative lesions is the largest among people above 65 years of age. Apart from the ageing of the population, the main cause of the majority of bone and joint system diseases are hazards resulting from changing of the human environment and a sitting lifestyle. A rapid progress of knowledge makes it possible to get to know civilization diseases better, identify high risk groups, address intervention activities properly and detect diseases earlier, which improves treatment effectiveness.

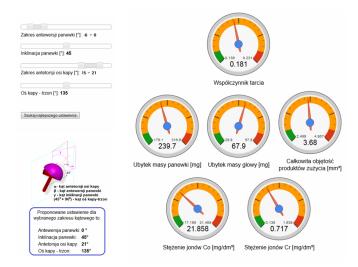


Fig. 5. A browser of optimum parameters of mutual position of the hip endoprosthesis elements— a screenshot

A significant element of the project includes biotribological tests and acquaintance with the mechanism of mutual influence of individual implant elements. The most comfortable method of obtaining information related to this issue is the correlation between clinical observations and an analysis of the issue from the point of view of biomechanical structure of the human organism. One of the tasks of the project entitled "Examination of parameters of friction and wear for different mutual position of endoprosthesis elements" is the structure of a physical model of the human hip joint implant elements. The analysis of suboptimum mutual position of the endoprosthesis elements is aimed at an improvement of the comfort of life of patients with a hip endoprosthesis implanted by increasing the reliability of the implants and, consequently, decreasing the risk of revision procedures.

The Innovative Economy programmed imposes an implementation of new materials and technologies, extension of specializations by new applications and products oriented at new markers in accordance with the propriety axis 1. PO IG:

- orienting scientific research at scientific fields and disciplines that contribute to a rapid civilization and economy development and building of economy based upon knowledge and transfer of the same to the industrial sphere;
- creation and increase of the scale of use of new solutions for the development of economy and improvement of competitiveness of enterprises as well as development of the Polish society;
- 3. increase of innovativeness.

Thus, the project correlates with the strategy of development of science in Poland until 2015 and the National Program of Research and Development Works in the 2nd research area: Health and 4th research area "Modern technologies for economy". A relevant prerequisite for undertaking of this Project is global development of new technologies of horizontal character that are significant for modern systems of social security and health protection.

The Project also shows a full correlation with the Regional Innovation Strategy entitled "INNOVATIVE WIELKOPOLSKA". The vision in the document is the province of Wielkopolska (Greater Poland):

- using its tradition and contemporary intellectual and economic potential for the creation of innovative grounds for development.
- considering innovations as the main factor of regional economic and social growth.
- 3. able to compete with other European regions.

Finally, it should be stressed that the realized Project directly fulfills one of the strategic aims of the Regional Strategy of Development i.e. the use of the research potential of the province of Wielkopolska for protection of health and increase of comfort of living for its society.

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18:00 Poster P29

Diamond-tungsten carbide nanocomposite

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Nanopowder mixtures of ASM5 0.1/0 diamond, and WO₃ were formed in the liquid. Air drying and thermal treatment of these mixtures in hydrogen were carried out. Composites were HP sintered from them [1, 2]. The structure and phase composition of the mixtures and sintered composites have been studied.

The WO_3 particle size is in the range of 150–250 nm, the particles are fragmented, the grain size varies from 10 to 150 nm. The size of the diamond particles does not exceed 100 nm. The relative position of the particles of diamond and WO_3 is homogeneous enough, however, a tendency to agglomerate particles is observed (fig. 1).

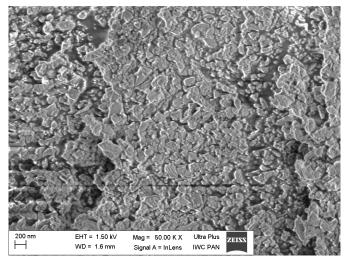
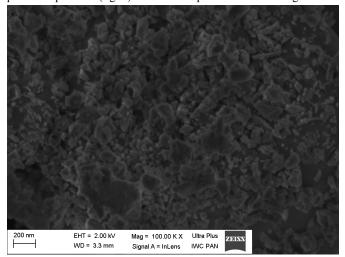


Fig. 1. The structure of the mixture of ASM5 0.1/0 statically synthesized diamond nanopowder and tungsten trioxide powder (46%) formedin a liquid after drying before annealing in hydrogen

During the mixture heat treatment in hydrogen at the temperature of 900 °C the complete recovery of WO₃ to the metal tungsten occurs.

In case of deviation from the heat treatment the W₃O oxide is present in the mixture, together with metallic tungsten and WO₂. The particle size of formed tungsten increased by 2–3 times compared to the WO₃ precursor particles (fig. 2). The formed particles are less fragmented.



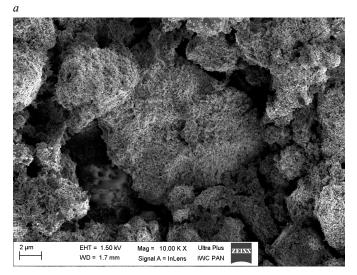
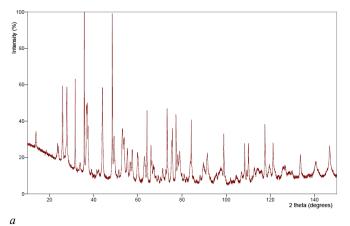


Fig. 2. Structure of the mixture of diamond nanopowder and tungsten trioxide after annealing in hydrogen obtained by SEM at different

b

magnifications (a, b)

The diamond and WC are the main phases in the composite sintered from the mixture which includes WO_2 , apart from diamond and tungsten. A small amount of W and WO_2 is also present. Amount of WO_2 has not changed compared to the initial mixture (fig. 3). WC formed as a result of the diamond and tungsten reaction is in the interspaces between the diamond particles and improves the connection between them (fig. 4). While the presence of WO_2 in the interspaces worsens the connection.



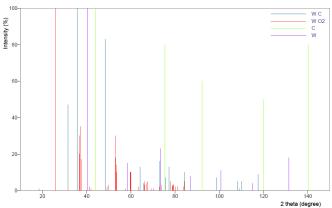


Fig. 3. Diffractogram (a) and diffraction pattern (b) of composite sintered from a mixture of diamond nanopowder and tungsten powder treatment in hydrogen

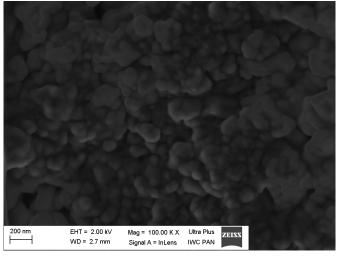


Fig. 4. The structure of the surface obtained by shearing the sintered composite sample

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18:00 Poster P30

Preparation and thermal stability of MAX-phases Ti-Al-(C,N)

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 Ti_3AlC_2 , Ti_2AlC , Ti_2AlN , $Ti_2Al(C_{1-x},N_x)_y$ are representative of nanolaminated ternary carbides called MAX phases of structural type 312 and 211, which combines the best properties of ceramics and metals. The aim of this work was to study resistance during heating in air by TG and DTG methods of MAX phases of systems Ti-Al-(C,N) structural type 312 and 211. Results of termal stability on the air showed increasing of layers of titanium carbide phase in the structure MAX increases its resistance to oxidation. The weight gain of the phase structure type 312 during heating up to 1000 ° was 3.5%, and the structural type 211 - 8.5-9.5%. Moreover, the substitution of carbon by nitrogen in the structure 211 causes a reduction in oxidation resistance.

18:00 Poster P31

High-pure nanostructured titanium obtained by cryogenic quasi-hydrostatic extrusion

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Titanium and its alloys are widely used in various fields of engineering and medicine. The requirements for their physical, mechanical and chemical properties are increasing, so we need for a new approaches of controlling of structure and characteristics of these materials. This applies, first of all to the commercially pure titanium and high-pure titanium, for which the possibility of increasing the strength characteristics to a large extent limited. At the same time, for some applications, in particular for manufacture of medical implants pure titanium is most preferable due to the high biocompatibility and its lack of toxic elements. Improving the strength of pure metals, including titanium, up to the strength level of heavily doped alloys is possible by different extreme effects on bulk material. These effects include the severe plastic deformation (SPD) and the low-temperature or cryogenic deformation (CD). Thus, the aim of this study was to investigate the role of the original grain size of iodide titanium obtained by SPD and subsequent annealing on the evolution of its structure and modify its properties after cryogenic deformation by quasi-hydrostatic extrusion.

Using SPD under the scheme of upsetting-extrusion-drawing was obtained the high-pure titanium with the grain size of about 160 nm. The subsequent annealing of titanium at the temperatures of 350-550°C allowed to create the microstructure with grain sizes from a few hundred nanometers up to 9 mkm. Samples with the different grain sizes were subjected of quasi-hydrostatic extrusion at liquid nitrogen temperature (77 K) and room temperature as described in, and then the microstructure and mechanical properties were investigated.

It is shown that quasi-hydrostatic extrusion leads to effective grain refinement, especially in the conduct of quasi-hydrostatic extrusion at nitrogen temperature. Thus, in titanium with the original grain size of 9 mkm the quasi-hydrostatic extrusion with degree of deformation of 45% leads to the grain refinement in 4 times under the deformation at room temperature and in 20 times in the case of deformation at 77 K. In the samples of submicrocrystalline titanium after quasi-hydrostatic extrusion at room temperature there was some decreasing of the yield strength and microhardness with an appreciable increasing of tensile strength. For samples that were deformed at cryogenic temperature all these parameters increased. In addition, the quasi-hydrostatic extrusion of submicrocrystalline titanium promotes to significant increasing plasticity of the material, in particular, the elongation to failure increased from 6.7% to 12.3%. Thus, the combination of methods of SPD with the cryogenic quasi-hydrostatic extrusion allowed to create a high-pure nanocrystalline titanium with the grain size of 75 nm, high strength ($\sigma_h = 930$ MPa) and plasticity ($\delta \approx 12\%$). This material may be of interest for various applications, in particular for the manufacture of medical implants.

18:00 Poster P32

Nano-QSAR modelling - efficient way of predicting the toxicity of metal oxide nanoparticles to human keratinocyte cell line

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Together with an increasing role of nanotechnology in our every-daylife, we can also expect increasing emissions and, consequently, increasing levels of nanoparticles (NPs) in the environmental compartments. However, there is an increasing number of contributions that report toxicity and/or ecotoxicity of selected NPs and highlight the potential risk related to the development of nanoengineering. Therefore, novel, fast and inexpensive procedures for identifying potentially hazardous NPs without necessity of extensive empirical and animal testing are needed. Toxicity of NPs can be predicted in alternative way by applying Quantitative Structure-Activity Relationships methods (QSAR). QSAR methods are based on the assumption that the variance in a given physico-chemical or biological (e.g. toxicity) property in a set of compounds (so-called endpoint) is determined by the variance in their molecular structures, encoded by so-called descriptors. Consequently, when the values of the endpoint are available only for a part of the group, it is possible to interpolate lacking data from an appropriate mathematical model.

The main purpose of the study was to illustrate the thesis above by developing a Nano-QSAR model that describes the relationship between the structure and toxicity of 22 nano-metal oxides to human keratinocyte (HaCaT) cell line. We have investigated the changes in cell viability and the process of generating reactive oxygen species by employing a human keratinocyte cell line as a model for dermal exposure. The experimental results expressed in term of LD₅₀ values indicated that ZnO was the most toxic one in the set of the NPs tested. The LD₅₀ value determined for ZnO was 27 μg/ml, while the values of LD₅₀ for the majority of the other NPs (In₂O₃, La₂O₃, SnO₂) were higher than 250 µg/ml. Additionally, in order to find the best structural parameters, reflecting the essential properties of the studied nanomaterials (shape, porosity, surface area, the electronic states resulting from quantum effects, etc.) we proposed a set of (i) image descriptors (based on images taken from Transmission Electron Microscopy) and (ii) quantum-mechanical descriptors (based on quantum-chemical

calculations). The combined experimental-theoretical study allowed us to develop a Nano-QSAR model that reliably predicts the toxicity of all considered compounds. Such a model could be applied not only to NPs investigated in the current work, but also to unexplored related species.

18:00 Poster P33

Application of polymeric material as a carton surface barrier for water-vapour, water and fat

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The latest packages' generations are produced more often with biodegradable biopolymers (4). This type of packages are used to improve functionality and harmony to EU Regulations (2,3). Available on the Polish market and mostly applied cellulose packages, coated with aluminium foil are the only proposition for high fat content products producers'. Costs of this kind of packages increase by 50% (1).

The main objective of the project is to develop a technology for the production of individual carton by using polymeric materials as biodegradable barrier resistance to water-vapour, water and fat, based on an innovative production process with the guarantee of microbiological purity, neutral in terms of the migration of organic chamicals.

As a result of the research is to develop a biodegradable polymer composition, which is an alternetive to the cartons coated polyethylene for application to greasy and moist foods.

Microscopic photos of carton's unit coated with biopolymeric layer are present below.

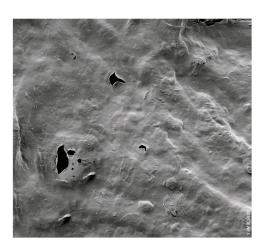


Fig. 1. Monolayer of biopolymer - composition 3W

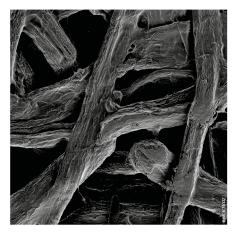


Fig. 2. Monolayer of biopolymet - composition 1B

Monlayer were not sufficient barrier to fats, bilayer substantially reduced barrier properties of carton against water-vapour and reduced absorption of soil. Strengh has also been improved for cardboard bending with unchanging resistance to breaking.

This work "Production unit carton with the use of polymeric materials as biodegradable barrier layers with increased resistence to water-vapour, water and fat, based on an innovate production process" is cofinansed by the European Regional Development Found (POIG.01.04.00-14-084/09-00).

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18:00 Poster P34

The contents of selected heavy metals in the food package's ink and packages cellulose materials

Zdzisław Polański, Agnieszka Ozimek, Karolina Jakubowska, Jacek Chapiński

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Inks for printing on cellulose packaging intended to come into contact with food must meet many requirements. In accordance with the EU regulation food packaging must be manufactured from materials with appropriately chemical composition, meeting safety requirements. Inapplicable materials, including paints used for graphic printing on packaging, can be a source of contamination of food compounds harmful to health. Inappropriate materials may cause the chemical composite on and organoleptic characteristic of the packaged products (1,4).

One of the most popular components of printing ink is black carbon. Black carbon is the most common plant, harvested by the combustion of different substances depending on the manufacturing technology,

destination and manufacturer (3). Soot forming dye can be of different sizes, some of them may be in nano size "natural nano-molecule".

The aim of this work was to determine the content of selected heavy metals (Pb, Cd and Cr) in food packaging and packaging inks, acquired on the local market.

Testing materials were cellulose packaging (primary and waste paper package) and inks (SunChemical) intended for the food packaging production (used to manufacture by the Arso-Polański sp. z o.o.).

After mineralization all the trials were analysed. Determinations were performed using atomic absorption spectrometer with electro-termic atomization Savant AA (double beam spectrometer with deuterium background correction) produced by GBC.

The average lead content in cellulose packaging was - 0,75 ppm, chromium – 1,5 ppm and cadmium <LOQ. Similarly average content in waste paper package was for Pb – 9 ppm, for Cd – 5,02 ppm and Cd < LOQ.

Minor content of individual metals in packaging are consistent with the results obtained for the inks. The largest average heavy metal content determined for cadmium 56,54 ppm in the paint red (Magneta), lowest also for cadmium <LOQ in black paint (Arso -Polański).

The result showed that the quality of the inks used in the Arso-Polański Sp. z o.o. enables the production of high-quality packing, in accordance with the requirements for food packing contained in Directive 94/62/EC. Directive indicate total amount of lead, cadmium, mercury and Chromium (VI) in the package or item should not exceed 100 ppm. In this case there were no exceeded of the limits according to Directive standard. Due to the fluctuations associated with the production of pigments (3) it is necessary to continue to monitor the content of the above metals, in order to produce high-quality packing.

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Wednesday, 18 September

REGISTRATION

Wednesday morning, 18 September, 8:30

Nano-Bio, Nano-Med.

Wednesday morning, 18 September, 9:00 *Chair: Andrei Sommer*

9:00

Oral

Creating a Polish Nano-Bio-Technology and Nano-Medicine Platform?

Witold Łojkowski

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I will present a proposition of creating a Poilsh Bio-Nano-Technology, Nano-Medicine and converging technologies platform.

9:10

Invited oral

Porous Calcium Phosphate Ceramics for Bone Tissue Replacement.

Janis Locs, Vita Zalite, Liga Berzina-Cimdina, Marina Sokolova

Rudolfs Cimdins Riga Biomaterials Innovation and Development Centre of Riga Technical University (RCRBIDC), Pulka street 3/3, Riga 1007, Latvia

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Calcium phosphate (CaP) bioceramics are popular implant material for bone repair, substitution or augmentation in orthopaedic and maxillofacial surgery. CaP materials exhibit biocompatibility with hard tissue and promote bone regeneration processe by release of calcium and phosphate ions. CaP ceramics are widely used in several clinical applications in different forms. There are various techniques to produce porous calcium phosphate ceramics. Introducing additional functions to the ceramic materials provides the possibility in applications as drug or cell delivery systems. Each of the applications requires specific microstruclutal and compositional architecture of all kind of elements. The newly developed technology for the preparation of porous calcium phosphate ceramics will be presented. The viscous slurry foaming using ammonium bicarbonate as foaming agent is an promising technology allowing to produce large scale porous implant materials.

9:25 Oral

Evaluation of bioceramic bone substitutes - hydroxyapatite (HAP), tricalcium phosphate (TCP) and biphasic ceramic (HAP/TCP) in vivo

<u>Ilze Salma</u>¹, Mara Pilmane¹, Girts Salms¹, Janis Locs², Liga Berzina-Cimdina²

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In clinical practice different bone substitute biomaterials are used for maxillary sinus floor augmentation. Due to biocompatibility with living body and bioactivityhydroxyapatite (HAP), tricalcium phosphate (TCP) and biphasic ceramic (HAP/TCP) are frequently used and are the most promising due to the particular biodegradation. Datain the literature of tissueresponses to the bioceramic materials are different and sometimes controversial. The aim of our study was to evaluate bone and soft tissue response in animal experiments.

CaP powders used for bone substitutes preparation was synthesized by wet precipitation reaction between calcium hydroxide and phosphoric acid. The green bodies – granules in size range from 0.5 to 1 mm were formed and sintered to obtain HAP, TCP or HAP/TCP ceramics.

Intraosal implantation of bioceramic granules was performed in rabbit jaw. Bone and soft tissue with biomaterial was obtained after three months. Tissues were proceeded for the detection of growth factors – bone morphogenic protein (BMP2/4) and transforming growth factor (TGF β), proinflammatory cytokines and using TUNEL method – apoptosis.

Results demonstrated *most* pronounced*proinflammatory cytokines expression after implantation of TCP bioceramic. Results after biphasic ceramic implantation showed less cell apoptosis.*

Conclusions. Our results demonstrated higher biocompatibility for biphasic bioceramic HAP/TCP (60/40) compared with HAP and TCP.

9:40

Polish Research Projects

MSS synthesis of highly biocompatible nano-HAP and coating of 3D power scaffolds

<u>Witold Łojkowski</u>, Aleksandra Kędzierska¹, Tadeusz Chudoba¹, Jacek Wojnarowicz¹, Dariusz Smolen, Dariusz Smoleń², Sylwia Kuśnieruk¹

1. Institute of High Pressure Physics, Warsaw 01-142, Poland 2. NanoVelos Sp. z o.o. (NV), Trakt Lubelski, Warszawa 04-870, Poland

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9:55

Oral

Creation organic nanofibres with the phosphazene additives

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Most synthetic polymers are created from organic chain but inorganic polymeric materials with similar properties can be also utilized. Phosphazenes are inorganic materials with a backbone of regularly alternating phosphorus and nitrogen atoms with two organic or organometallic substituents linked to each phosphorus atom. Phosphazenes occurred as monomers as well as polymers. Polyphosphazene derivatives have many advantages, such as flame retardancy, which could be introduced to mixtures of phosphazenes with commercial polymers, too

Our research focuses on synthesizing cyclic phosphazene derivatives with required properties via two different methods – nucleophilic substitution or condensation methods, with the aim to prepare additives to commercial polymers. Some mixtures are able to create nanofibres by electrospinning technology. These fibres have potential applications, e.g. as a flame retardant cover material, etc.

10:10

Oral

Quantum semiconductor nanostructures for applications in biology and medicine -development and commercialisation of new generation devices for molecular diagnostics on the basis of new Polish semiconductor devices

Marek Godlewski, Leszek Sirko

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The project of the Operational Programme - Innovative Economy POIG.01.01.02-00-008/08 aims at development ofmodern GaN, ZnO and related materials nano- and semiconductor structures for diagnostic devices for applications in biology, medicine and environmental protection, materials technology for sensors and molecular diagnostic applications.

The project is realized by the seven scientific institutions:

- 1. Institute of Physics of the PolishAcademy of Sciences coordinator
- 2. Institute of High Pressure Physics of the PolishAcademy of Sciences
- 3. Institute of Physical Chemistry of the Polish Academy of Sciences
- 4. Wrocław University of Technology, Faculty of Microsystems Electronics and Photonics
- 5. Institute of Electron Technology
- Nencki Institute of Experimental Biology of the PolishAcademy of Sciences
- 7. University of Warsaw, Interdisciplinary Centre for Mathematical and Computational Modelling

In the presentation we will discuss important technological and scientific achievements of the project. Some of them are outlined below.

The Institute of Physics PAS works on new generations of nanosensors based on thin films of two wide band gap semiconductors – ZnO and GaN, on their heterojunctions and quantum well structures. We also investigate biosensors based on nanoparticles for applications as fluorescence labeling. Examples of such applications will be presented.

The main achievement in the Institute of Physical Chemistry PAS was the development and fabrication of a series of novel chemosensing polymer films for recognition and determination of biologically important analytes, such as melamine, nicotine, or ATP. The devised polymer films based on derivatives of bis(2,2'-bithienyl)methane and zinc porphyrin were successfully deposited by electrochemical polymerization on either quartz crystal resonators, or metal electrodes to result in either piezomicrogravimetric or impedimetric chemosensors with high sensitivity, selectivity, and detectability toward studied analytes. One should also stress successful development of a wide range of film electrodes modified with nanomaterials and/or enzymes with prospective application in sensors and energy conversion devices.

The Institute of High Pressure Physics PAS developed the method of crystallization of large size GaN substrates, grown by combination of HVPE and High Pressure methods, with the size up to 2 inch and very low dislocation density, suitable to application as plasmonic substrates for blue and green lasers. IHPP PAS constructed, as the second laboratory in the world, the violet superluminescent diodes (SLED) of high power of 200 mW. Multi-laser lines (3-30), emitting at 400-440 nm of power higher than 4 W continuous work (CW) have been obtained.

10:25

Polish Research Projects

Novel optical oxygen sensor for life and health protection made of nano-zirconia

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Acronym: OxyNanoSen

<u>Title:</u> Novel optical oxygen sensor for life and health protection made of nano-zirconia

Project no.: ERA-NET 2010/0204/2DP/2.1.1.2.0/10/APIA/VIAA/010

Duration: 29.04.2009 -28.04.2012

Consortium:

1. Institute of High Pressure Physics, Polish Academy of Sciences, Poland

- 2. Institute of Solid State Physics, University of Latvia, Latvia
- 3. Bar-Ilan University, Israel
- 4. University of Modena and Reggio Emilia, Italy

Objective and methods:

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We discovered that photoluminescence efficiency of ZrO₂:Eu nanopowder was strongly influenced by partial pressure of oxygen in the surrounding gas atmosphere. Therefore, intensive work was devoted to the optimization of material properties with respect to sensor application. The far reaching objective of the project was commercialisation of an oxygen partial pressure sensor superior to the presently available ones, based on our discovery: the dependence of luminescence of nanocrystalline zirconia on oxygen partial pressure. The sensor is protected by a pending PCT application that obtained a preliminary approval of the European Patent Office.

Specific project objectives:

- Check which of the existing models better explains the underlying physical phenomena of luminescence dependence on oxygen pressure,
- 2. Extend the operation temperature range of the sensor,
- 3. Test life time in various gases,
- 4. Develop calibration procedures,
- Test particular technical solutions: how the material is optimally prepared: as thin film, porous ceramics, dense ceramics, method of illumination and light collection.

Results:

Synthesize and preparation techniques of ZrO₂ nanopowders doped with rare earth elements were developed in respect to the application as an optical oxygen sensor. Material chemical composition and heat treatment procedures were evaluated: ZrO₂:8% mol Eu nanopowder annealed in air was identified as material presenting the greatest photoluminescence sensitivity to oxygen partial pressure. Operating temperatures range of tested nano-sensor was established: ZrO₂:8% mol Eu nanopowder preserved oxygen sensitivity from sub-zero up to elevated temperatures. Prototype of oxygen sensor that surpasses the parameters of existing apparatus was constructed. Finally, a model of the processes responsible for the sensor phenomena was proposed. In conclusion, results of OxyNanoSen project have great practical importance, because they can be utilized in universal oxygen sensor device applicable in various temperature conditions.

COFFEE and POSTERS

Wednesday morning, 18 September, 10:40

Polish Research Projects: Nano-Bio technology

Wednesday morning, 18 September, 11:00

Chair: Radka Bacovska

11:00 Invited oral

Petri Dish Generation III

<u>Andrei P. Sommer</u>¹, Dan Zhu^{1,2}, Friedrich Gagsteiger², Hans-Jörg Fecht¹

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The tissue culture dish is the most basic tool in life science laboratories. The glass version, hence in vitro, and polystyrene version (disposable), were introduced by Petri (1887) [1] and Fisk (1956) [2], respectively. Polystyrene Petri dishes stand at the beginning of biomaterial tests (cell culture, animal model, clinical trial), where they are used to interrogate cells, but also at the beginning of human life, i.e., in vitro fertilization (IVF). With the exception of geometrical modifications and hydrophilic conversion, the polystyrene Petri dish remained virtually unchanged. Its biodurability was never challenged. By modelling the cooperative behaviour [3] of anchorage-dependent cells used as living probes, we proposed that the surface of polystyrene Petri dishes becomes soft in aqueous media and that the effect facilitates the establishment of a nanoscopic layer of reactive oxygen species (ROS) [4]. For clarification the surface hardness of polystyrene Petri dishes was probed by nanoindentation, in air and in water. The measurements in water were facilitated by a sample holder designed for extended measurements in aqueous media [5]. It was found that water softens the surface of polystyrene Petri dishes to a depth of ca. 100 nm. The surface softening is a precondition for the establishment of a stable nanoscopic layer of ROS, as can be deduced from physicochemical considerations. Previous models focusing on possible mechanisms of formation of primordial biopolymers indicated that natural hydrogenated diamonds may have provided the optimal condition to foster the chemical reactions believed to have given rise to life on Earth [6]. This picture was complemented by the experimental finding that cell performance was better on moderately hydrogenated nanocrystalline diamond surfaces than on polystyrene (Petri dish) [4]. There is observational evidence that the hydrogenation is stable in aqueous environments [7,8]. Because 99% of IVF procedures are conducted in polystyrene Petri dishes we eventually decided to test a new type of Petri dishes for IVF: Quartz glass Petri dishes coated with a transparent film of moderately hydrogenated nanocrystalline diamond (Figure 1). Their applicability was tested in a sperm cell model using polystyrene Petri dishes as controls [9]. The nanocrystalline diamond coated Petri dishes (Petri dish generation III) preserved the performance of the sperm cells 20% better than polystyrene. Our result could help improve the quality/reliability of in vitro work.



Figure 1. Petri dish: nanocrystalline diamond coated quartz glass (left) - polystyrene (right).

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11:20

Polish Research Projects

Biomimetic coatings for implants

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Ageing societies demands "spare parts" but the number donors is always too small. Technology of artificial organs for humans is developing very quickly but the still unsolved problem is an interface between artificial lifeless structure and living organism. Especially implants having a constant contact with human blood, like heart or vascular prosthesis, are a source of constant problems. Contact activation of blood platelet causes clot formation at the implant surface or somewhere in our circulatory system. Clot formed at the implant surface can block the blood flow and function of the prosthesis; clot formed in the circulatory system can block small vessels in lungs or brain, what can be fatal for a patient. We propose to solve this problem by the biomimetic approach, formation of coatings that mimics natural inner surface of human vascular system at three different levels of mimicry. To check this approach three types of coatings for medical polyurethanes have been synthesized. First is a poly vinyl pyrrolidone based hydrogel coating, which mimics extracellular matrix and exhibit a very low surface energy. This prevents small protein adsorption and activation of complement immunological system. Second type of the coatings is mimicking outer layer of cellular membrane. Coating is made of dipalmitoyl phosphatidylcholine, which molecules are organized like in the cellular membrane. Third type of the coatings is a macromolecular nanostructure constructed step by step at the polymer surface. This structure is able to present certain sequence of aminoacids. The aim of this structure is to attract and anchor a certain type of human cells – endothelial cells, which monolayer naturally covers all tissues which have contact with blood. Once anchored cells start to divide and form a monolayer of self healing properties. Endothelium is able to actively prevent platelet activation by presenting proper surface ligands and releasing substances able to prevent cascade activation and even to deactivate platelet and dissolve small clot pieces. First two types of coatings were tested with human blood in static and dynamic conditions, and the third one was tested with human umbilical cord endothelial cells. All the coatings are very promising candidates for various types for blood contacting medical devices.

11:40 Oral

Nanofibrous mats as a protective materials in neurosurgery

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Electrospinning of polymer nanofibers is a versatile method of producing ultrafine fibers and has attracted large attention in the field of tissue engineering, wound healing and drug delivery systems. High surface area to volume ratio and similarity to Extracellular Matrix makes it potential for the use as a 3D scaffold as well as a drug delivery system. The aim of our work was to prepare active wound dressings for the prevention of excessive cicatrization after brain injury or spinal cord surgery. Traumatic Brain Injury (TBI) and its consequences are a serious public health problem. TBI can lead to pathological changes in the brain and glial scar formation which is an effective barrier preventing regeneration of damaged tissue. Produced in our laboratory electrospun mats composed of biodegradable polymer with medical approvals deliver drugs like: antioxidants – α -tocopherol and important in terms of regeneration of brain tissue NGF (Neural Growth Factor). Controlled release of these drugs can provide the supply of the appropriate medicine dose over a period of 8 to 14 days. It is found that dressing of surgically induced injury with nanofibrous mat did not induce inflammatory response but can promote correct healing process of the tissue. Construction of such system is a tedious experimental task. Hence, we take advantage of numerical simulation to construct a 3D finite element model which can help to optimize composition and geometrical structure of the nanofiber mat.

11:55

Polish Research Projects

Novel antimicrobial and biocidal filtration media

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A human being weighting 70 kg needs to drink 2.1 litres of water per day to sustain life. Yet 1% of the water available on earth is drinkable. This means that one of the major challenges of the 21st century will be to overcame the severe problem of guaranteeing the safe drinking water for rapidly increasing world population.

Our research is focused on development of novel water prefiltration media functionalised towards antimicrobial and biocidal properties. As a final goal, commercial polypropylene (PP) candle filters will be modified using silver (Ag) or zinc oxide (ZnO) nanoparticles or composite materials of such nanoparticles deposited on multiwall carbon nanotubes (MWCNTs). Our current research achievements include successful functionalization of non-woven polypropylene (NWPP) fabrics by mentioned above nanomaterials. Preliminary tests of antimicrobial and biocidal properties of synthesized materials show promising results, especially in the case of Ag or Ag/MWCNTs modified fabrics.

Scope of research

Commercially available MWCNTs were subjected to thorough cleaning and hydrophilization using strong acidic/oxidising solution treatment. Such prepared MWCNTs were dispersed in isopropyl alcohol under ultrasonication and/or by high pressure stream (Microfluidics).

Commercial PP fabrics made via MeltBlown technique with average fibre diameter of few micrometers were chosen as a base filtrate material. Surface of the fibres were directly modified by *in situ* formation of ZnO or Ag nanoparticles using sol-gel or reduction method, respectively.

MWCNTs deposition on PP fibres were realized by flushing of the fabrics with low concentration nanotubes suspensions in isopropyl alcohol. Such MWCNTs decorated fabrics were further subjected to nanoparticles formation process.

Due to the poor coverage ratio especially in the case of ZnO and Ag nanoparticles preliminary PP fibre surface activation process via oxygen plasma treatment was developed.

Antimicrobial and biocidal properties of modified fabrics were tested against *Escherchia coli*, *Pseudomonas fluorescens*, *Staphylococcus aureus*, *Salmonella typhimurium* and *Enterococcus faecalis*.

Results

- 1. Plasma pretreated and Ag/MWCNTs modified PP fabrics showed the best biocidal properties. Biocidal activity of this material was observed against all types of bacteria. The strongest effect was observed for Gram-positive *E. coli* and *S. typhimurium* (living cell reduction by factor of 10⁶ and 10⁵ respectively).
- Plasma pretreated and Ag modified material was found to be less effective (reduction factor 10⁶ and 10⁴). This material was inactive against *P. fluorescens*.
- 3. Materials modified without plasma pretreatment were found to be much less effective.

Research has been funded by NCRD within the MNT-ERA.NET programe no. ERA-NET/MNT/NFSM/1/2011.

12:10

Polish Research Projects

BioElectricSurface Project

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Acronym: BioElectricSurface

Title: Bio Electric Surface: Electrically Modified Biomaterials' sur-

faces: From Atoms to Applications

Project no: 212533

Duration: 2008-10-01 - 2011-09-30

Consortium:

- 1.University of Limerick
- 2. Politechnika Wrocławska
- 3. TU Dresden
- 4. Danish Technological Institute
- 5. Univerzita Komenskeho v Bratislavie
- 6. Universitatea Politehnica Bucuresti
- 7. Ben-Gurion University of the Negev
- 8. Peter Brehm
- 9. Cook Medical

- 10. Akademia Medyczna we Wroclawiu
- 11. Balton Sp. Z.o.o.
- 12. National University of Ireland in Galway

Objective and methods:

The BioElectricSurface project (2008-2011) was a FP7 small/medium collaborative project that used nanotechnology and novel instrumentation to generate new scientific understanding of biological interactions at the nonbiological surfaces. In the project there were 12 collaborating Partners from seven countries: Ireland (4 partners), Poland (3 partners), Germany (1 partner), Denmark (1 partner), Slovakia (1 partner), Romania (1 partner), Israel (1 partner). The project targeted four medical applications and was successful in demonstrating prototype devices as follows:

- a) MRSA-resistant textiles,
- b) Plaque resistant cardiovascular stents,
- c) Stone free urinary stents and
- d) Faster healing bone implants.

Given the complexity and challenges associated with medical devices and their development, it is remarkable that the BioElectricSurface developed high quality scientific knowledge and led to prototypes within the 3 years duration. One of these prototypes (textiles) is currently being licensed. The other two prototype devices (stents) are under further technological development by the respective industrial partners.

The project is a prime example of the use of nanotechnology in the field of medical devices especially in generating ground-breaking knowledge in challenging areas e.g. bio/non bio interface. The project successfully demonstrated how nanotechnology could enable new knowledge critically needed for breakthrough medical device technology. The project also demystified the field of surface charge in biomedical devices by employing innovative scientific approaches.

Thanks to the close collaboration of University of Limerick, Wroclaw University of Technology and Wroclaw Medical University durable, washable, photosterilisable MRSA resistant textiles were developed. It was demonstrated that by incorporation of specially designed nanoparticles into the textiles, it will be possible to obtain antibacterial clothes and hospital garments. Wroclaw University of Technology was a leader of the workpackage devoted to the antibacterial textiles development.

In addition to this, Wroclaw University of Technology has now become engaged into a Marie Curie IndustryAcademia Partnership with former BioElectricSurface project partners (National University of Ireland, Galway, Balton sp. z. o.o. and Wroclaw Medical University).

The project resulted in one edited book, 17 book chapters, 6 journal articles and 22 conference presentations within the duration of the project.

Polish partners: Wroclaw University of Technology and Wroclaw Medical University were key partners and leaders in 2 workpackages and successfully developed antibacterial textiles and together with Balton sp. z.o.o. new type of cardiovascular stents.

Results:

A. Scientific Achievements:

There has been the following scientific and technology breakthroughs from this project:

- 1. MRSA resistant textiles;
- 2. Plaque resistant cardiovascular stents;
- 3. Stone resistant Urinary stents;
- 4. Confirmation of piezoelectricity of synthetic bone mineral;
- 5. Creation of electrostatic domains in biomaterials without topographical change and
- 6. Prototype compact s-SNOM for biological applications

Prototype devices have been validated by industrial and clinical end users for Achievements 1-3. Achievement 1 is now very close to the commercial development due to the technology transfer by licensing. This achievement also generated huge commercial interest due to the elimination of any binder to incorporate nanoparticles into textiles. Overall this technology has been considered as a global breakthrough both scientifically, technically and commercially. Achievements 2 and 3 have been considered as technical breakthroughs by respective industrial partners who are now developing the technologies further. Achievements 4 and 5 highlighted key scientific breakthroughs and have been published in prestigious journals. The prototype s-SNOM is first of its kind and represents a key technological breakthrough for its substantial reduction of noise as well as a major scientific breakthrough due to its ability of extracting interfacial information.

Together, the achievements of this project addressed health related societal challenges, which are clearly shared by Member States of the European Union and are, in many cases, global challenges.

B. Impact Achievement:

The achievements of BioElectricSurface project are directly related to medical devices used in therapeutic applications in cardiovascular, orthopaedic and urinary applications. MRSA resistant textiles impacts hospital acquired infections such as the prevalence of MRSA superbugs which prolongs hospital stay for patients who received surgical treatments in relation to the above three and other diseases.

The MRSA bug is one of the major causes of hospital acquired infections. In June 2007, the European Centre for Disease Prevention (ECDC) has identified anti-biotic resistant micro-organisms as the most important infectious disease threat in Europe. One in 10 patients entering a European hospital can expect to catch an infection caused by drug-resistant microbes. Every year, around 3 million people in the EU catch a healthcare-associated infection, which causes approximately 50,000 deaths. Better cleanliness and hygienic practices in hospitals can reduce such occurrence significantly. Non-surgical drapes, uniforms, bed linens, pillow covers thus form an integral part of this cleanliness and hygiene.

The breakthrough technology, MRSA resistant textiles, will significantly improve cleanliness in the hospitals and help to reduce the occurrence of hospital-acquired infections.

It is estimated that at least 10% of the population in the industrialised world is afflicted by urinary tract stone disease. The annual incidence of stones amounts to around 2000 per million inhabitants in Europe and North America. For a group of first time stone-formers, the expected risk of recurrent stone formation during a 10-year period was estimated to be 30% and in those who had formed at least two stones at the start of follow-up the corresponding figure was as high as 70%. Urinary stents deployed in these stone formers suffer from the highest level of stone encrustation that increases pain and requires further surgery. The achievement in inhibiting the stone formation will increase the patency of these urological devices by a factor of two and reduce pain and the frequency of replacing such stents with significant cost saving.

Cardiovascular disease is the cause of half of all deaths in Europe. The main form of this disease is coronary artery disease, which occurs when the coronary arteries becomes hardened and narrowed, due to the build-up of plaques on the inner walls or lining of the arteries (atherosclerosis). This causes a reduced amount of blood flow. About half of all deaths from cardiovascular diseases derive from coronary artery disease. This is the most common cause of death in Europe, accounting for two million deaths each year. Over 22% women and 21% men in Europe die from this disease. The technology developed within the project reduces the plaque growth without the use of any drug and will reduce in-stent plaque-growth.

The above achievements thus represents significant beyond the state of the art solutions to the

problems associated with European health care and will contribute to the improvement of our quality of life, longevity and productivity. In addition to this, the commercial significance of the achievements is very high. The US and

European market size for medical textiles was estimated to be over \$7 billion and current sales only meet one third of the market potential. Our anti-MRSA textile technology will be used to produce practical, economical and effective products for this huge potential market. World Health Statistics 2007 show that there are 7.94 million nurses registered in Europe and USA. Assuming the average cost of a standard reusable uniform to be around \$40, the team estimates a total annual value of \$634 million for this segment alone. The medical device industry contributes to over 1% of the total EU-25 manufacturing value added and to over 1% of the total EU-25 manufacturing employment. This sector has sustained the wave of economic down turn and has a potential to grow at a rate of over 15% per annum. Two of the industrial partners of the BioElectricSurface consortium, COOK Medical, Ireland and Balton Ltd., Poland are important players in the in-dwelling medical device market. The achievements from this project will bolster their innovativeness, enhance competitiveness and sustain and promote employment.

Together, on economic terms, the achievements of the BioElectricSurface project directly impact a market that is over €5 b in size. The MRSA-resistant textiles is a product close to the market. A license deal has been negotiated to capture a part of the applications. A spin out company tis envisaged towards the end of 2013 that will commercialise the technology in the remaining fields of applications. This is a significant achievement for a small and medium scale project.

The BioElectricSurface project has received wide media attention and its key results have been disseminated to reach the wider public as well as the immediate scientific audience. Partners contributed to a book published by the Royal Society Chemistry (RSC). This book has been awarded the Book of the Month prize in August 2012 by the RSC. MRSA resistant textiles in particular received huge public attention in Irish and Polish television networks and satellite channels.

C. Achievements on EU Approach:

MRSA resistant textiles, cardiovascular coatings and urinary stents have been tested under International standards such as the IATCC standards for antimicrobial textiles, ASTM standards for cardiovascular and urinary stents. These tests have been performed in collaboration with the relevant end-users and industry partners. Research samples from BioElectricSurface e.g. nanocrystalline hydroxyapatite have been independently tested by a number of peers in Europe (Aveiro, Portugal and Linz, Austria) and Asia (Hong Kong, Peoples Republic of China). So the research results and solutions are applicable across Europe and beyond. Besides, a number of project achievements have been led by the New Member States of the European Union, who at the time of the project commencement were not very active in biomedical device research. The project demonstrated their ability to contribute to and even lead in ground breaking research related to medical devices.

12:25

Polish Research Projects

Nanomaterials for drug delivery and imaging

Stefan Jurga

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The new nanomaterials used for targeted drug delivery, bioimaging and MRI imaging are very challenging research areas for application

of nanotechnology to biomedicine. In this talk the theranostic and multimodal approach of novel nanostructures fabricated in the Centre will be discussed. Emphasis will be made on nanocarriers of nucleic acids and various drugs, including the polymeric star and hybrid magnetic and nonmagnetic core-shell structures, semiconducting coreshell CuInS2/ZnS nanocrystals and multiwall carbon nanotubes filled with magnetic particles.

LUNCH

Wednesday afternoon, 18 September, 12:40

NANO-FORCE NANO-DEALS. Polish Research Projects: Nano-Bio technology

Wednesday afternoon, 18 September, 13:30 *Chair: Stefan Jurga*

13:30

Polish Research Projects

Development of resorbable sealing patch for the prevention of anastomotic leakage after colorectal cancer surgical treatment

Władysław Fediuk, Sabina Urbańska-Gołąb

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Acronym: AnastomoSEAL

<u>Title</u>: Development of a resorbable sealing patch for the prevention of anastomotic leakage after colorectal cancer surgical treatment

Project no.: 280929

Call (part) identifier: FP7-NMP-2011-SMALL-5

Duration: May 1st, 2012 - April 30th, 2015

Consortium:

- 1. Universita Degli Studi Di Trieste, Trieste, Italy
- 2. Universiteit Maastricht, Maastricht, The Netherlands
- 3. Sigea SRL, Trieste, Italy
- 4. RESCOLL RC, Pessac Cedex, France
- 5. FMC Biopolymer AS FMC, Sandvika Norway
- 6. Innovation Enterprise "IMPULS" mgr inż Władysław Fediuk, Gdansk, Poland

Introduction:

AnastomoSEAL is a collaborative three-year project co-funded by the European Commission under the 7th Framework Program. The project is carried out under the theme "Biomaterials for tissue engineering for age-related cancer and sensory organ diseases".

The purpose of the AnastomoSEAL project is to design a resorbable, biocompatible and biodegradable patch to counteract anastomotic leakage, which can be a consequence of the gastrointestinal surgery.

Anastomotic Leakage (AL) is a major complication of any bowel resection, which occurs when proper and rapid regeneration of the resected bowel extremities do not take place. It has a negative impact on the health of the patient and often leads to death.

Colorectal cancer is one of the highest causes of cancer related death in Europe and it is the second most common tumor in Poland for both men and women. The number of AL cases in Europe varies between

different countries. Numbers up to 21% of patients and mortality rates up to 39% of cases are reported.

The occurence of AL after surgery requires longer treatment, application of new drugs and often re-operation. These factors raise the costs of the patient therapy. The solution of the project helps to eliminate or reduce the risk of AL and would lead to significant savings for the health care.

Methods:

The patch will be made from natural-derived bioresorbable polysaccharides. The patch is intended to be used at the end of the gastrointestinal procedure and is placed around the newly formed bowel anastomosis. The patch is designed to promote tissue regeneration, and to seal the external part of the intestine, which will increase early structural integrity and reduce leakage.

Results:

First prototypes of the biopolymeric patch have been prepared in different sizes and shapes. The test methods for characterization of the properties of the patch (*in vitro*, *ex vivo* and *in vivo*) have been developed and are continuously updated. Different sterilization procedures have been investigated and selected methods are found eligible for this sensitive product.

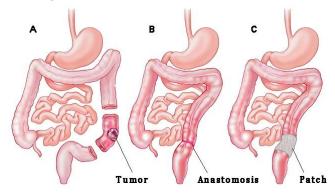


Figure 1. A) Colorectal cancer resection; B) Sutured bowel (anastomosis); C) Sealing of the anastomosis with a biomaterial patch.

The AnastomoSEAL consortium involves:

Two academic units: University of Trieste and Maastricht University; Four industrial partners: RESCOLL, SIGEA, FMC Biopolymer, IMPULS.

The University of Trieste is the project coordinator.



13:45

Polish Research Projects

New generation barrier materials protecting man against harmful impacts of the environment - POIG 01.03.01-00-006/08 ENVIROTEX

Jadwiga Sójka-Ledakowicz¹, Wojciech Czajkowski¹, Teofil Jesionowski², Anetta B. Walawska¹

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ENVIROTEX individual key project no. POIG 01.03.01.-00-006/08

Operational Programme Innovative Economy 2007-2013

Priority Axis I Research and development of modern technologies

Measure 1.3 Support for R&D projects for entrepreneurs carried out by scientific entities

Submeasure 1.3.1 Development projects

Project duration: 28.02.2007 - 28.02.2014

Head of the Project team: dr eng. Jadwiga Sójka-Ledakowicz, Associate Professor

Project is co-financed (85%) by European Regional Development Fund

Project leader: Textile Research Institute (IW), Łódź

Partners:

Wroclaw Technical University, Faculty of Electronics, Institute of Telecommunication, Teleinformatics and Acoustics and Faculty of Electrical Engineering, Institute of Electrical Engineering Fundamentals

Poznan University of Technology, Faculty of Chemical Technology, Institute of Chemical Technology and Engineering

Nofer's Institute of Occupational Medicine, Lodz

Central Institute for Labour Protection - National Research Institute, Warsaw

The Institute of Security Technology Moratex, Lodz

Main research areas:

Barrier materials protecting against electromagnetic fields

Barrier materials protecting against ultraviolet radiation

Barrier materials protecting against static electricity

Modifiers for barrier materials protecting against UV radiation

Barrier camouflage materials protecting against UV - VIS, IR and microwave

The aim of research works of the whole project is:

- technology development of new generation barrier materials based on textile carriers/substrates, protecting men against harmful impacts of the environment: electromagnetic fields (EMF), static electricity as well as ultraviolet radiation (UV) visible, infrared and microwave,
- introduction onto local and European market newly developed materials which will enlarge the range of Polish made protective goods matching the quality and innovative character of world class products.

BARRIER MATERIALS PROTECTING AGAINST ULTRAVIOLET RADIATION

One of the main tasks within the project concerned protection against ultraviolet (UV) radiation emitted by natural and artificial sources. The task aim was the development of textiles which could be applied as elements for work apparel protecting men in workplaces and as protective covers (blinds, screens) for museum collections, old prints, etc.

Currently people are more aware of the harmful influence of UV onto human skin. Medical tests confirm that UVB range is responsible for changes occurring in DNA structure as the result of photo-chemical processes; this might cause skin cancer. Workers performing their job in the open air (building, fishery, farming) are mostly exposed to the danger of serious skin changes due to sun rays.

With reference to UV sources it was necessary to develop the range of protective textiles which could be applied for different UV waverange expositions, e.g. only for UVC, UVA and UVB, UVC and UVB, or the whole UV spectrum.

Functionalisation of textiles by nanotechnology methods, especially by introduction to textile substrate or into its surface functional nanoparticles allowed to obtain new textile UV barriers.

A range of new absorbers of UV radiation has been developed to enhance barrier properties of textiles:

- inorganic compounds: nanoparticles of titanium dioxide or zinc oxide,
- organic compounds: non-ionic "direct" dyestuff type (anionic or cationic) or "reactive" dyestuff type,
- compositions of organic and inorganic substances.

The studies were directed onto the development of new organic UV absorbers and UV blockers in the form of micronized particles of metal oxides (Ti, Zn) and hybrids (TiO₂/SiO₂, ZnO/SiO₂). The surface of micro- and nano-particles of metal oxides and their hybrids was additionally modified.

Inorganic compounds:

The achieved metal oxides were incorporated into the structure of textile fabrics applying dip coating and coating methods. A very important stage was the suitable pre-treatment of textile substrate to enhance its adhesive properties. It has been stated that textile fabrics modified with metal oxides show:

- good barrier properties against UV radiation,
- very high photo-oxidative activity and the inhibitory activity towards microorganisms. Barrier materials achieved by incorporation of micronized particles of ${\rm TiO_2}$ or ZnO to textile fabrics can be successfully used for elements of individual protection items and as elements of interior furnishing.

Organic compounds

Within this project the synthesis of a range of new, efficient UV absorbers was worked out. Obtained compounds display high affinity for cellulose fibres and significant molar absorption in UVB range of radiation. So called "direct type" absorbers show increased resistance to hydrolysis when compared with "reactive type" absorbers. New absorbers can be applied on fabrics the same way as direct dyes during dyeing process, or used as additives to common detergents and siftening liquids. In case of fabrics made of cellulose fibres it is adavantageous to apply absorbers which in their particle contain systems able to react with cellulose hydroxyl groups. Chemical bonding of absorber with fibres provides long-term barrier properties resistant to multiple washing cycles.

As a reslut high-tech barrier materials (UPF > 40) for special applications have been produced which are suitable for protective clothing for various profession groups (farmers, road and rail workers, food industry workers) and to protect museum collections (art and book collections, archives, etc).

Achieved absorbers - inorganic and organic, and textile fabrics modified in laboratory conditions, after detailed tests, carried out in Institute of Occupational Medicine, Have been considered as safe for potential users. They have been applied to obtain protective clothing containing cotton and bamboo viscose fibres providing high comfort of wearing. New fabrics have been used in clothing items, i.e. shirts, caps, hand covers - a trial batch has been tested in real conditions and approved for work apparel/protective covers by the workers of:

Municipal Transport Service (MPK) in Lodz - road and rail workers (natural UV),

Polish Security Printing Works (artificial UV), Food industry company (artificial UV),

Strzemiński Academy of Fine Arts Lodz (artificial and natural UV).

Project achievements / UV area

Publications: 23

Patent applications: in Poland, abroad 2

Utility designs: 1; Industrial designs: 3; Trademark: 1

Conference presentations: ca. 20

Currently the project team enters into commercialisation procedure of:

- Technology of obtaining inorganic modifiers
- Technology of organica UV absorbers
- Method of obtaining UV barrier materials for elements of individual protection equipment (apparel, caps)
- Technology of obtaining UV barrier materials for special applications (e.g. book collection covers, old prints covers etc.

Presented technologies and products have been awarded at international fairs of inventions, e.g. in Brussels (Brussels Innova), Paris (Concours Lepine), Nuremberg (IENA), Warsaw (IWIS), Seul (Women Inventors).

This publication was prepared within the key project ENVIROTEX - PO IG no. 01.03.01-00-006/08 co-financed from the funds of European Regional Development Fund within the framework of the Operational Programme Innovative Economy 2007-2013.

14:00

Polish Research Projects

Synthesis of doped sol-gel nanoparticles and examination of their photophysical and biological properties

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The project "Synthesis of doped sol-gel nanoparticles and examination of their photophysical and biological properties" (2008-2011) for a young researches who start scientific carrier (PhD student), was financed by the Ministry of Science and Higher Education Project.

This project was focused on a preparation of silica- and titania-based nanomaterials doped with silver nanoparticles and subsequent characterization of structural, optical and antibacterial properties of the obtained compounds in order to apply in the enhanced antimicrobial photodynamic therapy (APDT).

The main tasks of this work were:

- Synthesis of silica and titania matrices by sol-gel method and examination of their physical and structural properties.
- Immobilization of silver nanoparticles on the surface of silica and titania matrices and examination of their physical and structural properties.
- Examination of optical and structural properties of nanosilver and photosensitizer doped sol-gel materials.
- Examination of the enhancement of the photodynamic effect in the presence of silver nanoparticles.

 Examination of biological properties of synthesized biomaterials doped with biologically active silver nanoparticles and photosensitizers.

The ceramic powders (silica nanoparticles and titania nanoparticles in amorphous and crystalline form) were prepared by modified sol-gel method, so thus to obtain a homogeneous distribution of particle sizes. Silica and titania nanoparticles were used as support for immobilization of silver nanoparticles.

Structural properties were characterized by TEM and SEM followed by computer aided image analysis. The nanoparticles of the following diameters were synthesized: silica nanoparticles with the diameter 180±30 nm, amorphous titania nanoparticles in two morphological structures with the diameter about 113±20 nm and 205±25 nm and titania nanoparticles in crystalline form with the diameter about 30 nm were obtained. The average size of silver nanoparticles in silica and titania nanopowders were about 14–35 nm.

Atomic Absorption Spectroscopy (ASA) for determination of total metallic silver concentration was used. The results confirmed that the Ag-SiO $_2$ based materials may contain 8.53% of silver. The average concentration of silver in Ag-TiO $_2$ nanomaterials in amorphous form can be as high as 11.68% and in crystalline form 6.32%.

Attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR) for analyze the composition of the synthesized nanomaterials revealed characteristic vibrations of Si–O–Si, Ti–O–Ti bond and hydroxyl groups for appropriate materials were observed.

Chlorine e6 based photosensitizer Photolon in various concentration was used as dopant. The absorption spectrum of silver doped silica and titania nanoparticles had a maximum between 420–445 nm, which is characteristic for silver nanoparticles. It was proved that for certain concentration of metallic silver in Photolon solutions the absorbance is high.

Antibacterial properties of synthesized nanomaterials against Grampositive and Gram-negative bacteria were tested. Minimal inhibitory concentration of metallic silver was determined. Antimicrobial photodynamic activity against *Pseudomonas aeruginosa* was examined. Tested samples were non irradiated and irradiated with laser light 662 nm. The silver doped silica and titania nanoparticles had antibacterial activity, but in the presence of Photolon, the antibacterial activity of nanomaterials in APDT was much more effective.

The results of the project showed that it is possible to create a new system to fight with growing number of drug resistant bacteria. APDT efficiency may be enhanced by metallic nanoparticles with antibacterial activity. Photosensitizers combined with nanomaterials can be applied to eliminate bacteria, viruses, fungi and parasites.

14:15

Polish Research Projects

Polymers Nanocomposites with Increased Resistance Against Microorganisms

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Polymers Nanocomposites with Increased Resistance Against Microorganisms realized within The Operational Programme Innovative Economy, financial perspective between 2007-2013 Priority axis 1. Research and development of modern technologies, Measure 1.3 Support for R&D projects for enterpreneurs carried out by scientific entities, Sub-Measure 1.3.1 Development projects. The project was realized

by Consortium: § INDUSTRIAL CHEMISTRY RESEARCH INSTI-TUTE, Warsaw - COORDINATOR & UNIVERSITY OF TECHNO-LOGY AND LIFE SCIENCES, Bydgoszcz § OPOLE UNIVERSITY § TECHNICAL UNIVERSITY OF LODZ § CHEMICAL PLANT POLISH SILICONES Ltd., Nowa Sarzyna The aim of the project was to develop the innovative technologies of silica nanofillers and polymer nanocomposites with enhanced resistance to microorganisms (fungi and / or bacteria) and with biocidal properties. Biocidal properties of silica nanofillers were achieved due to immobilization of silver or copper nanoparticles on the surface of silica nanospheres. The most important advantages of polymer nanocomposites containing the developed additives are high antimicrobial and antifungi activity as well as improved mechanical, thermal and barrier properties and can be applied eg. in kitchen appliances, kitchen, bathrooms, hospitals and other public facilities. The developed technologies will make the use of natural products such as wood fiber and the recycling of plastics waste possible, which offers a positive effect on the environment, as an added-value. The developed technologies are the key factor for increasing the value of innovative products offered by manufacturers producing and applying polymeric materials. Substantive outcome of the project is the development of the following optimized processes: · manufacturing of nanosilica containing immobilized silver or copper nanoparticles · manufacturing of compatibilizer · manufacturing of polimer nanocomposites with incresed resistance to microorganisms The results of research carried out under the project support the assumption that the incorporation of nanosilver and nanocopper provides a stable nanostructure of these metals and, consequently, nanocomposite of expected and satisfactory properties can be obtained. The investigations have shown that nanoparticles of silver and copper built in with the polymer matrix (PE, PP, PVC) are not released to the environment. A comparative analysis of the results show that antibacterial activity is not dependent on the type of polymer but the concentration and distribution of the nanoparticles, both silver and copper. Considering various techniques receiving the composites, it was found that the compression of the material in the form of a thin layer or double-sided surface of the biocides reducing the susceptibility of the material to bacterial adhesion and to effectively shortens their lifespan. Both the copper and the silver introduced in the form of immobilized on nanosilica in concentrations of 5-15% are effective in reducing the viability of the bacteria. The bacteria E. coli, S. aureus, S. Typhimurium are more sensitive than the bacteria isolated from the environment, such as Listeria monocytogenes and Burkholderia cepacia. Moreover, the addition of wood flour composites increases the adhesion of bacteria, but they lose a significant metabolic activity. This effect is an expected consequence for the modified polymer nanocomposites. Any structural changes in the modified polymers treated with bacteria after three months were not found by using infrared spectroscopy and Fourier transformation methods. The solution is competitive with conventional plastics and polymer composites due to the unique properties of these materials resulting from the use of nanofillers, which built into the polymer chains (polyolefins and PVC) increase their barrier properties, strength (eg thermal resistance) and resistance to microorganisms. The technology of nanosilica with built-in copper or silver nanoparticles was confirmed in pilot trials carried out in the Chemical Plant Polish Silicones Ltd., Nowa Sarzyna. The results of the project are as follows: technology of manufacturing nanofillers and polymmer nanocomposites (6 technologies) · 5 patent applications in Poland and 2 international patent applications (PCT mode) covered by the separate project of Innovative Economy · several published papers · 2 dissertations for Masters Degree · experimental part of two PhD as well as several awards on international invention fairs eg. gold medal with distinction at the Brussels Innova 2010 fairs and the Cup of the Minister of Economy.

COFFEE and POSTERS

Wednesday afternoon, 18 September, 14:30

E-MRS Plenary session

Wednesday afternoon, 18 September, 16:00

Thursday, 19 September

REGISTRATION

Thursday morning, 19 September, 8:00

Opening

Thursday morning, 19 September, 9:00 *Chair: Jarosław Piekarski*

9:10

Polish Research Projects

Highlights on Polish participation in FP7 NMP calls

Jarosław M. Piekarski

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Highlights on Polish participation in FP7 NMP calls

Jarosław Piekarski

Polish National Contact Point for Research Programmes of the European Union (KPK) at IPPT PAN, *Krzywickiego 34, 02-78 Warsaw, Poland*

Abstract

NMP Theme (Nanosciences, nanotechnologies, materials & new production technologies) constitutes fourth largest theme of the FP7 Cooperation Specific Programme (2007-2013) with the 7-year budget $3.4B \in$. But, due to an enabling role the NMP-related activities have been present also in other areas of FP7. For instance, in the report ??? was shown that the FP7 expenditures for nanotechnologies-related projects attained $3,45B \in$ while the budget attributed to Nanotechnologies within NMP Theme was about $1,17B \in$.

In the presentation the participation of Polish teams in FP7 projects related to nanosciences and nanotechnologies, advanced materials and manufacturing is discussed. Poland is eleven as concern the number of participants and fifteenth as concern received EC contribution???. NMP projects contribute significantly to Polish participation in FP7 Cooperation Specific Programme, with 14% share in PL participation and 16% share in received EC funds, while NMP Theme funds constitutes less than 11% of total Cooperation budget.

The NMP Theme objective is to support applied research. Therefore the participation of industry is a must. Polish industrial partners are present in a number FP7 NMP projects, their average share among Polish participants is similar to the total share of industry. Small and medium-side enterprises from the construction sector are particularly active. The share of Polish industry attains 70% in calls of Energy Efficient Buildings Initiative.

Polish researchers are also active in initiating investigation. Eight cooperation international projects leaded by Polish coordinators received funding from FP7 NMP Calls. Among them are *ENSAMBLE*, *MAT*-

RANS, SUPERSONIC, NANOMINING, NANOPUZZLES, presented during the NAMF2013 Conference.

??? Nanotechnology: the invisible giant tackling Europe's future challenges, EUR 13325 EN, 2013

??? Udział Polski w 7 Programie Ramowym. Statystyki po zakończonych 355 konkursach. Warszawa, maj/czerwiec 2013 (http://www.kpk.gov.pl/pliki/12715/7PR Raport po 355 konkursach.pdf)

9:30

Invited oral

The new SEM analysis reality from Bruker

Thomas Grafe

Bruker Nano GmbH, Berlin 12489, Germany

e-mail: thomas.grafe@bruker-nano.de

Having 5 cutting edge analytical tools for SEM out of one source now becomes reality.

Discover the new Bruker range offering EDS, EBSD, WDS, Micro-CT and Micro-XRF analysis opening up unrivalled analytical possibilities.

Polish Research Projects: ICT applications of nanotechnologies

Thursday morning, 19 September, 10:00

Chair: Łukasz Nieradko

10:00

Polish Research Projects

InTechFun: Innovative technologies of multifunctional materials and structures for nanoelectronics, photonics, spintronics and sensor techniques

Anna Piotrowska

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e-mail: ania@ite.waw.pl

Web-site: http://www.ite.waw.pl/intechfun/

Programme: Operational Program Innovative Economy, National

Cohesion Strategy

Project co-ordinator: Institute of Electron Technology, Warsaw

Starting date: 01.09.2008 **Duration:** 64 months

Participants: Institute of Electron Technology

Institute of Physics Polish Academy of Sciences

Warsaw University of Technology

Silesian University of Technology

Technical University of Lodz

Military University of Technology

Project aim

The project is aimed at the development of innovative technological processes and designs, and based on these, novel semiconductor devices making use of wide bandgap semiconductors – ZnO, group III-nitrides, and SiC. The research embraces the following main topics:

- 1. doping of ZnO in p-type,
- 2. epitaxial growth of GaN/AlGaN structures on Si substrates,
- 3. fabrication of photonic crystals in GaN and ZnO,
- 4. fabrication of metal/semiconductor contacts with specific electrical and optical properties (ohmic contacts, Schottky barriers, transparent contacts), suitable for high temperature operation,
- 5. fabrication of semiconductor/dielectric with specific electrical properties (gate dielectric, surface passivation).

The research includes modeling studies and thorough characterization of materials and device structures. High level of R&D activities The leading group for this project, the Dept. of Micro- and Nanotechnology of Wide Bandgap Semiconductors (http://www.ite.waw.pl/en/Z03.php) has a broad experience in the process technologies and device architectures for optoelectronics and electronics. Relevant experience includes processing of III-V devices involving nanostructures for photonics, high frequency, high-power & high-temperature electronics, development of thermally stable ohmic contacts toGaN, surface passivation methods, TCOs, p-type ZnO. The team was involved in a number of externally funded research projects on both national and international levels. Relevant European projects (6th& 7th FP) include: TERAVI-SION, AGETHA, AMORE, DENIS, NANOPHOS, HYPHEN and MORGAN. The Department is teamed with the Laboratory of Low Temperature Physics at the Institute of Physics of Polish Academy of Sciences and is a part of National Network of Excellence"New Materials and Sensors for Optoelectronics, Information Technologies, Energy, and Medicine". The processing laboratory is fully equipped for device fabrication by optical and DUV lithography, nanoimprint and laser lithography and ICP/RIE etching. The key facilities used in the project include thin film deposition systems (Leybold L-560 vacuum evaporation and Z-400, Z-550 DC/RF magnetron sputtering systems) and UHV unbalanced magnetron sputtering system Gamma 1000C from Surrey NanoSystems Ltd., PECVD deposition module (Oxford Instruments DP 100), high-resolution photolithography (Karl Suss MJB-21 double side and MJB-3 DUV mask aligners), nanoimprint lithography (Obducat Eitre3 system) laser lithography (DWL 66FS Heidelberg Instruments), thermal processing (AST SHS 100 RTP & conventional furnaces), wet and dry chemical processing (Oxford Instruments PlasmaLab System 100ICP180), facilities for electrical, structural and optical characterisation (including Philips X-ray diffractometer and Tencor Thin Film Stress measurement system). The team has access to the extensive facilities for structural characterisation such as SIMS (Cameca IMS 6f), RBS (3SDH-2 Pelletron accelerator), advanced xray analysis, Variable Angle Spectroscopic Ellipsometry (VASE), AFM, SEM. Project results Process technologies were integrated into technological modules and their effectiveness validated in the specific electronic and photonic devices, and sensors. The demonstrators include:

- 1. AlGaN/GaN HEMTs on Si substrate,
- 2. SiC MESFETs and MOSFETs,
- 3. GaN-based 385 nm LEDs,
- 4. optical gas sensors based on ZnO and GaN.

The expected impact of the project will be establishing of interdisciplinary R&D platform capable to perform creative work, both within the project duration and in long-term timescale (minimum 5 years after the end of the project).









10:15

Polish Research Projects

Pol-HEMT: AlGaN/GaN microwave HEMT transistors on monocrystalline GaN substrates

Anna Piotrowska

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Programme: Applied Research Programme supported by the National

Centre for Research and Development

Project co-ordinator: Institute of Electron Technology, Warsaw

Starting date: 01.11.2012 **Duration:** 36 months

Participants: Institute of Electron Technology Institute of Physics Polish Academy of Sciences

Warsaw University of Technology

Institute of High Pressure Physics Polish Academy of Sciences

Ammono S.A. TopGaN Sp. z o.o.

Project aim

The objective of this project is to research and develop a new type of microwave S band HEMT (High Electron Mobility Transistor) based on novel AlGaN/GaN heterostructures grown on bulk monocrystalline semiinsulating GaN substrates. The substrates will be fabricated by ammonothermal method and their size scaled for 1" to 1.5". Two techniques - MOVPE and MBE will be used for the epitaxial growth of HEMT structures with high concentration and high mobility electron 2D gas, and improved structural quality. The fundamental approach behind the workplan is based upon the interaction between four key technical areas of expertise:

- 1. HEMTs modeling and design,
- 2. material growth and characterisation,
- 3. devices fabrication and
- 4. packaging and chip assessment.

A number of specific processing steps will be optimised including the definition of the active device area, RIE/ICP etching for ohmic contacts and gate recessing, through wafer via holes fabrication. Reliability issues will be of particular concern.

Key issues

One of the main issues in GaN-based HEMT technology is the lack of a lattice-matched semiconductor substrate. At the moment the best results in demanding radio frequency (RF) applications are obtained using SiC substrates. Alternatively, for less demanding applications Si substrates are used for HEMT structure growth. A significant problem arising with the application of non native substrates is the degradation of HEMT structures under high load conditions (i.e. high voltage bias resulting in the creation of high electric fields in the active structure) due to a high defect density in such epitaxial structures (most importantly in the buffer layer but also in the AlGaN barrier layer yielding charge trapping in the HEMT channel region). Further complex issues in the structurisation and mounting arise. We expect, that the research strategy of the project will enable to develop a fully nitride AlGaN/GaN HEMT technology using free-standing GaN substrates yielding a significant increase in long term reliability of the transistors.

High level of R&D activities

Poland has long been known as a country of origin of some of the most significant R&D institutions and companies in the film of GaN-based electronics. This project is based on the cooperation of the leaders in the field, in particular AMMONO S.A. specializing in bulk GaN crystal growth using ammonothermal growth yielding state-of-the-art crystal quality as well as TopGaN specializing in MOVPE nitride epitaxy used in HEMT structure growth in particular known for their blue optoelectronic devices. The processing of the transistors will be carried out by the Institute of Electron Technology using technology developed as a result of R&D works carried out since 2004 under several EU Framework Programmes and National Cohesion Strategy Programmes. Institute of High Pressure Physics PAS and Institute of Physics PAS bring into the project their unique expertise on the physics of the material as well as on MBE growth of the nitride structures and Institute of Radioelectronics of the Warsaw University of Technology, a worldwide recognized expert on the design and implementation of high power high frequency amplifiers will be responsible for the characterization and specification of the completed transistors.

Expected project impact

The HEMT realized in the frames of the project will target the needs of polish defense and civilian industry, in particular the polish radiolocation industry based on the 50 years of experience of PIT and RADWAR (currently in the BUMAR consortium). The development and future implementation of microwave high power HEMT transistors on GaN substrates will be a world-class achievement in semiconductor technology. It will form a relevant basis for simultaneous work on high power electronics, in particular for energoelectronic systems as well as for green technology systems (concerning renewable energy sources and hybrid vehicles). Another potential application area is the one of harsh environmental sensors working at high temperatures and in corrosive environments.



10:30

Polish Research Projects

Functional materials for resistive switching memories

<u>Jacek Szade</u>, Jerzy M. Kubacki, Marek Kulpa, Dariusz Kajewski, Michał M. Pilch, Roman Wrzalik, Marcin Wojtyniak

A. Chełkowski Institute of Physics, University of Silesia, Katowice 40-007, Poland

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Acronym: FMRSM

<u>Project no.</u>: NCBiR/ERA-NET-MATERA/3/2009 <u>Duration</u>: April 29th, 2009 – February 28th, 2012

Consortium:

• Research Centre (FZ) Jülich, Germany

 Institute of Solid State Physics (ISSP) at the University of Latvia in Riga (Latvia)

 Faculty of Mathematics, Physics and Chemistry, A. Chełkowski Institute of Physics, University of Silesia in Katowice

Project Coordinator: Research Centre (FZ) Jülich, Germany

Objective and methods

The aim of the project was to develop the basic science and technology for future resistive switching memories based on new functional ternary oxides. Since extended defects as dislocations or defect clusters with nanoscale dimensions are considered to be the single resistive switching units in doped ABO₃-perovskites, one has to gain deeper understanding of the complex correlation between defect structure, elementary and defect distribution and switching properties.

The project was carried out by an international research team consisting of three partners from different countries that are members of the consortium ERA NET Matera - Research Centre (FZ) Jülich, Germany, Institute of Solid State Physics (Institute of Solid State Physics-ISSP) at the University of Latvia in Riga (Latvia) and the Faculty of Mathematics, Physics and Chemistry, University of Silesia in Katowice. Polish part of the project was carried out by a team from the Institute of Physics, University of Silesia. The studied materials were in the form of epitaxial thin films of SrTiO3 doped with iron and manganese. The samples were grown in the Research Centre Jülich with the use of PLD method.

Results

Thin films of SrTiO3 doped with up to 5% iron in place of titanium have shown interesting properties of resistive switching as compared to the undoped films. This suggests that they may be used in a new type of non-volatile memory. The studies of the Polish part of the project were concentrated on detailed characterization of the electronic structure. High resolution photoelectron spectroscopy, resonant photoemission and x-ray absorption methods were applied. Our results showed the dopant contribution to the electronic states of the valence band including the energy gap. These electronic states can be related to the increased electrical conductivity within the filaments – the basic elements of the resistive switching phenomenon in SrTiO₃ based materials. The studies performed with the use of synchrotron radiation demonstrated that iron ions are in two oxidation states 2+ and 3+, and enabled determination of a partial density of states originating from iron dopant. Similar data were collected for Mn doping. It was shown that doping with manganese leads to worse switching properties in comparison to iron.

10:45

Polish Research Projects

PRIAM - "Printable functionalities for truly autonomous, intelligent lighting and signalling systems"

Andrzej J. Mościcki, Andrzej E. Kinart, Anita K. Smolarek

Amepox Microelectronics, Ltd. (AMEPOX-MC), Jaracza 6, Łódź 90-268, Poland

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Project's objective:

Seventh Framework Programme Theme 3; ICT-2009.3.3 Flexible, organic and large area electronics.

Project's website:

http://www.priam-project.eu

Project's start date:

01-01-2010

Project's end date:

31-12-2012

The Consortium:

- 1. Centro Ricerche Fiat S.C.p.A., Italy
- 2. Commissariat al Energie Atomique et aux Energies Alternatives, France
- 3. Teknologian Tutkimuskeskus VTT, Finland
- 4. Microtec Gesellschaft fuer Mikrotechnologie MbH, Germany
- 5. Centro Ricerche Plast-Optica Spa, Italy
- 6. Amepox Microelektronics Ltd., Poland
- 7. Solari di Udine S.p.A., Italy

Project's description:

The main objective of PRIAM is the **development of new autonomous road signals and lighting modules (tail lights) integrating heterogeneous functionalities on plastic foils** by high throughput homogenous processes. Specifically the project addresses the development of two prototypes on a flexible substrate: energy harvesting and storage, communication, control, sensor to measure light intensity, intelligence and light emitters.

Project's objectives:

PRIAM addresses the development of two new product families:

- light emitting autonomous road signs;
- autonomous car signals and taillights.

The underlying technology includes a plastic foil containing:

- · a solar cell;
- a thin film battery;
- solid state light sources;
- · a sensor of ambient light;
- a Radio Frequency RF communication element;
- an energy management processing unit.

The developed systems do not need to be connected to an external source of energy, there is no need of expensive cabling or dedicated infrastructures.

Organization's profile:

Amepox-Microelectronics Ltd., launched in 1991, is a producer of materials for electronics and microelectronics applications. Company's

production line includes: the highest purity silver powder and flake, formulations with silver fillers as electrically conductive adhesives, pastes and inks, thermally conductive greases and adhesives and magnetic flux conductive formulations. Amepox has many unique products like "heavy" silver powder, "heavy" copper powder, highly resistant for environment and extremely high temperature formulations or solderable formulations. The newest and the main direction of AXMC development for actual and future activity are multifunctional nanoformulations (polymer binder base formulations with nanosize of fillers) for nanotechnology purpose.

Amepox has significant achievements in this field, especially with electrically and thermally conductive nanoformulations. The organization is working with novel electrically conductive formulations for Jet Printable technology.

The organization aims to become the producer of highest purity and quality electronic materials. Amepox is working very close with several High Technical Institutes and Universities. Some of the work was presented on several international conferences and special workshops for experts for electronic industry. Amepox works very closely together with customers and is very sensitive to their requirements. We see this as a key to success. Amepox developed **new technologies for preparing extremely small silver powderand several binders which could be useful for Jet-Printing techniques**.

Amepox is a small sized company but most of its employees are highly educated and with many years of experience in materials for electronic applications filed. The organization performs all types of activities, such as marketing, production, R&D and QC lab, shipment and management. We expect to increase the number of the employees by about 25% after the completion of the project.

Organization's role in the project:

Amepox is involved in the technological development of proper electrical contacts between electro-photonic devices with inorganic light emitting nanostructures, especially with using Jet-Dispenser techniques. Moreover Amepox is involved in the development of novel packaging solutions for the proposed nanophotonic devices. Special nanoformulation for interconnection of nanostructures is connected with Jet-Printing technique, which is the best for nanosized structures. One of the main problems connected with the newest production technologies is interconnection technique in case of packaging of hi-tech circuits. This is a crucial issue, especially in case of nanosize structures. Amepox is working with this type of problems and has wide experience in this field.

COFFEE and POSTERS

Thursday morning, 19 September, 11:00

Polish Research Projects: Nanomaterials in transport, construction and energy applications

Thursday morning, 19 September, 11:20 *Chair: Malgorzata Sopicka-Lizer*

11:20

Polish Research Projects

Supersonic deposition of nano-structured surfaces

Jan Kusiński, Kazimierz Kowalski, Sławomir Kąc

AGH University of Science and Technology, Faculty of Metals Eng. and Industrial Computer Sci., al. Mickiewicza 30, Kraków 30-059, Poland

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Acronym: SUPERSONIC

Title: Supersonic deposition of nano-structured surfaces

Project no.: FP7-NMP-2008-LARGE-2 CP-IP 228814-2

Duration: December 1st, 2009 - November 30th, 2013

Consortium:

- 1. AGH University of Science and Technology, Kraków, Poland
- 2. MBN Nanomaterialia Spa, Carbonera, Italy
- 3. Katholieke Universiteit Leuven, Leuven, Belgium
- 4. Impact Innovations GmbH, Heldenstein, Germany
- 5. Universitat de Barcelona, Barcelona, Spain
- 6. Matres Scrl, Treviso, Italy
- 7. SKF Aerospace France, Saint Vallier, France
- 8. Alhenia AG, Baden-Dättwill, Switzerland
- 9. Institutul National de Cercetare, IMNR Pantelimon, Judet Ilfov, Rumania
- 10. Granta Design Ltd, Cambridge, United Kingdom
- 11. Falex Tribology NV, Rotselaar, Belgium

Objective and methods:

The main aim of the project was to select coating materials, to produce powders using mechano-chemical reactions by High Energy Ball Milling (HEBM) and to deposit coatings on engineering components by means of Cold Gas Spraying (CGS) suitable for 3 different industrial applications:

- 1. Aeronautical: coatings for high temperature resistant and long life load bearings for front and rear aircraft pylon attachments.
- 2. Mechanical: abrasion resistant and fretting resistant coatings for aluminum alloys (floorboards, press-fitted gear wheel and hollow shafts in wind turbines).
- 3. Biomedical: a new class of nanostructure Ti based coatings intended for implant joints of elbow, knee and hip of improved performances, biocompatibility and high wear resistance.

Selection of coating materials and substrates for the defined end-user applications based on experimental and theoretical feedback from the partners. Material design methodology was also aimed to establish the methods of materials characterization. Further activity consisted of development of the post deposition finishing and the process efficiency enhancement. The decisive role played the industrial end-users qualification tests of coatings. The crucial point of the project was to integrate the production of the appropriate powder with the coating technology and thus the project was focused on:

- 1. Investigation and development of HEBM synthesis route of reactive and non-reactive powder systems that could be suitable for final application:
- 2. Development of CGS equipment;
- 3. Development of supersonic CGS deposition process for nanostructured systems.

Results:

A detailed overview of the materials characteristics identified for each application have been provided by the end-users involved in the pro-

ject. Eco-design software for material selection was created. A case-by-case study to define the boundary conditions related to the performance required by the end-users provided input to this task by the creation of a knowledge capture template designed to encourage the end-users to think about the factors which would influence the selection of appropriate materials for their applications. An objective in the definition of the planning of a matrix of systems was to enhance the wear and friction characteristics of the components whilst minimizing the environmental impacts. Based on key design requirements, drawing upon feedback from thermodynamic modeling and tribological testing, and taking into account processing capabilities and constraints, the initial list of candidate material systems has been refined.



Successful systems:

- 1. For aeronautical applications Ni-NiSn (nickel with intermetallics) coating on steel of the following characteristics: dense, thick and nanostructured; higher hardness and better friction behavior against temperature; elevated temperature corrosion resistant; coefficient of friction better than benchmark.
- 2. For biomedical applications Ti-TiC (titanium reinforced) coating on Ti6Al4V alloy of the following characteristics: dense, thick and nanostructured; enhanced hardness and wear resistance; biocompatibility.
- 3. For mechanical applications FeCuAl-Al₂O₃ (FeCu reinforced by alumina) and Co-WC (WC in cobalt matrix) on aluminum alloys of the following characteristics: dense, thick and nanostructured; higher hardness and much better abrasive resistance than the substrate; coefficient of friction better than benchmark; high repeatability.

For each application a powder with the right characteristics for cold spray was achieved, safety procedure for most reactive powder were well defined, some refinement work will be carried out to increase production yield and to optimize process. To achieve a high coating quality it is very important that every process step is executed with the maximum attention starting from powder production. Powder quality control involves: powder shape, size, reactivity and particle size distribution. The supersonic deposition process controlled parameters are following: feeding rate, nozzle (different, depending of deposited material) working gas temperature, variable gas pressure, particles velocity and a stand-off distance. One of the project aims is also to use reactive powders. Considerable effort was devoted to overcome problem of nozzles clogging because powder tends to react inside it and so to block it. To do this a special powder feeder as well as dedicated nozzles were developed. Some work was concentrated on setting the prequalification test of coatings and adaptation of the existing equipment for tribological experiments. The appropriate tests for the evaluation of abrasion resistance as well as fretting wear resistance of the coatings have been defined.

11:35

Polish Research Projects

Technology of diffusion (ZrO₂+Y₂O₃)+Ni-Al-type composite layers deposited on the blades of aircraft engine turbines

Ryszard Sitek

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Project No: N R15 0007 06

Duration: September 1st, 2009-August 30th, 2012

Objective and methods:

The goal of the project was to produce Thermal Barrier Coatings (TBC/BC) on the aircraft engine turbine blades cast of Inconel 100 and Inconel 713C. The coatings were produced in the three stages:

- a) deposition of a diffusion layer which contains the AlNi intermetallic phase using AlCl₃ vapours in an atmosphere of hydrogen as the carrier gas (Chemical Vapour deposition CVD);
- (b) plasma spraying of the $(ZrO_2+Y_2O_3)$ thermal barrier;
- (c) glow discharge-assisted annealing.

The thermal barriers thus produced were examined in NIKON EPI-PHOT 200 and HITACHI SU70 electron scanning microscopes with a micro-area analyser (EDS-Energy Dispersive Spectroscopy), manufactured by the ThermoNoran Co. The phase composition was analyzed based on the X-ray diffraction patterns obtained using a Bruker D8 instrument with CuKα radiation. The heat resistance was examined in air at a temperature of 1100°C. Samples with and without the thermal barrier were subjected to 24h/1h and 24h/24h heating-cooling cycles which were highly dynamic since the samples were placed in a furnace heated to 1100°C and then cooled in air at ambient temperature. After each cycle, the samples were examined with the naked eye and in a microscope. In some selected samples, the chemical composition of the individual layers was analyzed. The chemical composition of a cross-section surface of the samples was examined by XPS (PHI 5000 VersaProbe).

Results

By combining (CVD) aluminizing deposition, plasma spraying, and final heat treatment realized by glow discharge annealing we can produce a $(ZrO_2+Y_2O_3)+AlNi$ thermal barrier on the surface of turbine blades cast of the Inconel 100 and Inconel 713C nickel superalloy (Fig. 1). The AlNi intermetallic layer evidently reduces the diffusion of chromium into the outer $(ZrO_2+Y_2O_3)$ thermal barrier. Due to its diffusive character, the thermal barrier coating adheres well to the substrate and shows exceptionally good corrosion resistance - especially at high temperatures.

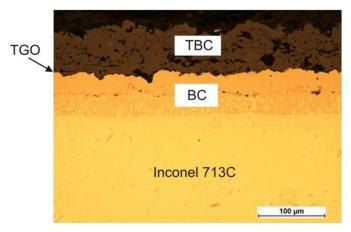


Fig. 1. Microstructure of the TBC/BC thermal barrier formed on an INCONEL 713C nickel superalloy substrate

11:50

Polish Research Projects

NanCore: Microcellular nanocomposite for substitution of Balsa wood and PVC core material

<u>Kinga Jurczuk</u>, Andrzej Galeski, Ewa Piorkowska, Marcin Zarod, Jerzy Morawiec, Zbigniew Bartczak, Artur Rozanski, Ewelina Szkudlarek

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CMMS PAS - contractor of Work Packages: WP2 and WP6 in the NanCore project. Andrzej Galeski, coordinator from Polish side

Project acronym: NanCore

Project full name: Microcellular nanocomposite for substitution of Balsa wood and PVC core material, NMP3-LA-2008-214148

Name of programme and competition: 7th Framwork Programme UE, Theme 4.

FP7-NMP-2007-2.1-1, Nano-structured polymer-matrix composites

Application year: 2008

Implementation time of the project: 1st December 2008 - 30st November 2012

Participants of the project:

- 1. LM Glasfiber A/S recently known as LM WindPower Blades, LMG, Denmark (coordinator of the project)
- 2. Aalborg University, AAU, Denmark
- 3. Huntsman Polyurethanes, HPD, Belgium
- 4. Katholieke Universiteit Leuven, KUL, Belgium
- 5. Universität Kassel, UKA, Germany
- 6. University of Valladolid, UVA, Spain
- 7. Azimut-Benetti S.P.A., AB, Italy
- 8. Centre of Molecular and Macromolecular Studies, CMMS, Poland
- 9. Institute of Occupational Medicine, IOM, United Kingdom
- 10. Technical University of Denmark, DTU, Denmark
- 11. EconCore N.V., Econ, Belgium
- 12. FOCAL Limited, Focal, United Kingdom
- 13. Sekisui Alveo AG, S-A, Switzerland

The principal objective of the NanCore project was to design a novel microcellular polymer nanocomposite (MPNC) foam, with mechanical properties and cost characteristics allowing for a substitution of Balsa wood and PVC foam as core material for lightweight composite

sandwich structures. Specifically, the NanCore project aimed to achieve a cost reduction of at least 30 percent for the finished MPNC core material as compared to current core materials.

Project activities begun with manufacturing and end-user requirement specifications, proceeded to scientific and technological development within each of the project's core problem areas, and finalized with test and demonstration of the developed core material. Project activities were divided into following Work Packages (WPs):

WP1 Requirement specification with regard to process, production and mechanical characteristics

WP2 Selection of functionalized components of polymer nanocomposites

WP3 Foaming of nanocomposites

WP4 Industrial processing of microcellular nano-composites

WP5 Object oriented characterization, multiscale modelling and simulations of microcellular nanocomposites

WP6 Integration of the MPNC into a sandwich structure

WP7 Demonstration and full scale testing

WP8 Life Cycle Assessment of new core material

WP9 Safety Issues of Nanoparticles from Nanocomposites

WP10 Dissemination and Exploitation

WP11 Consortium Management

CMMS participated in several Work Packages of the NanCore project: mainly CMMS coordinated WP2. WP2 was focused on providing base materials for WP5, WP4 and WP3. The main task of WP2 was to prepare polypropylene, PP, nanomaterials with high melt strength, which would be used for foaming. CMMS developed and produced two types of nanocomposites including PP reinforced with polytetrafluoroethylene, PTFE, nanofibers and PP with montmorillonite, MMT.

The idea for simplification of producing nanocomposites with nanofibrillar reinforcement was to find a way to strongly deform crystalline inclusions (i.e. PTFE powder grains) during shearing or compounding and preserve the shape of generated PTFE nanofibers in the PP matrix. Pilot experiments on compounding devices using PP of high melt viscosity and reaction grade PTFE clearly showed that it is relatively easy to generate all-polymer nanocomposites with nanofibrillar reinforcement just by compounding the two polymers. One of the most interesting feature for NanCore project of PP+PTFE nanocomposite is its high melt strength and high extensional viscosity, the property that is required for efficient foaming. PTFE nanofibrils built up a continuous network, which proved to be necessary for melt strengthening even for PP matrix that originally does not show any melt strength. The discovery of the method of all-polymer nanocomposite fabrication was patented first in Poland and then applied for European patent.

CMMS established optimal preparation protocol for PP nanocomposites with nearly fully exfoliated MMT platelets, describing all steps of the process from clay drying to nanocomposite pelletizing. This protocol was also a base for preparation by IOM a document for health, safety and environmental precaution for research conducted in other WPs. A new staining method for the purpose of transmission electron microscopy, TEM, was developed to show compatibilizer localization in PP+MMT nanocomposites. Using osmium tetraoxide, it was possible to prove that compatibilizer adheres to MMT platelets in a semi-uniform fashion. It also confirmed that no platelets exfoliation could occur without compatibilizer surrounding. Observations were used in molecular modelling in AAU and KUL. CMMS also has proven that even a complete MMT exfoliation does not guarantee melt strengthening during the foaming process. Hence, the use of high melt strength PP for foamable nanocomposites is strongly advised and recommended.

The effort of CMMS in the NanCore project was also focused on nanocomposites foaming. CMMS gathered observational data linking foam structures with processing routes. Additionally, CMMS developed

new foam examination method based on Neutron Magnetic Resonance. It showed that addition of MMT and foaming alters spin relaxation behaviour of CHx groups in PP chains. Data gathered from this method were used for modelling tasks.

Laboratory trials performed in CMMS have proven that melt strengthening of a nanocomposite is more important for foaming than the strength in solid state. During the up-scalling tests, it was also proved that heat transfer from interior of a foam and its dissipation is crucial in larger scale nanocomposites foaming. Following the problems with upscaling the foaming process, it was decided to use another foaming technique, focusing on micro-bead technology foaming. CMMS examined materials from all stages of industrial foaming trials. Micro-bead structure analysis revealed that foams have fully closed cell structure and have a density within the range desired by the project. Providing materials for successful foaming marks the success for the WP2. Both PP+PTFE and PP+MMT nanocomposites have foaming potential, which will be utilized during the future research and industrial trials.

The following industrial companies expressed their interest in applying developed polypropylene nanocomposites for foaming or moulding, all of them according microbead technology: ANTOS Chemical Co., Zamosc, Poland; HSV Co., the Netherlands; Izo-Blok, Poland; and Neue Materialien Bayreuth GmbH, Germany.

12:05

Polish Research Projects

Graphene nanocomposite for the reversible hydrogen storage

<u>Piotr Kula</u>, Robert Pietrasik, Konrad Dybowski, Radomir Atraszkiewicz, Witold Szymański, Łukasz Kołodziejczyk

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Acronym: GraphRoll

<u>Title:</u> Graphene nanocomposite for the reversible hydrogen storage

Project no.: GRAF-TECH/NCBR/07/24/2013

Duration: January 1st, 2013 - December 31st, 2015

Consortium:

- Lodz University of Technology, Łódź, Poland
- 2. SECO/WARWICK S.A., Świebodzin, Poland

Objectives and methods:

The main aim of the project is to work out the technology for producing the functional nanocomposite material "GraphRoll" on the basis of polycrystalline graphene for reversible storage of hydrogen. The area for a potential application of the new nanomaterial in the nearest time horizon will be industrial technological facilities for heat and thermochemical treatment where hydrogen is a process medium or is used in high pressure gas quenching. Pumpless compressors based on "GraphRoll" will be provided for hydrogen recycling in the processes of High Pressure Gas Quenching (HPGQ) as well as for hydrogen separation from exhaust mixtures of gases during operations of thermochemical treatment (low pressure carburizing - FineCarb and PreNitLPC).

Moreover, upon further improvement of the technology, it is expected that these new materials, when reaching the critical threshold of 6.5%

wt. hydrogen absorption on an industrial scale, will be used for the storage of hydrogen in the vehicles of the future.

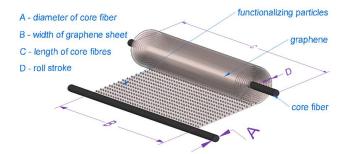


Figure 1. The idea of nanocomposite material "GraphRoll"

The nanostructure of that nanocomposite is based on the original method of polycrystalline graphene synthesis from the liquid phase. In the next stages the graphene is decorated by PVD spark and/or laser sputtering, then separated from the surface and finally rolled helically around the core fiber to reach 3D helical nanostructure with strongly developed area of active graphene with oligomeric pillars in between (Figure 1).

Results:

The obtained results confirmed that the reported method of graphene manufacturing from the liquid phase could be successfully used (Figure 2).

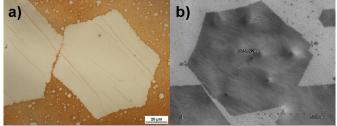


Figure 2. Optical image (a) and SEM image (b) of polycrystalline graphene

The intermediate structures could be also synthesized and it is possible to study the mechanisms of graphene nucleation and growth. Moreover this technique allows to produce large graphene panels, so they will provide a base for the production of nanocomposites targeted at hydrogen storage applications.

12:20

Polish Research Projects

Micro- and nanocrystalline ceramic - metal functionally graded materials for transport applications

Katarzyna Z. Pietrzak

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MATRANS aims at development of novel metal-ceramic functionally graded materials (FGMs) for aerospace and automotive applications in: (i) exhaust and propulsion systems, (ii) power transmission systems, and (iii) braking systems, with the main objective to enhance the mechanical properties of these materials through spatial variations of material composition and microstructure. Specifically, MATRANS deals with two groups of bulk FGMs: (i) ceramics-copper/copper alloys, (ii) ceramics-intermetallics. These FGM systems have not yet been used in the transport sectors targeted. The MATRANS methodology is problem-oriented and comprehensive combining interrelated activities of material processing (core activity of the project), characterisation, modelling and demonstration. The processing will encompass

starting materials (e.g. nanopowders) and the resulting FGMs. Characterisation of the FGMs will include detailed description of microstructure, measurements of physical and mechanical properties and residual stresses. The modelling will be carried out at a design phase and for the material response to combined thermomechanical loading and extreme service conditions. Extensive use of multiscale approaches and numerical methods will be made. The project addresses the joint design of the FGM and the structural component it is intended for. Economical and ecological aspects of processing are included. Risks aspects of material non-performance will be tackled, too. MATRANS has mobilized a critical mass of interdisciplinary expertise and highly specialized equipment. The consortium includes leading groups from materials science, physics, chemistry, mechanical engineering and computer science. The industry and SME involvement in the project is substantial. As the exploitation measures, the industrial partners will define business plans and start pilot cases during the project, followed by upscaling activities after the project end.

LUNCH

Thursday afternoon, 19 September, 12:35

Polish Research Projects: Nanotechnology-based modern materials

Thursday afternoon, 19 September, 13:35 *Chair: Katarzyna Pietrzak*

13:35

Polish Research Projects

NANOMINING-Development of new nanocomposites using materials from mining industry.

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The Nanomining (www.nanomining.eu) is one of four projects selected in the first joint EU-Mexico call (announced in 2009) within the priority NMP.2010.1.2-4 pt. Adding Value to Mining at the Nanostructure level. The time of realization is 3 years (2010-2013).

Partners from Three European countries (Poland, Germany, France), Mexico and Canada are involved in the Project Consortium. Polish Partners have significant input to the Project and are nearly one third of the whole Consortium, which consists of the following organizations:

- 1. Instytut Obróbki Plastycznej (INOP), Polska, Coordinator
- 2. Centro de Investigación en Materiales Avanzados, S.C. (CIMAV), Mexico.
- 3. Universidad Autónoma de Ciudad Juárez (UACJ), Mexico,
- 4. Centro de Investigación en Química Aplicada (CIQA), Mexico,
- 5. Tamuse Systems (Tamuse), Mexico,
- 6. IfU Diagnostic Systems GmbH (IfU), Germany,
- 7. Instytut Metali Nieżelaznych (IMN), Poland,
- 8. PROJECTION PLASMA SYSTEME (2PS), France,
- 9. Tessonics Inc. (Tessonics), Canada,
- 10. Uniwersytet Medyczny im. Karola Marcinkowskiego w Poznaniu (UMP), Poland.

The Project deals with silver recovery, silver nanoparticles and silver based nanostructured composites. The environmental problems related to jarosite are significant all over the world. In many cases jarosite waste contains precious metals and especially silver in quantities that make their extraction attractive.

The Nanomining aim is to develop materials starting from waste jarosite through nanoparticle manufacture to end products addressing the whole value chain. The processes assessed are of interest for their applications in biomedicine and electrical component industry.

Project goals:

The main goal of the Project is to develop:

- 1. Clean and efficient procedure of silver recovery from waste: Combined Mechanical Activation Thermal Oxidation Processing of jarosite type residues to alleviate and accelerate the following precious metal leaching;
- 2. Combined nanotechnology of biological synthesis (using Mexican plants) of Ag nanoparticles and its deposition on implant surfaces by electrophoretic, spraying and other techniques;
- 3. Nanostructuring technology of Silver based nanocomposites manufacturing for electrical contact applications.
- 4. Pilot production and trials of developed Ag nanoparticle modified implants and Ag based nanostructured composites:
- Hydroxyapatite Ca10(PO4)6(OH)2) coated implants which are widely used in orthopaedic surgery because of their good biocompatibility related to the osteoconductive properties of calcium phosphate coating;
- Ag-SnO contacts for electrical systems; these composites combine high resistance to welding and to electric arch erosion of the refractory phases with the high electric and thermal conductivities

Methods

Three groups of technologies were examined:

- 1. Thermal oxidation and reduction processes of mechanically activated Ag and Zn containing waste products;
- 2. Biosynthesis of Ag nanoparticles and its'incorporation into Hydroxyapatite coatings;
- 3. Silver based nanostructured composite forming technologies of Ag-SnO and other contacts for electrical systems.

The scanning and transmission electron microscopy, precision chemical analysis, atomic force microscopy, spectroscopy and other methods were applied to study and products characterization. The in-vivo and in-vitro tests of AgNp modified implants were performed.

Ag NPs of size 2–18 nm have been synthesised, characterised and incorporated into Hydroxyapatite implant coatings. The Ag NPs deposition processes to coat surgical implants were developed using Ag nanopowders. The Ag-based nanocomposite fabrication methods were chosen and the laboratory samples of end-user requested parts were made. The results are opening the stage to novel Ag-based nanomaterials that could create new products as well as novel biosynthesis mechanisms that could be exploited in other ways.

The Nanomining project generated following results:

- 1) Mechanically Activated Thermal Oxidation (MATO) Technology and MATO machine for Jarosite Treatment:
- a) Fluidized Bed Reactor for Jarosite MATO processing
- b) Rotary Furnace for Jarosite MATO processing

There were two strategies examined in the Project: jarosite mechanical activation –Jet Mill + Microwave Rotary Furnace Oxidation and Jet Mill + Fluidized Bed Oxidation. A pilot fluidized-bed oxidation reactor was built to carry out the pilot experiments under the operating conditions of interest and to make possible observation and recording the process parameters during the runs.

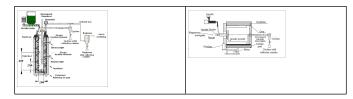
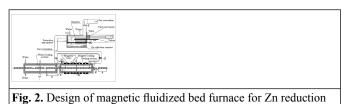


Fig.	1a.	Fluidized	bed	reactor	Fig.	1	b.	Schematic	diagram	of
scheme					the microwave heating system					

2) MAREC Technology Penoles Metallurgical Zn containing Slag Treatment

The Zn reduction MAREC (Mechanical Activation-Thermal Reduction) process was developed and elaborated for Zn containing waste. The new furnace design was developed by INOP for reduction of Zn and Pb, and collection of the product in protective atmosphere (Fig. 2). Application of magnetic field allows to create the high porous fluidized bed similar to those of fluidized bed reactor. MAREC technology was examined in the laboratory furnace using two types of waste: W1- after melting, W2 –after electrolytic processing. The reduction of Zn was made in reduction atmosphere (75% H2 + 25% N2)



3) Ag Nanoparticles bio-synthesis using dessert plant

The synthesis of silver nanoparticles of different chemical compositions, sizes and controlled monodispersity was developed and elaborated. Nanoparticle organic content control is a recent innovation of newly emerging synthesis methods. At the present, there is a growing need to develop environmentally friendly nanoparticle synthesis methods that do not use toxic compounds in the synthesis protocol. As a result, Mexican Partners in the area of nanoparticle synthesis have turned to biomimetic or biological systems for inspiration. The use of plant extract is one of the most active areas to develop synthetic methods to obtain non-toxic and biocompatible AgNp. Fig. 3 shows the developed reaction conversion as function of nopal extract used in the reaction. As can be seen, the rate of AgNO3 conversion to AgNp is directly proportional to the amount of nopal extract used in reaction.



Fig. 3. Effect of the nopal extract on the AgNps reaction conversion

This process is defined to be highly efficient and silver content is close to the maximal obtained by using other synthetic methods. One of the main concerns of these nanostructures is to identify all the possible organic components present in the AgNps, mainly if these nanomaterials are intended to be used for medical or environmental applications. To scale up the production of AgNPs to higher volumes, the new reactor with the downstream process was developed and tried to isolate and purify the AgNPs for biomedical applications.

4) Plasma Spraying of HAP coatings containing Ag micro- Nano-particles

The disc and rod samples for in-vitro and in-vivo tests, sedimentation of AgNp on their surface, adjustment of spraying and feeding parameters and deposition of HAP coatings on disk samples were made. Partners have fabricated cylindrical implants for rabbits and hip cup implants using the optimized plasma deposition technology

Implantation of cylindrical implants into rabbits legs, monitoring of rabbits state after surgical operation were made. The following works were made within this activity:

- Cytotoxicity analysis and SEM analysis of tissue cultures after incubation. Implants covered by HAP+NpAg3% and HAP+Ag3% (nanoparticles produced by biosynthesis) were incubated for 1, 2, 3 and 4 weeks in culture medium in incubator (37°C, 5% CO2).
- Antimicrobial activity was evaluated against the ATCC 35984 Staphylococcus epidermidis (LGC Standards) strain. Anti-bacterial activity tests were made and have shown significant antimicrobial properties of the implant coatings containing silver nanoparticles.
- In vivo study was conducted on the group of rabbits, males. The titanium implant prototypes (cylindrical) were inserted into the rabbit's legs. It was decided to use two methods developed in the frame of the Project for deposition of HA coating doped with Ag: (1) deposition of HA+Ag pre-mixed powder (10% of Ag); (2) deposition of initial HA coating with further sedimentation of AgNp

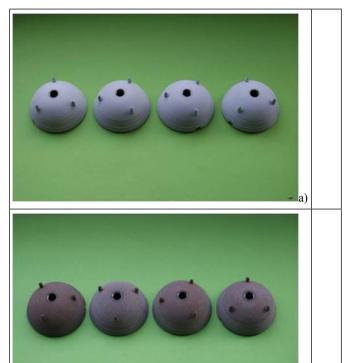


Fig. 4. Hip cups fabricated by 2PS for testing and characterization a) without NpAg b) with NpAg

5) Diagnostic system for plasma spraying on-line monitoring of HAP

The precision control of plasma deposition parameters was needed to achieve the desired structure of the Hydroxyapatite coating with AgNp. This task was fulfilled with the new diagnostic system developed by IfU. The main idea of the measurement method consists on the illumination of passing particles by a pulsed infrared laser. The reflected part of the pulsed laser radiation signalised the passage of the particle through the measurement window. The time of flight is an inverted signal to the particle velocity. During the time of passage the emitted thermal radiation of the particle is captured by two fast detectors with different spectral ranges. The ratio of the two emission signals results in a fast temperature measurement. Therefore a single particle statistics for velocity and temperature is possible. This information is used to benchmark the quality of the particle beam.

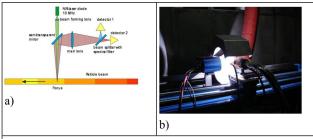


Fig. 5. Optical design (a)of introduced sensor and measurement arrangement (b)

IfU has approved the developed measurement method during a lot of particle measurements at real thermal processes. The measurement equipment was optimised to HAP-plasma spraying process.

6) Electrical contacts based on Ag-based nanocomposites

Based on results of study of nanocomposite technologies in the industrial environment the bimetallic electrical contact components were fabricated, characterized and tested. Bimetallic contacts were made of BT6/1,5x3/3,3 type (both AgSnBi and AgZn materials), 10BW4/1x2/2,5 (AgRe material) and 15BW6/1,52x3/2,63 (AgSnO2Bi2O3 material). The technologies for production of nano-composite contact materials of Ag-Re10, AgSn02Bi2O3 10 and Ag-Zn010 type were transferred into pilot scale. The pilot installation for upscaling of electrical contact production using the developed technology was completed with application of INM-INMET pilot plant equipment, and the first batch of electrical contacts was fabricated and sent for trials by the end-user. Contact rivets made of AgSnO2Bi2O310 nano-composite material were implemented by RELPOL company in solar relays which were then tested to determine their applicability in operation.





Fig. 6. Bimetallic contact rivet 15BW6/1,52x3/2,63 made of Ag-SnO2Bi2O3 (a) and solar relay with nanocomposite AgSnO2Bi2O3 contacts implemented (b)

The work has resulted in generating Polish patent for Contact material Ag-Re and method for producing contact material. The second patent application has been submitted for Method for production of Nanocomposite contact materials in silver matrix.

According to specification of the Project end-users, Silver nanoparticles (AgNp) and silver based nanostructured composites are believed to be used in a variety of other biomedical and industrial applications, such as an antimicrobial agents, lead-free solders, electric contact materials, gas-sensitive sensor, etc.

Within the Project there were created several publications. The following are the main ones:

- 1. J. Sulej-Chojnacka: Methods of recovering precious metals from jarosite type mining waste, Metal Forming, Periodical, v. 23, No.1, Poland, 2012, p. 45-56
- 2. S. Księżarek, M. Woch, D. Kołacz, M. Kamińska, P. Borkowski, E. Walczuk: Progress in fabrication technology of silver-based contact materials with particular account of the Ag-Re and Ag-SnO2Bi2O3

composites, Proceedings of ICEC-ICREPEC 2012, China, May 14, 2012

- 3. M. Ignatev, T. Rybak, G. Colonges, W. Scharff, S. Marke: Plasma sprayed hydroxyapatite coatings modified with silver nanoparticles, Acta Metallurgica Slovaca, Faculty of Metallurgy, Technical University of Košice, Slovaka, p. 20-29
- 4. S. Księżarek, M. Woch, T. Klir, D. Kołacz, M. Karpiński, K. Rudnicki: Environmentally friendly silver-based contact materials, ORES AND NON-FERROUS METALS, R57 (8, 2012), p.549-555
- 5. D. Garbiec, F. Heyduk: Sintering of titanium and hydroxyapatite by Spark Plasma Sintering, THE METALLURGIST Metallurgic news, Volume LXXIX, No. 8, 2012, p. 569-574

Acknowledgements:

- 1. The research leading to these results has received funding from the European Union Seventh Framework Programme FP7/2007-2013 under grant agreement No. NMP4-CP-2011-263942.
- 2. Research work financed from public funds for science in period 2011-2013, granted for realization of international co-financed project.

13:50 Polish Research Projects

NanoMat: The Application of Nanotechnology in Modern Materials

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The NanoMat Project is realized by Wroclaw Research Centre EIT+ in the frames of the European Funds for Regional Development (POIG, Sub-action 1.1.2). It involves interdisciplinary efforts aimed at manufacturing technologically advanced materials and developing nanotechnology for applications in electronics, photonics, power engineering, medicine, construction materials, polymer industry and other related fields. The goal of our research is to obtain materials featuring new or improved properties, develop technologies for their manufacturing and determine their potential in terms of applicability. Results obtained in the course of the project are patented and will be commercialized through technology licensing and spin-offs. Nanomat connects projects managers, experienced researchers, and specialists responsible for intellectual property protection and commercialization.

NanoMat program it is over 20 research projects within the following research areas:

- Nanomaterials for photonics and biomedical applications.
- · Nanomaterials for optoelectronics and sensing applications.
- · Lasers and optical fiber amplifiers.
- Microstructural polymer fiber optics.
- · Functional polymer materials.
- · SMART materials and nanocomposites.
- Electromagnetic radiation detectors and converters for digital medical diagnostics and document and banknote security systems.
- Intermetallic alloys absorbing hydrogen and permanent magnets based on lanthanides – development of magnetically hard nanocomposite materials based on domestic raw materials.
- Materials and technologies for advanced energy conservation and storage systems.
 - Development of modern biodetection and cellular bioimaging methods with the use of luminescent nanotags
 - Biosystems for detection of microbiological hazards
 - Technologies associated with laser micromachining and their application.

- Nanomaterials manufactured through sol-gel technology, intended for medical and sensory application.
- Polymer and ceramic nanocomposites for electrotechnical application.

Seventeen patent applications were applied, national and international, within two years. We are in fourth place in terms of the number of patent applications amongst R+D institutions in Poland.

14:05

Polish Research Projects

ERA-NET project: Manufacturing and characterization of Al-based composites with ultra-fine dispersion of Si3N4 particles

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The paper presents results of formation ultra-fine Al-based composite particles with Si₃N₄ and glassy carbon reinforcement as a result of high-energy ball milling. Two types of reinforcing particles were chosen in order to compare the chemically inert system (Al-Al₂O₃) and the reactive system of Al-Si₃N₄ with possibility for the replacement reaction. Milling was conducted in an inert argon atmosphere in silicon nitride or alumina lined milling jars with silicon nitride milling media. Various milling energy was applied. Oxygen content, particle size distribution, morphology (SEM, TEM) and phase composition were applied for characterization of the resultant powder. It has been found that chemical reaction in the Al-Si₃N₄ system significantly affects the milling performance in terms of ceramic particles distribution in Al matrix-particles. The resultant composite particles were then compacted and densified with or without external pressure and their mechanical properties, wear resistance were examined and related to the microstructure. It has been found that the chemical bond in-between ceramic particles and metal matrix combined with ultra-fine ceramic particles resulted in excellent wear resistance of composites.

14:20

Polish Research Projects

Nano-particle products from new mineral resources in Europe

Andrzej H. Chmielarz, Witold Kurylak

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e-mail: andrzejch@imn.gliwice.pl

Acronym: ProMine

Project no.: FP7-NMP-2008-LARGE-2, Grant Agreement Number

228559

Duration: May 1, 2009 - April 30, 2013

Consortium:

1 GEOLOGIAN TUTKIMUSKESKUS GTK, Finland

2 BOLIDEN MINERAL AB BOLIDEN, Sweden

3 KGHM CUPRUM SP Z OO CENTRUM BADAWCZO-ROZWO-JOWE CUPRUM, Poland

4 AGC MINAS DE PORTUGAL AGCMP, Portugal

5 HELLAS GOLD HG, Greece

6 PYHASALMI MINE OY, Finland

7 OY KESKUSLABORATORIO-CENTRALLABORATORIUM AB, Finland

8 CALDURAN KALKZANDSTEEN BV, Netherlands

9 WOLA CHEMISCH-TECHNISCHE ERZEUGNISSE GMBH, Germany

10 KGHM ECOREN S.A. ECOREN, Poland

11 SELOR EEIG SELOR, Netherlands

12 KEMAKTA KONSULT AB, Sweden

13 INTEGRATED RESOURCES MANAGEMENT (IRM) COMPANY LIMITED, Malta

14 G.E.O.S. FREIBERG INGENIEURGESELLSCHAFT MBH, Germany

15 INSTYTUT METALI NIEZELAZNYCH, Poland

16 BUREAU DE RECHERCHES GEOLOGIQUES ET MINIERES, France

17 INSTITUTO GEOLOGICO Y MINERO DE ESPANA, Spain

18 INSTITUTO GEOLOGIKON KAI METALLEYTIKON EREYNON, Greece

19 LABORATORIO NACIONAL DE ENERGIA E GEOLOGIA I.P., Portugal

20 VALTION TEKNILLINEN TUTKIMUSKESKUS, Finland

21 LULEA TEKNISKA UNIVERSITET, Sweden

22. TECHNISCHE UNIVERSITEIT EINDHOVEN, Netherlands

23 INSTITUT NATIONAL POLYTECHNIQUE DE LORRAINE, Greece

24 THE UNIVERSITY OF WARWICK, United Kingdom

25 BANGOR UNIVERSITY, United Kingdom

26 TECHNISCHE UNIVERSITAET BERGAKADEMIE FREIBERG TU, Germany

27 KWH-MIRKA, Finland

28 GRECIAN MAGNESITE, Greece

29 MILTON ROY MIXING, France

30 L'AIR LIQUIDE S.A., France

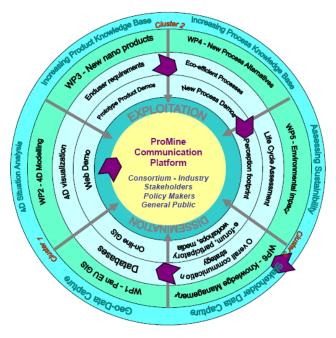
31 KIJLSTRA BETONMORTEL B.V., Netherlands

The objectives of the ProMine project responded to the European Comission's concerns over the annual 11 billion \in trade deficit in metal and mineral imports by focusing on two parts of the supply chain from extractive to end-user industries.

Upstream, a Pan-EU GIS based on mineral resource and advanced modelling system for the extractive industry was created for the first time ever, showing both known and predicted mineral occurrences across the EU, for both metallic and non-metallic minerals; detailed 3D and 4D computer models were produced for four metalliferous regions. Upstream work also included demonstrating the reliability of new technologies (including biotechnology) for an ecoefficient production of strategic metals, driven by the creation of added value on site and the identification of specific needs of potential end-users.

Downstream, a new strategy was developed for the European extractive industry which looks not only at increasing production but also at delivering high value, tailored nano-products which formed the new raw materials for the manufacturing industry. ProMine research focused on four new products (rhenium and rhenium alloy powders, nano-silica, iron oxyhydroxysulphate (schwertmannite) and new nano-particle

based coatings for printing paper), which will have a major impact on the economic viability of the extractive industry. They were tested at bench scale, and a number selected for development to pilot scale where larger samples were provided for characterisation and testing by end-user industries. It included production, testing and evaluation of these materials, with economic evaluation, life cycle cost analysis, and environmental sustainability.



The ProMine consortium of 31 partners had heavy major industrial involvement in both technical and management work. The project aimed on the one hand to develop to prototype production methods for new nano-particle based products. At the same time ProMine developed a methodology from resource assessment to new product development which can be applied beyond the framework of the project.

ProMine was treated by the European Commission as a flagship project of Raw Material Initiative, presenting good practices and contributing to the establishment of the European Innovation Partnership on Raw Materials.

14:35

Polish Research Projects

Development of characteristic of the process for production of environmentally friendly copper alloys of controlled and high level of mechanical properties

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The results of research project concerning the new environmentally friendly copper alloys are presented. The conducted studies consisted of selection and optimization of chemical composition, including potential for application of scrap and production waste, as well as development of parameters of melting and casting processes. The analysis covered the following parameters: melting point and casting point, method of casting, material and temperature of the mould. It was found that the optimum temperature range for melting of bronzes is 1040-1080°C, while for the brasses it is 970-1010°C. In evaluation of the

process of soldification of the newly-developed alloys the method of thermal and derivative analysis (TDA) was applied. Also influence of temperature gradient at the liquid alloy-casting mould border on changes in structure of examined alloys was studied. The alloys were cast into graphite and metal moulds preliminary heated up to the temperature of 200-300°C, and also tests with sand moulds were conducted. In the next stage influence of casting conditions, including the type and temperature of the mould and ingot diameter on macro- and microstructure was determined as well as on mechanical properties. The group of newly-developed environmentally friendly bronzes and brasses presents higher casting and mechanical properties than the widely used for components of fittings alloys standard alloys: CuSn5Zn5Pb5 and CuZn39Pb1Al, respectively. In the analysis of technological properties of the examined alloy group also their machinability was examined. The conducted pilot tests of production of the final components out of the examined group pf alloys confirm possibilities of their application in components of fittings. The newly-developed alloys show high capability to fill the mould which may result in production of the casts of complex shape without any surface defects.

COFFEE and POSTERS

Thursday afternoon, 19 September, 14:50

Polish Research Projects: Nanostructures, nanomaterials and nanosensors

Thursday afternoon, 19 September, 15:10 *Chair: Ewa Jędryka*

15:10

Polish Research Projects

FP7 NMP Ensamble: Plasmonic materials and metamaterials by bottom-up approach – manufacturing and properties

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Acronym: ENSEMBLE

<u>Title:</u> ENgineered SElf-organized Multi-component structures with novel controllaBLe Electromagnetic functionalities

<u>Project no.</u>: NMP4- SL – 2008-213669 <u>Duration</u>: 01.05.2008 - 30.04.2012 Website: https://www.ensemble-fp7.eu



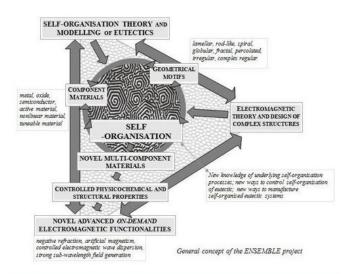
222

Project Partners:

- 1. Institute of Electronic Materials Technology (ITME), Warsaw, Poland
- 2. University of Zaragoza, Instituto de Ciencia de Materiales de Aragón (ICMA), Spain
- 3. Brunel University (BU), Kingston Lane, Uxbridge, United Kingdom
- 4. Consejo Superior de Investigaciones Científicas (CSIS), Instituto de Óptica, Madrid, Spain
- 5. Foundation for Research and Technology Hellas (FORTH), Heraklion, Greece
- 6. University of Southampton (UoS), Southampton, United Kingdom
- 7. Leibniz-Institut für Kristallzüchtung (IKZ), Berlin, Germany
- 8. Research Institute for Solid State Physics and Optics (RISSPO), Budapest, Hungary



General CONCEPT: Growth of eutectics is recognized as a paradigm for pattern-forming or 'self-organising systems'. Self-organised structures on size scales reaching down to the submicron and nanoscale regime emerge due to the interplay of chemical diffusion and capillarity. The fundamentally novel CONCEPT of the project was to utilize - for the first time - eutectic self-organisation for the preparation of multicomponent and multi-scale structures with controlled physicochemical and structural properties, with geometrical motifs capable of generating novel, predictable and controllable electromagnetic functionalities. This required a deeper understanding of factors influencing eutectic self-organisation mechanisms on the submicron/nanoscale.



The OBJECTIVE of this project was to design, manufacture, and characterize self-organised multi-component and multi-scale structures, which display:

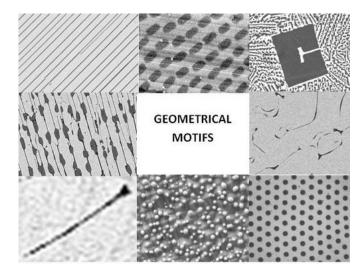
- **O1:** Controlled geometrical motifs, including rod-like, lamellar, globular and percolating patterns, which can provide the expected electromagnetic functionalities;
- **O2:** Controlled composition and physico-chemical structure, realized by combining materials with different refractive indices or different optical properties, which can provide the theoretically-predicted electromagnetic functionalities;
- **O3:** Controllable size of structuring ranging from micro- to nano scale following the theoretical predictions;
- **O4:** Novel predictable and controllable electromagnetic properties. At least one of the following properties can be obtained: controlled effective refractive index,; artificial magnetism; controlled dispersion; controlled sub-wavelength field generation and enhancement.
- **O5:** Metamaterialscapabilities which extend to applications such as: imaging, high compact optoelectronic devices, efficient photovoltaics and active devices.

RESULTS: Research and industrial development depends on novel materials, on their associated novel properties and enabled by them functionalities. That is why the overall objective of ENSEMBLE project was to design, manufacture, and characterize self-organised multicomponent and multi-scale structures, which display: controlled geometrical motifs, controlled composition and physico-chemical structure, controllable size of structuring ranging from micro- to nano, novel predictable and controllable electromagnetic properties and metamaterials capabilities.

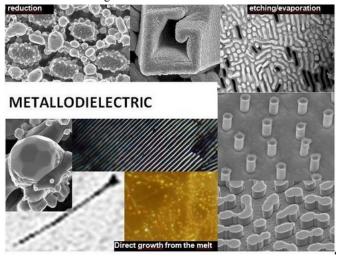
In order to achieve the main objective ENSEMBLE Project developed: (i) new modelling tools in order to gain insight into eutectic self-organization and the control mechanisms behind, (ii) new modelling tools enabling design of composite materials with special electromagnetic properties as well as comparing the experimental results for obtained materials with theory, (iii) novel technologies and novel materials which were characterized by often specially designed characterization set-ups (iv), as well as finally we demonstrated new materials exhibiting special electromagnetic properties (v).

To understandthe self-organization processes in eutectic growth we have developed multi-scale modeling tools. These tools all working in 3D are: (i) an atomistic phase-field theory [the phase-field crystal (PFC) approach] for unary and binary solidification; (ii) a quantitative phase-field working on the submicron to micron scale; (iii) a quantitative phase-field model coupled with hydrodynamics (with the Navier-Stokes equation) in three dimensions relying of spectral methods combined with operator splitting; and (iv) a quantitative multi-phase-field theory working above the micron scale.

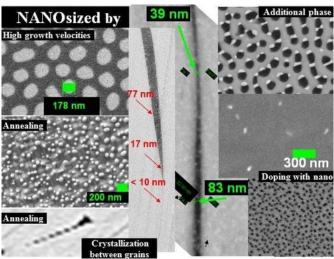
Programme Programme



To understand the electromagnetic properties of the manufactured materials and to be able to design materials with special electromagnetic properties on-demand we generated simple models and numerical methods that allowed electromagnetic modeling and simulations of eutectic self-organised micro and nanostructures, and enabled assistance in the design and characterization.



We have successfully grown self-organized materials as eutectic and eutectic-like materials with controlled geometrical motifs including rodlike, lamellar, or with such unusual geometries like eutectics with geometries resembling working metamaterial structures (split-ringresonator and fishet-like structures). We have demonstrated control of eutectic structures, from fibrous to lamellar, according to the growth rate used. We have successfully grown materials with controlled composition and physico-chemical structure, realized by combining materials with different refractive indices or different optical properties aiming at specific electromagnetic functionalities. We have successfully obtained metallodielectric materials incorporating metals at the melt extraction step or after subsequent treatment or by producing templates for subsequent metallization. We succeeded in pushing the refinement of the structure down to the nanoscale utilizing various methodologies such as increasing the solidification rate of the materials, adding additional phase, doping with nanoparticles. By combining the controllable composition, geometrical motifs and size of structuring we demonstrated materials with predictable and controllable electromagnetic properties.



We demonstrated the potential metamaterial-based applications of self-organized eutectics: (i) photoactive eutectics as photoanodes in photoelectrochemical cells (PECs) – experimental demonstration; (ii) bulk nanoplasmonic materials with localized surface plasmon resonance (LSPR) in visible and infrared, as well as enhanced photoluminescence due to LSPR in manufactured nanoplasmonic materials – experimental demonstration, (iv) subwavelength resolution imaging (superlensing) and subwavelength propagation and transmission in polaritonic eutectics, due to four different phenomena (all numerically demonstrated): hyperbolic dispersion relation, backward radiation in negative permittivity materials, subwavelength Mie resonances in permittivity near zero materials, surface phonon-polariton modes and gap-phonon polariton modes.

15:25

Polish Research Projects

Laser decoration an marking of ceramic and glass products

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Information about project:

Authors are proposing presentation of past and current results of three, precisely thematically related projects:

- 1. **Technology of laser decoration of ceramic products,** Technological Initiative I, National Centre for Research and Development, submitted in 2007, realised in 2008-2011 by the Institute of Ceramics and Building Materials in Warsaw in participation with team from Military University of Technology.
- 2. G-LAS **Innovative technology of colour, laser decoration of flat glass with ceramic agents,** Applied Research Programme, Competition 1, submitted in 2012, realised in 2012-2015 by the Institute of Ceramics and Building Materials in Warsaw (coordinator) and industrial partner Ceramika Paradyż Co., in participation with team from Military University of Technology.
- 3. Laser technology of marking and decoration by activation of ceramics and glass, Competition 40, National Research Centre, submitted in 2010, realised in 2011-2014 by the Institute of Ceramics and Building Materials in Warsaw in participation with team from Military University of Technology.

Projects aims and methodology

Works, conducted in the frames of basic and applied research, were and are connected with development of two methods: laser sintering of colour means on ceramic (1) and glass (2) products, as well as laser activation of ceramics and glass (3), where word "activation" means only change of substrate colour under illumination by laser beam, without any changes of its surface.

The main aim of all three projects is implementation of laser technique into the technology of colour decoration and marking of ceramic and glass products, with capabilities of reduction of production process costs and significant reformation of technological processes. Methodology of conducted research consists of investigations of fundamental physicochemical phenomena in laser-ceramic (glass) interaction and development of fundamental process elements: composition and application method of colour agents, laser system parameters, and software configuration according to the end user requirements.

Description of project results

The technologies being presented are researched and optimised using the market's cutting-edge diode-pumped laser systems, coupled with "galvo" type beam scanners with professional industrial software. Experimental analyses of phenomena and decoration results are conducted by means of X-ray spectroscopy methods (SEM, EDS, XRD) as well as digital optical 3D microscopy.

The most important results achieved are as follows:

1. Determination of the fundamental physicochemical phenomena, conclusive for the optimal selection of laser process parameters and decoration quality [1].

Selected key research results are presented in fig. 1. In the case of laser sintering, it has been confirmed (fig. 1a) that surface tension gradients in the glaze melt zone (the so-called Marangoni effect), as well as the plasma cloud recoil pressure caused by evaporation, dependent on thickness and composition of the colouring material as well as the temperature of the process, have the fundamental impact on the decoration quality (colour intensity, smoothness, resolution). In the case of laser activation (fig. 1b), colour change results from a temperature decomposition of particular components of the material, which are responsible for the substrate colour.

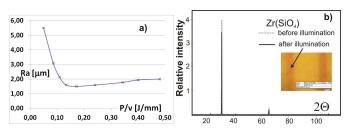


Fig. 1. a) Decoration roughness as a function of laser energy dose; b) X-ray diffraction spectrum of the glaze containing zirconium silicate before and after laser activation.

2. Development of a wide range of colouring agents fulfilling the quality criteria for laser processes [2]

A palette of colouring agents meant for laser sintering process has been developed in the Institute of Ceramics and Building Materials in Warsaw (fig. 2). The complete laser systems along with the processing software were created in cooperation with the MUT Institute of Optoelectronics. The computer control of the process allows for transfer of any suitable vector or raster images.



Fig. 2. a) Colour palette on tableware; b) Decorated cup (left) and stove tile (right).

Ceramika Tubądzin, the industrial partner interested in the implementation, and one of the top Polish producers of ceramic tiles and glass decoration plates, participates in the Applied Research Programme being currently realised. The laser sintering process is adapted to decorate glassware, characterised by other than ceramic material composition and lower melting temperature (fig. 3)



Fig. 3. Examples of laser decoration of glass plates from Ceramika Tubadzin.

Laser decoration technology for the ceramic products has been awarded with silver medals at the International Warsaw Invention Show IWIS 2012 and Moscow International Invention Salon "Archimedes 2013".

Selected papers resulting from the implementation of the projects:

- [1] D. Chmielewska, B. Synowiec, A. Olszyna, J. Marczak, A. Sarzyński, M. Strzelec, Physics Procedia, vol.5, p.1 (2010).
- [2] D. Chmielewska, R. Gebel, K. Szamałek, A. Olszyna, J. Marczak, A. Sarzyński, M. Strzelec, Proc. of SPIE Vol. 8703, 87030I (2013).

15:40

Polish Research Projects

Utilization of biomass for preparation of environment-friendly materials

Danuta Ciechańska, Ewa Wesołowska, Ewa Kopania, Janusz Kazimierczak, Justyna Wietecha, <u>Arkadiusz S. Bloda</u>

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1.

Programme Programme

1. BIOMASA, "Utilization of biomass for preparation of environment-friendly materials", The project is carried out within the Operational Program -Innovative Economy, 2007-2010, Priority 1: Research and development in modern technologies, Action 1.1.: Support to research in the creation of a knowledge–based economy, Sub-action 1.1.2.: Strategic programs of research and development. Time frame of the project 2009-2014.

Members of the Consortium:

- Lodz University of Technology, Łódź The leader of the project
- § Faculty of Biotechnology and Food Sciences
- § Faculty of Chemistry
- § Faculty of Material Technology and Textile Design
- Institute of Biopolymers and Chemical Fibres, Łódź
- Centre of Molecular and Macromolecular Studies, Polish Academy of Sciences, Łódź
- University of Agriculture, Kraków
- Central Mining Institute, Katowice

The main objective of the project is to develop a range of technologies to produce polymeric fibrous and composite materials based on raw materials deriving from processing of various types of plant biomass by biotechnological methods, utilizing specialized enzyme preparations or cultures of microorganisms. Technologies elaborated within the project will have an impact on the development of the knowledge-based economy. Individual objectives of the project include development of technologies or methods for:

- multi-enzyme preparation from *Aspergillus niger* for efficient degradation of plant biomass toward cellulose nanofibers,
- enzymatic saccharification of plant biomass for fermentation of L-lactic acid,
- immobilized lipase from *Mucor circinelloides* and *Mucor racemosus* for preparation of polyols from biomass derived from oilseeds,
- selection of lactic acid bacteria strains for stereospecific synthesis of L-lactic acid,
- producing cellulose nanofibers using enzyme preparations,
- producing polyols from oilseeds using lipase from $M.\ circinelloides$ and $M.\ racemosus$,
- fermentative production of L-lactic acid,
- the synthesis of L, L-lactide,
- polymerization of tactic polylactide (PLA) by L, L lactide for fiberand thermo-forming,
- preparation of aliphatic-aromatic copolyesters with the use of oil-seed-derived polyols,
- $\hbox{-} \ \ preparation of fibers from obtained aliphatic-aromatic copolyesters,}$
- production of non-wovens directly from melt of aliphatic-aromatic copolyesters,
- innovative nonwoven products using new generation of biodegradable polymers for hygiene, technical, agronomic and clothing applications,
- technology for preparation of elastomer composites and nanocomposites with specific time of use involving products from biomass processing,
- new materials based on polylactide and mineral fillers for use in packaging,

- thermoplastic composites reinforced with cellulose nanofibers,
- agrotextiles based on PE and PLA matrix reinforced with cellulose fibers,
- biological processing of post-consumer polymeric materials prepared within the project,
- practical management of degradation products of polymeric materials prepared within the project.

Synthetic results of

Workpackage 1.2. Pre-treatment of selected plant material (biomass) used to achieve the objectives of the project and Workpackage 3.1. Preparation of cellulose nanofibers from plant materials.

Plant biomass is widely considered as a vast and renewable source of various biopolymers such as cellulose, hemicelluloses, lignins and pectins. Among them, cellulose is the most known raw material and cellulose fibres are characterized by a wide range of useful properties including relatively high strength, high stiffness, low density as well as biodegradability. Within the scope of the project several types of plant biomass i.e. stems of hemp and flax (both fibre- and seed varieties) as well as waste flax textile fibres were selected as a starting material. The stems were subjected to a complex preliminary treatment in order to liberate cellulose from non-cellulose matrix composed of lignins, hemicelluloses and pectins. As a result of biomass purification process, cellulose pulp in the form of fibres with high alpha-cellulose content and only slight amount of lignin was obtained. Multi-enzyme preparations developed at Technical University of Lodz from Aspergillus niger fungal strain as well as commercial preparations of cellulases were applied in order to facilitate further fibrillation of cellulose fibres to micro- and nanofibres during high-shear mechanical treatment. After the process morphological changes in the cellulose structure were examined by scanning electron microscope and alpha-cellulose and lignin content (Kappa number) was determined. After subsequent enzymatic and mechanical treatment of cellulose fibres from flax and hemp straws appearance of cellulose nanofibers with diameters ranging from tens to several hundred nanometers was observed in the SEM images.

The fibrous materials and composites prepared within the project will be further utilized for obtaining new functional materials with various potential applications such as sanitary textiles, noise-absorbing materials, sweat-absorbing textile inserts, filtration materials, geo- and agrotextiles. So far, as many as 10 patent applications concerning the results of the project has been filed in Polish Patent Office.

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Polish Research Projects

BIOPHOSENS: Carbon nanomaterials based electrochemical biosensor for detection of highly toxic organophosphate pesticides

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Acronym: BIOPHOSENS

 $\underline{\text{Title:}}$ Disposable sensor for organophosphate detection in drinking water"

<u>Programme:</u> ERA-NET MATERA+, Call 2009, May 2011 - December 2013,

Consortium:

1. Inkoa Sistemas S.L. (Spain-Basque Country),

- 2. Fundacion CIDETEC (Spain-Basque Country),
- Institute of Physical Chemistry, Polish Academy of Sciences (Poland)

Project aim and framework

Organophosphate pesticides are very popular in agriculture and industry due to their insecticidal activity [1]. Pesticides are one of the most important pollutants, because of their accumulation in the environment and their high toxicity. They affect our health, attack neurological systems and even cause death. Therefore, the monitoring of the concentration of pesticides in drinking water is mandated by the EU.

This project is aimed to the construction of low-cost disposable biosensors for the monitoring of organophosphate pesticides in water, to substitute the time consuming chromatographic techniques used till date. Its main objectives are (i) to build suitable matrices to immobilise cholinesterase enzymes into screen printed electrodes (ii) to prepare low-cost polymeric electrodic supports for this bio-composite matrices (iii) to obtain long- life sensors where the enzyme is immobilised in the working electrode in optimum conditions, without leakage, and permitting a reproducible response to organophosphate pesticide detection.

Within the framework of this project our team constructed, characterised and tested new carbon nanomaterials based electrochemical biosensor for detection of highly toxic organophosphate pesticides. In construction we explored the fact that organophosphate pesticides toxicity results from the inhibition of the enzyme - acethylcholinesterase. This enzyme is capable to produce tiocholine from acetylthiocholine and the concentration of the product of this reaction marks enzyme activity. Thiocholine can be electrochemically oxidised and nanostructured electrodes provide favourable conditions for this reaction and good affinity to enzyme immobilization.

Organophosphate pesticide biosensor architecture based carbon nanomaterials (nanoparticles, nanotubes) were prepared by the layer-by-layer method. A conductive film is created by several alternate immersion of the substrate into suspensions of oppositely charged carbon nanoparticles and silicate submicroparticles or single walled carbon nanotubes. This substrate significantly decrease the overpotential of thiocholine oxidation. The effect of the amount of deposited material on the catalytic properties toward thiocholine oxidation is demonstrated. We have found the superiority of the sol-gel processed functionalised silicate matrix for the enzyme immobilisation. We will demonstrate the response of the sensor on different organophosphate pesticides like malathion, phosmet, dichlorovos; its stability; reproducibility; and detection limit.

Other partners are working on the transfer of this technology to screen printed electrodes.

Selected results were already presented during international conferences. At the moment patent application is prepared. After its submission preparation of 2 manuscripts to international electrochemical/analytical journals is planned. The obtained results will be a significant part of PhD Thesis.

16:10

Polish Research Projects

Impact of the structural funds (POIG) initiative on microsystem technology development in Poland (MINTE and MNS-DIAG projects)

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Enormous progress of silicon based micro- and nanotechnologies, aimed at development of increasingly complex integrated circuits according to a Moore's law, brought us numerous, extremely refined process technologies. This in turn, created vast opportunities for development of much broader range of highly innovative micro- and nanoscale devices utilizing a knowledge gathered in virtually all branches of physics, chemistry, biology, medicine, material sciences and other areas of science. Important features characterizing this new, "Morethan-Moore" domain are - much lower cost of the technology, as compared to advanced IC's manufacturing and much broader diversity of required expertize, knowledge, materials etc. Thus, MtM domain, in particular the area covering microsystem and sensor technologies became a field of choice for many academic research communities collecting pools of well-educated innovative minds with diversified expertise and knowledge. However, despite the lower technology cost, difficult access to Clean Room facilities and non-standard micro-processing constitute an important barrier hindering research for many academic teams. With this in mind two important projects financed through the Structural Funds mechanism, envisaged as a package underpinning implementation of an innovative technologies based on application of a microsystem technology have been undertaken in ITE, Warsaw, Poland. Both of them are shortly presented in this contribution

The goal of the project "Microsystems and Electronic Nanotechnologies for Innovative Economy - MINTE", POIG.02.01.00-14-081/09 (1.01.10 - 31.10.2013) is to modernize existing research infrastructure (RI) of the Silicon Microsystem and Nanostructure Technology Division of ITE. The Institute, as a leading research institute in the field of micro/nano-electronic technology in Poland is equipped with over 1000 sq.m. Clean Room facility in Piaseczno near Warsaw, where CMOS/MEMS R&D technology line is located. The processing line renders possible research in the area of micro/nano-electronics, photodetectors and silicon microsystems and next, development work up to a prototype stage with minimum feature size down to 3 um (proximity aligners) or 0,8 um (wafer stepper and direct writing laser tool). The weak side of the RI was the obsolete equipment with average age well over 20 years. In spite of that, the team achieved remarkable research results participating 25 European research projects, proving high research competences and reliable partnership, providing partners and customers with a cost effective service path, from a first concept up to a prototype stage, with flexibility allowing for undertaking nonstandard endeavors. Our main specialization lays in development of a specialized silicon detectors and resonance based MEMS sensors (physical and bio/chemical), including also MEMS characterization, modeling and simulation as well as device integration.

Within the frame of the MINTE project a new building has been built providing space for the measurement labs as well as for new employees. The main effort however has been put to improve reliability of the Clean Room facility (HVAC) systems and to replace the obsolete technological equipment with a modern one. The most complex upgrade has been made in the plasma processing bay where PECVD reactor (for SiO2 and Si3N4 deposition) as well as two new ICP reactors (for noble metal etching and for etching of dielectrics) and two RIE reactors (for standard CMOS etching needs) have been installed. Furthermore, three new diffusion furnaces (total 9 tubes) and 2 LPCVD reactors have been purchased and put into operation. The wafer processing line has been strengthened also by installing of 2 new sputter deposition reactors (for noble metals and for standard CMOS/MEMS applications). The lithography bay has been equipped with a new proximity aligner specialized for the MEMS technology, providing large depth of focus and double side alignment. Besides, an e-beam lithography tool has been installed over the existing SEM allowing for writing narrow lines below 100nm. A new plasma immersion ion im-

planter together with newly purchased RTA furnace will allow for elaboration of a highly innovative MEMS technologies. Finally, a set of advanced measurement equipment has been purchased with a CASCADE MEMS prober equipped with a Polytec Micro-Scanning Laser Vibrometer (MSV) being the center of the new MEMS characterization capabilities. All new equipment allows for processing wafers up to 150mm diameter. The whole investment process will be concluded in October 2013. It has to be emphasized however, that due to the limited financing (approx.. 11 mln Euro – which is modest as compared to needs of the largest Microelectronic RI in New Member States, after 30 years pause in investment), not all existing gaps have been filled and further investment effort will be needed. Nevertheless a considerable progress has been made bringing the research potential in the More-than-Moore domain in Poland to higher level.

The second project presented shortly here is "Microsystems for Chemistry and Biomedical Diagnostics - MNS DIAG" POIG.01.03.01-00-014/08-00 (2009-02-01 - 2013-03-31) envisaged to support access of academic research teams to advanced micro/nano-technology. The project has been aimed at investigation of ten new device concepts with a goal to develop five innovative demonstrators. To address the important grand societal challenge related to well-being and health in the ageing society, the project has been focused on application of microand nano-systems for chemical/bio-medical diagnostics. To satisfy the needs of the broad range of research undertaken within the project, 17 Polish research teams from universities and institutes have been gathered within the consortium coordinated by ITE, namely: ITE (2) teams), Institute of Biocybernetics and Biomedical Engineering PAS, Technical University of Lodz, Warsaw University of Technology (4 teams), Wroclaw University of Technology (2 teams), Poznan University of Life Sciences, Warsaw University of Life Sciences SGGW, Institute of Immunology and Experimental Therapy PAS, Jagiellonian University, AGH University of Science and Technology (2 teams), Wroclaw Medical University. The main outcome of the project includes:

- 1. CE Lab-on-a-chip system with amperometric detection for determination of psychotropic drugs (tricyclic antidepressant) in saliva samples.
- 2. Diagnostic instruments for analysis of nanoliter volumes of body secretion for fertility and pathological states monitoring.
- 3. A novel methodology and lab-on-a-chip diagnostic instruments for fast optical characterization & evaluation of single living porcine and bovine oocytes and embryos.
- 4. Bio- micro-reactors for cell culture.
- 5. Integrated MEMS detector for dynamic humidity measurement in medical applications allowing to reach up to 40 detections per second i.e. some 100 to 1000 times faster than conventional dew point hygrometers.
- 6. Microsystem device for the analysis of the exhaled air for medical applications, equipped with pre-concentrator.
- 7. Piezoelectric polymer sensor system for posture & gait diagnostic.
- 8. Nano-mechanical resonance sensors for chemical/bio-chemical analytics.

Achieved results has been published in 121 papers and 160 conference presentations. Besides, a broader monograph is being prepared and is expected to be published in the next year. The project brought important technological progress in application of the microsystem technology for analytical instrumentation, however it has to be emphasized that the development within the MNS DIAG project has been considerably hindered due to parallel investment process of the MINTE project, delayed due to financial reasons. Nevertheless, the main goal of the

project i.e. gathering broader research community around ITE providing access to the technology and elaboration of the demonstrators have been successfully achieved. Further work towards development of the prototypes and industrialization of the developed products will be carried out within next projects with strong industrial participation. Together, both presented projects in this package created a strong base for further progress of the More-than-Moore technologies in Poland.

Acknowledgments:

Authors would like to acknowledge a collaboration with leaders of the MNS-DIAG consortium. Also a financial support by The National Centre for Research and Development, Poland under POIG projects: MINTE; POIG.02.01.00-14-081/09 and MNS-DIAG POIG.01.03.01-00-014/08-00 is acknowledged.

Transfer to IWC PAN, Laboratory of Nanostructures CEPT, Prymasa Tysiąclecia 98

Thursday afternoon, 19 September, 16:30

Opening of NL 4 Laboratory of Nanostructures for Photonics and Nanomedicine CEPT: Visit to laboratory and round table discussion on perspecives of participation in research projects

Thursday afternoon, 19 September, 17:15

Discussion on integration of Regional Research and Horison 2020 programs

Thursday afternoon, 19 September, 17:30

Programme 69

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