Book of Abstracts

Title page

Book of Abstracts: Title page

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Table of Contents

Welcome

Symposia

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Programme

Wednesday, 15 October

REGISTRATION
Wednesday morning, 15 October, 10:30

POSTERS A & B

Poster's fixing to be displayed 3 days
Wednesday morning, 15 October, 11:00

Session 1

Introduction to nanotechnology
Wednesday morning, 15 October, 11:30
Chair: Anna Boczkowska

11:30	Keynote

Quo Vadis nanotechnology- for various stakeholders

Witold Łojkowski

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The meaning and perspectives of nanotechnology for science, industry, regulatory bodies and society are disscussed. For each of these groups nanotechnology has a different meaning and different future.

For science - it is everyday life. The question is where next discoveries will appear soon.

For industry - it is a marketing question and price question. Is the label nano good for an increase of sales? Will aditional cost of nanotechnology be payable?

For regulatory bodies - a challenge. Strong need for nano proeducts, but they are difficult to measure and characterise.

11:45	Keynote

Nanomaterials for better life

Rodrigo F. Martins

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Session 2a

Nanometrology
Wednesday afternoon, 15 October, 12:00
Chair: Anna Boczkowska

12:00	Invited Oral

Nanoparticle size and zeta potential analysers Zetasizer and NanoSight from Malvern Instruments.

Marek Sterczewski, Adam Próchniak

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Due to the growing interest in nanomaterials, measuring nano-sized species is becoming the parameter of great importance. To meet these expectations Malvern Instruments company offers Dynamic Light Scattering equipment - Zetasizer Nano ZS. It is high-performance system combining Dynamic Light Scattering for nanoparticle sizing, Electroforetic Light Scattering for zeta potential, and Static Light Scattering for molecular weight measurements.

To overcome some DLS limitations Malvern offers also NanoSight family nanoparticle size analysers. These instruments utilize a technique called Nanoparticle Tracking Analysis - NTA, to measure particle size.

In the presentation the advantages of both techniques, and applications examples will be discussed.

12:15	Invited Oral

Laboratoria nano i biotechnologii

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Firma IGHT s.c. została założona w 2007 roku przez młodych pasjonatów nanotechnologii, jako przedstawicielstwo firmy NT-MDT (www.ntmdt.com) na terenie Polski. W kolejnych latach działalność rozwinęła się na inne techniki badawcze z zakresu nanotechnologii. Staramy się zapewniać kompleksową obsługę klienta, nie tylko w zakresie sprzedaży urządzeń, ale również zapewnienia akcesoriów, materiałów eksploatacyjnych oraz wsparcia naukowego i serwisowego. W 2013 roku otworzyliśmy własne laboratorium pokazowe, które sukcesywnie wyposażamy w najnowsze z oferowanych przez nas rozwiązań, a także stworzyliśmy markę Labnatek, dzięki której możemy scalić większą ilość produktów.

Nasza oferta oprócz urządzeń laboratoryjnych i badawczych zawiera także akcesoria, materiały eksploatacyjne oraz systemy kontroli warunków otoczenia. Najważniejsze marki w naszej ofercie to:

Accurion, CTS, LabBubble - podstawowe wsparcie dla wszelkich badań nanotechnologicznych, stoły antywibracyjne, izolacja drgań, komory przygotowawcze dla biologii, komory czyste i rękawicowe.

Dino-Lite - proste cyfrowe mikroskopy optyczne dostępne z różnymi opcjami oświetlenia, montażu oraz zestawem akcesoriów.

NT-MDT - Lider na europejskim rynku mikroskopii sił atomowych i integracji ze spektrometrią Ramana. Najbardziej rozbudowana charakteryzacja właściwości mechanicznych, elektrycznych, magnetycznych i optycznych w skali nano. Unikalna technologia NT-MDT pozwala na obrazowanie ramanowskie z rozdzielczościami poniżej 20 nm.

Hysitron - Lider na światowym rynku badań mechanicznych i nanoindentacji. Najdokładniejsze indentery świata, które dodatkowo można połączyć in-situ z technikami SEM i TEM.

Izon Science - liczniki cząstek bazujące na nowatorskiej opatentowanej technologii TRPS. Pozwalają na określenie średnicy cząstek w cieczy, cząstka po cząstce, przy cenie będącej ułamkiem kosztu technik DLS czy NTA.

Dla wszystkich technik zapewniamy pełen zakres materiałów eksploatacyjnych oraz najwyższej klasy akcesoria i plastiki laboratoryjne amerykańskiej firmy SSI.

IGHT company started in 2007 as a local representative of NT-MDT, providing research level SPMs for Polish market. After few years we expanded our offer with other products and in 2013 created a brand LABNATEK which groups all products that we deliver in Poland.

12:30 Invited Oral

Advanced materials characterization by thermal analysis techniques

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Do wpisania

12:45 Invited Oral

Advanced characterisation of nanomaterials according to ISO 17025 Norm

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DISCUSSION

Discussion and questions to all speakers
Wednesday afternoon, 15 October, 13:00
Chair: Anna Boczkowska

LUNCH & POSTERS

Wednesday afternoon, 15 October, 13:10

Session 2b

Nano-metrology
Wednesday afternoon, 15 October, 14:00
Chair: Giancarlo Cravotto

14:00 Invited Oral

Characterization of nanomaterials using molecular spectroscopy methods

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Molecular spectroscopy is a non-destructive research tool that gives information on the molecular structure and its changes for large amount materials and compounds. It is also a suitable instrument for the study of nanomaterials with any chemical composition. The most useful methods include Raman spectroscopy (RS) and Fourier Transform Infrared Spectroscopy (FTIR), due to high sensitivity to the physical and chemical properties of investigated materials and vulnerability on the influence of external conditions . In recent years, a lot of work concerns the study of carbon nanomaterials, due to easily distinguish between allotropic forms of carbon (fullerene, graphene, carbon nanotubes, etc.). Great potential for analysis of the surface homogeneity of molecular composition gives the Raman imaging (Raman mapping) method. The only limitation of this method is the spatial resolution associated with the penetration depth and the diameter of the used laser beam.

The aim of the presentation will be to show the possibilities of measurement techniques in study the structure of nanomaterials, particularly carbon nanostructures (fullerenes, nanotubes) and carbon-metal nanocomposites (C-Pd, C-Ni), including Raman mapping technique.

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[2] R. Belka M. Suchańska, " Properties of the carbon-palladium nanocomposites studied by Raman spectroscopy method", Proc. of SPIE 8903 (2013).

[3] Suchańska M. [rozdział:] „Spektroskopia ramanowska nanostruktur Ni-C" [w:] Nanomateriały węglowe. Układ węgiel - nikiel, Oficyna Wydawnicza Politechniki Warszawskiej, Warszawa 2012, 100-124

[4] Belka R., Suchańska, M., Czerwosz E., Chiasera A., Ferrari M. "The Optical Study of Nanoporous C-Pd Thin Films" Proc. of SPIE vol. 8070 (2011).

Acknowledgments

This research was co-founded by the European Regional Development Fund "Perspektywy RSI Świętokrzyskie – IV etap" WND – POKL.08.02.02 – 26 – 001/12 Human Capital Operational Programme, Priority VIII, 8.2.Knowledge transfer, 8.2.2 Regional Innovation Strategy

14:15 Invited Oral

SAXS/WAXS/USAXS polymer investigation of orientation induced by deformation in semi-crystalline polymers

Nicoleta Galatanu[1], Sandra Desvergne[1], Pierre Panine[1], Manuel Fernandez Martinez[1], Frederic Bossan[1], Yongfeng Men[2], Yaotao Wang[2]

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During the industrial transformation, most polymers are involved in specific processes resulting in the macromolecular chain orientation. Such orientation is appearing from the local phase to the mesophase and sometimes even up to the macroscopic phase.

Small Angle X-Ray Scattering (SAXS) is a technique well suited for investigating nano-materials and nano-structures of polymers. Information is collected on sample structure parameters such as particle shape or size, size distribution, orientation, surface to volume ratio… in the range from 1 nm to beyond 100 nm. Moreover, orientational functions can be derived from 2 dimensional x-ray patterns. Length-scales down to 0.1 nm can also be investigated in combination with Wide Angle X-ray Scattering (WAXS). In the case of samples with internal structures larger than 200 nm, USAXS (Ultra Small Angle X-Ray scattering) experimental conditions are required. USAXS allows to access heterogeneities about some hundreds of nanometers while providing the opportunity to investigate the large-scale structure evolution during the deformation of polymeric materials [1]. The progress in the performances of x-ray components and subsequent assembly offers such characterization methods in the laboratory. Hence, investigation of an injected semicrystalline polymer has been performed, emphasizing the nanostructure orientation and processing relationships. Further, recent results obtained on in-situ stretched Polybutene-1 (PB-1) [Figure 1] illustrate the capability to perform lab measurement equivalent to Synchrotron USAXS [2] highlighting microscopic structural evolution and macroscopic strain-whitening phenomenon correlation.

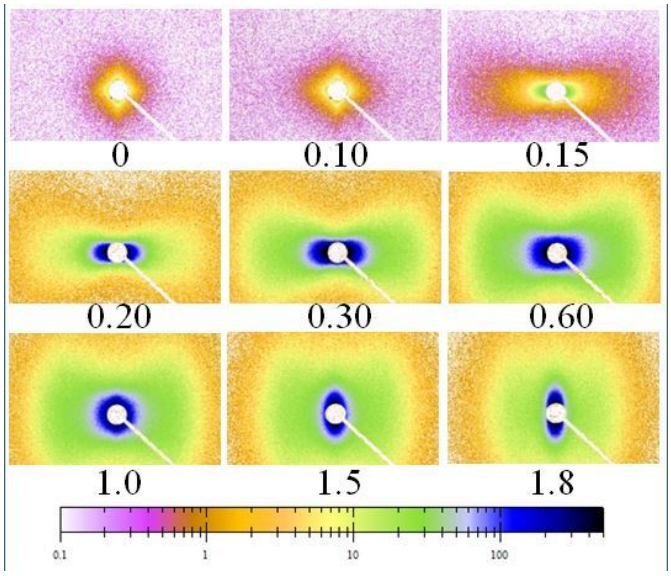

Figure 1: 2D-USAXS patterns of PB-1 crystallized at 60°C stretched at 30°C as a function of engineering strain - 50s exposure time. Stretching direction horizontal. Data courtesy of Pr. Men and Y. Wang.

References

[1] - Y. Men et al, Macromolecules 37, 9481 (2004)

[2] - Y. Men et al, under submission (2013)

14:30 Oral

Analysis of density waves in CdSe, SiC, and diamond nanocrystals by application of NanoPDF software package to experimental Pair Distribution Functions.

Svitlana Stelmakh[1], Stanisław Gierlotka[1], Kazimierz Skrobas[1], Witold Palosz[2], Bogdan F. Palosz[1]

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A new approach to the determination of the true atomic structure of nanocrystals is presented. It is based on the analysis of the Pair Distribution Function. A model of a nanocrystal with a modulation of the atomic density that has a form of a wave going from the center of a nanocrystal towards it's surface is examined. A dedicated software package NanoPDF [1] which serves for modeling and PDF data analysis is presented.

There has been a common agreement that the surface of the nanocrystals is different, in terms of the atomic arrangement, from the bulk, what follows from basic physical laws referring to crystal symmetry, surface energy etc. A diffraction pattern essentially contains all the information on the atomic arrangements in an investigated sample. There exist sophisticated methods to investigate fine details of atomic arrangements in quasi-infinite crystals, and there exist methods to determine structure of materials without any long-range order. True nanocrystals fall in between the two categories: their size matters when one plans the experiment and analyses the data

and their atomic structure is only periodic-like.
In the past we have predicted and showed experimentally that in diffraction patterns of nanocrystals the position of every Bragg peak points to a slightly different lattice parameter. Examination of this behavior led us to elaboration of a core-shell model of various nanomaterials [2]. However, when analyzing only average lattice parameter calculated from Bragg reflections one rejects some important information contained in a diffraction pattern. The method that uses all available data is the Total Scattering Analysis. Reconstruction of the direct space is performed through calculation of the Atomic Pair Distribution Function (PDF) by Fourier transformation of the diffraction data. Such a transform, usually denoted G(r), contains complete information on inter-atomic distances present in the investigated material. In case of a macroscopic crystal peak positions in G(r) are uniquely defined by the sample's lattice parameter. In case of a nanocrystal which lacks ideal periodicity of the crystal lattice the peaks positions in the G(r) function are displaced with respect to the positions they would have for a perfectly periodic lattice [3].
To evaluate deviation of the true structure of nanocrystals from a parent crystal lattice the experimental curve is scanned by fitting inside limited sections of G(r) for given r-intervals. We have derived experimental function δ(r) describing deviations of individual interatomic distances from those of a perfect crystal lattice. In order to propose an atomistic model for a given nanocrystalline sample we examined theoretical models of a nanocrystal which are composed of a core surrounded by several shells where inter-atomic distances change from shell to shell in a quasi periodic manner. While the analysis of the experimental data can be automated, the step of finding the best matching model is a sort of a trial-and-error procedure.
Both the analysis of the experimental G(r) and the calculation of the models is handled by the dedicated NanoPDF software [1].
With use of the above procedure models of nanocrystalline diamond, SiC and CdSe were proposed.
This work was supported by NCN through grant No. 2011/03/B/ST5/03256 and 2011/01/B/ST3/02292.
[1] K. Skrobas et al.,NanoPDF Software Package; http://www.unipress.waw.pl/soft/crystallography/nano.pdf.
[2] B. Palosz et al., Z. Kristallographie 225 (2010) 588.
[3] B. Palosz, Denver X-ray Conf. Proc., Advances in X-ray Analysis, Volume 55 (2011).

Session 3

Synthesis of nano-materials
Wednesday afternoon, 15 October, 14:40
Chair: Helmut Schmid

14:40 Keynote

Kinetics and thermodynamics in nano metal oxides formed by sol–gel technique

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Sol–gel technologies have been extensively used for the preparation of nanocrystalline ceramic oxides, since the pioneering works of Yoldas, Brinker and others in the nineteen-seventies. Due to inherent simplicity and low costliness of these synthetic processes, they are prone to scale-up to industrial amounts. Since sol–gel products are usually prepared at RT, the final products are tailored by treatment of the xerogels. It was found that the phase amounts, the crystallinity and the grain size are determined by the xerogel composition and the conditions of thermal annealing (time and temperature). It is well known that the treated sol–gel products are in many cases different from those appearing in the equilibrium phase diagrams for bulk materials. It is quite common to obtain unusual amorphous, nanocrystalline phases and solid solutions which are not seen in the phase diagrams. In the past the surface energy was used to model the kinetics of phase transformations especially during the nucleation stage. Embryos with surface energies higher than the bulk driving force were regarded as unstable. The kinetics of the transition from the unstable embryos to the stable nuclei is well documented. In the sol–gel products we deal with a variety of structures stabilized by lowering the surface energy. It is the contention of our group and others, that the grain size also acts as a thermodynamic variable in addition to concentration, temperature and pressure which are the conventional ones, when determining the stability of nanocrystalline phases, considered by others as metastable. Therefore, it is interesting to study the correlation between kinetics of grain growth and phase transformation. In this presentation will be shown an *in situ* observation of grain growth and phase transformations of magnesium titanates formed by the sol–gel technique. These ceramic materials have dielectric constants which are unchanged within a wide range of temperatures and frequencies.

15:00 Oral

Microwave Solvothermal Synthesis of High Quality Nanoparticles

Jacek Wojnarowicz

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Expertise of Laboratory of Nanostructures 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 ultra-pure 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 ZrO_2 nanopowders.

We have constructed innovative reactors MSS-1 and MSS-2 (Fig. 1) 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 cm^3
- Microwave Heating at 2450 Mhz
- Microwave power up to 3 kW
- Chamber material PTFE and ceramic Al_2O_3

15:10 Invited Oral

Nanocrystalline structures in steels produced by heat treatment processes

Krzysztof Wasiak, Kamil Wasiluk, Adam Gołaszewski, Szymon Marciniak, Julita Dworecka, Karolina Dudzińska, Piotr Pawluk, Emilia Skołek, Wiesław A. Świątnicki

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The heat treatment processes, which lead to the formation of homogeneous nanostructure in low and medium-alloy steels were designed within the Project "**Production of nanocrystalline steels using phase transformations" which is implemented at the** Warsaw University of Technology, Faculty of Materials Science and Engineering. This technology is based on a carefully designed heat treatment which allows to produce nanostructure in the finished elements made of steels that have specially selected chemical composition. The main step of that treatment is isothermal annealing within the range of temperatures in which bainite is formed. Isothermal hold temperatures have been selected to be above Ms temperature to avoid martensite transformation which causes brittleness. Heat-treatment parameters were selected on the basis of dilatometric tests and transmission electron microscopy. The microstructures obtained as a result of austempering consisted of a nano-size carbide-free bainite and retained austenite which provide high ductility. High strength, ductility and good service parameters of nano-size carbide-free bainitic steels make themcompetitive in comparison with highly alloyed martensitic steels. Within the Project another technology of heat treatment which allows to obtain nano-sized carbide-free bainite or low-temperature bainite in carburized surface layer was also developed. Such microstructure in carburized surface layers have better wear resistance in comparison to microstructure obtained by hardening and low-temperature tempering. Moreover, isothermal holding within the range of temperatures in which bainite is formed results in reduction of residual stress level and distortion in comparison to quenching and tempering. Because of long-time isothermal holding which sometimes last several days, the endeavor to adapt austempering to industrial production was carried out. As a result a new technology of heat treatment which allows obtain in short time microstructure composed of nano-size bainite, nano-size martensite and retained austenite was developed. This technology allows to obtain optimum mechanical properties such as high tensile strength while maintaining high ductility. Our research shows that nanocrystalline steel besides of high mechanical parameters is characterized by great performance and technological parameters.

15:25 Invited Oral

Microwave sintering of nanosctructured metallic powders

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Electromagnetic field assisted sintering techniques (FAST) demonstrated the possibility to minimize the grain growth, obtaining, at the same time, final densities near to the theoretical ones. Besides the high speed of the process, this can be ascribed also to a series of phenomena, like electromigration, the local increase of the diffusion coefficient or the action of ponderomotive forces. Very soon, among these techniques, microwave (MW) assisted sintering reached a strong experimental evidence of accelerated densification, lower porosity and achievement of peculiar microstructuresin the final products, including metal-based ones [1, 2].

In this work, nanostructured stainless steel powders, obtained by high energy milling have been uniaxially pressed in order to obtain 20 mm diameter cylindrical compacts. Microwave sintering of the green compacts has been performed in a single mode microwave applicator, at 2.45 GHz.

Maximum sintering temperature of each sample, monitored using a sapphire optical fibre, was varied in the 900-1150 °C range. Microwave forward power was varied accordingly, in order to maintain sintering times in the 120- 500 seconds range.

Rapid microwave sintering helps maintaining the nanostructure, despite a non homogenous densification due to the uneven electromagnetic field distribution in the single-mode applicator. A more homogenous multi-mode applicator, as well as the samples movement during sintering helped increasing the homogeneity and simultaneously sintering multiple green parts. Numerical simulation of the electromagnetic field distribution inside the multi-mode applicator allowed to select the best experimental conditions in terms of samples homogeneity and energy efficiency.

References

[1] R. Roy, D. Agrawal, J. Cheng, S. Gedevanishvili, Full sintering of powdered metal bodies in a microwave field, *Nature* 399, 1999, 668-670.

[2] C. Leonelli, P. Veronesi, L. Denti, A. Gatto, L. Iuliano, Microwave assisted sintering of green metal parts, J. Mater. Proc. Technol. 205, 2008, 489-496.

Discussion

Discussion and questions to all speakers
Wednesday afternoon, 15 October, 15:40
Chair: Helmut Schmid

Coffee & Posters

Wednesday afternoon, 15 October, 15:50

Session 4

Advanced nanostructures
Wednesday afternoon, 15 October, 16:20
Chair: Giancarlo Cravotto

16:20 Oral

Tailoring polymer surface properties by extreme ultraviolet (EUV) radiation

Inam Ul Ahad[1,2], Henryk Fiedorowicz[1], Andrzej Bartnik [1], Dermot Brabazon[2]

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More than 100K deaths and 1.7 Million infections per year are reported due to the low biocompatibility of biomaterials only in the USA. Surface modification techniques including chemical and plasma treatments, ion implantation and ultraviolet irradiation are used to increase the degree of biocompatibility, however associated with undesirable effects. Extreme ultraviolet radiation extends energies from 10 eV to 124 eV (corresponding to wavelengths of 124 nm to 10 nm respectively). The wavelengths in this range make possible to write nano- and micro-patterns. A single EUV photon is able to break several bonds on organic polymer surfaces as it has energy way above the binding energies of C–C (3.6 eV), C–N (3.2 eV), and C–O (3.7 eV) and C-F (5.0 eV) bonds. In this presentation, surface modification of various polymers by the use of a laser-plasma EUV source based on a double-stream gas-puff target, irradiated with 3 ns/0.8J Nd:YAG laser pulse at 10Hz is demonstrated. The physical and chemical properties of EUV modified surfaces are characterized by SEM, AFM, XPS, and water contact angle (WCA) measurement. Pronounced wall type nano- and micro-structures, nitrogen deposition (1.1 atomic %), increased hydrophobicity (+20 degree change in WCA), and increased surface roughness up to many folds observed in the EUV modified surfaces. In-vitro cell culture studies, using L929 mouse fibroblasts demonstrated strong cell adhesion and increased cell viability in EUV modified polymer surfaces. EUV surface modification can be successfully employed for biocompatibility control in polymeric biomaterials with no undesirable effects known to-date.

Acknowledgements

The authors acknowledge financial support from the EU FP7 Erasmus Mundus Joint Doctorate Program EXTATIC under framework partnership agreement FPA-2012-0033. With support from the 7th Framework Programme's Laserlab Europe project (No. 284464).

16:40 Oral

Thermoplastic nanocomposites with enhanced electrical conductivity

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During recent years, there has been a growing demand for composite materials with integrated multifunctional capabilities. The promising substitutes for traditional composites are nanocomposites consist of polymers and filler with nanosize. One of the most known nanofillers are carbon nanotubes. These hollow, low dense three-dimensional structures demonstrate extraordinary mechanical and electrical properties capable to reach the functionality of composites. However, their great industrial potential cannot be fully realize due to current manufacturing challenges. It happens because even the small addition of carbon nanotubes into polymer matrix alters the properties of polymer significantly. On the one side, the improvement of mechanical and electrical behavior of polymer is observed. However, on the other side, the jump of viscosity and decrease in macromolecules flowability lead to many hindrances in their processing. Hence, a lot of efforts is put to overcome these obstacles to find the possible way of nanocomposite's processing on industrial scale.

The conducted research are focused on the three levels. The first one is the fabrication of thermoplastic composites with carbon nanotubes from polyamides and thermoplastic elastomers using laboratory mini extruder. Polymers used in the study are commercially available and show the wide range of melt-flow index. Samples were produced in the form of strips and fibers. The process conditions were found and then optimized to make the manufacturing route continuous. Afterwards, the factors affecting electrical conductivity of nanocomposites are deeply investigated. First and foremost, the significant increase in electrical conductivity. The higher amount of carbon nanotubes the higher conductivity is achieved. However, this is necessary to achieve the percolation threshold which is different for various polymer. It was found, that the differences in carbon nanotubes arrangement in strips and fibers lead to completely dissimilarities in the conductivity values. Moreover, the carbon nanotubes available on the market did not give the same results what is dependent on their dimensions and purity.

The second area of the study is related to development of processing route of nanocomposites towards products which could be commonly used in different industrial sectors. Aerospace and aviation fields are mainly under our interests because the novel lightweight multifunctional nanocomposites are still desired. They potential usage include lighting strike protection and improvement of the mechanical properties. The most needed form of the final product is non-woven fabric called veil. It is understood as a thin textile material with random distribution of fibers characterized by low aerial weight (GSM factor). The method of fabrication thin veils with carbon nanotubes on the laboratory scale was the main chal-

lenge within this area of interest. The production starts with nanocomposites fibers extrusion with carbon nanotubes using mini-extruder. Then fibers with different carbon nanotubes content and with diameters from 80μm to 300μm were fabricated. They were further cut and press together into the veil without using of any binders or additional chemical compounds. The temperature and time of pressing were adjusted resulting in good quality veils. Non-woven fabrics doped with carbon nanotubes were used in the laminate infusion process as an interlayer. The improvement of electrical and mechanical performance of laminate was observed.

The third area of study is connected to development of veils manufacturing process on the half-industrial scale. For this purpose, melt-blow technique was chosen as a well known in the polymers processing. This one step approach possesses higher capacity and is much faster than others. Thermoplastic polymer with carbon nanotubes was loaded into the hopper and melted. Then, owing to high speed hot air the material is going through the head equipped with small nozzles on the moving drum. As a result, veils with carbon nanotubes were obtained. Veils with 5wt.% of nanofiller with minimum areal weight 7g/m^2 were successfully fabricated. They were used as a interlayers in the laminate infusion process.

The main motivation of our work is the lack of non-woven veils with carbon nanotubes on the market. Moreover, their huge potential and demand from different industrial sectors.

The part of the study was supported by ESA as a contract no. 4000107904/13/NL/KML

16:50 Oral

Polyester electro conductive Fibers with carbon nanotubes and graphene sheets

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In this study the effect of different shape carbon nanofillers by means of carbon nanotubes (1D) and graphene sheets (2D) on morphology, mechanical and electrical properties of poly(etylene terephthalate) based composite fibers was investigated. The amount of carbon nanotubes that were utilized to prepare the conductive fibers was 5wt.%, whereas the amount of graphene sheets was only 2.5wt.%. The masterbatches were prepared by Institute of Materials Science and Engineering. The polyester electro conductive fibers with carbon nanofillers and preliminary characteristic of synthesised nanocomposites were prepared by Torlen Sp. z o.o. in the course of a joint research project MNT ERA NET. The results show that both nanofillers can be incorporated into PET matrix with good dispersion. It was seen that PET nanocomposites demonstrated better mechanical performance especially seen in the tensile modulus.

Fig. 1. Preparation process of electro conductive fibers with graphene sheets and carbon nanotubes prepared by Torlen Sp. z o.o. (SME, consortium partner in MNT ERA NET APGRAPHEL)

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17:00 Oral

$LiMn_2O_4$/graphene oxide as a cathode material for lithium ion battery

Monika Michalska[1], Dominika Ziółkowska[2], Jacek Jasiński[3], Ludwika Lipińska[1]

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Lithium manganese oxide (LMO, $LiMn_2O_4$) of spinel structure is very promising as a cathode material for secondary lithium ion batteries. This compound has several advantages like: low cost and easy preparation, non-toxicity, high discharge potential (4V vs. lithium metal), a satisfactory practical capacity (120 mAh/g), high-energy density and low self-discharge. One of the drawbacks of lithium manganese oxide is its modest electronic conductivity. There are several ways of enhance it: i) introducing metal particles onto $LiMn_2O_4$ internal surfaces, ii) coating the spinel particles by conducting polymers, iii) the most popular - using carbon either as thin layers or mixing as-synthesized $LiMn_2O_4$ with carbon species.

In our studies we used graphene oxide (GO) as a carbon species. The pristine nanocrystalline $LiMn_2O_4$ powder was synthesized by modified sol-gel method [1-3]. Graphene oxide was prepared by a modified Hummers method [4,5]. The wet low temperature chemical method was used to modify the LMO grains using graphene oxide.

The structure and morphology of the synthesised powders were characterized by: X-ray powder diffraction (XRD), Raman spectroscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and X-ray photoelectron spectroscopy (XPS). The electrochemical charge-discharge tests were performed in three electrode cells with $LiMn_2O_4$/n-GO as working and lithium as a reference and counter electrode. A lithium hexafluorophosphate $LiPF_6$ in a mixture of ethylene and dimethyl carbonates (1:1) was used as

an electrolyte. The working and counter electrode was detached by Celgard 2400 separator. Every cell was cycled using constant current mode in potential range between 3.5 V and 4.5 V Charge – discharge current rates for LMO/n-GO tests varied from 1 C to 30 C, where 1 C corresponds to current density of 148 mA/g.

Acknowledgments

This work was supported by The National Centre for Research and Development through the research grant PBS1 (contract no. PBS1/A1/4/2012).

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17:10 Oral

Tb^{3+} ion luminescence enhancement in yttria host lattice obtained via microwave hydrothermal process

Jarosław Kaszewski, Sergiy A. Yatsunenko, Ewelina A. Wolska, Bartłomiej S. Witkowski, Łukasz Wachnicki, Marek Godlewski

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Yttrium oxide is very attractive optical material considering its properties: low optical loss, low phonon energies, high refractive index and wide band gap. And since the non-radiative relaxation from excited states is not efficient in Y_2O_3, the material is suitable as host lattice for optically active lanthanide ions. Yttrium oxide can be easily doped with the ions such as Tb^{3+}, Pr^{3+} and Eu^{3+} having characteristic emission lines in visible region.

In our study, the growth of Y_2O_3:Tb was conducted via microwave driven hydrothermal process. It was found that as prepared material did not contain pure cubic yttrium oxide phase, the hydroxides, oxohydroxides and nitrate complexes were also present. The annealing cycles were performed to show material crystallization evolution. The crystallites were found to be strongly agglomerated into needle-like aggregations. The characteristic trivalent terbium emission lines from $^5D_4$ and $^5D_3$ sublevels to ^7F_J were observed in all the samples. The Tb^{3+} ion shows spectrally narrow green emission bands in Y_2O_3, excited at relatively low energy, due to $4f^8 \rightarrow 4f^7 5d$ intra-ionic transition. After annealing an increase of intensity of the luminescence emission was observed. Interestingly, the mechanism of the trivalent terbium excitation has changed after calcination. The as grown samples exhibit the series of 4f intra-shell excitation lines with the maximum at 413 nm. On the other hand, the annealed samples have shown the host lattice - lanthanide ion coupling resulting in the appearance of the broad charge transfer bands at 306 and 337 nm. Such activation of the Tb^{3+} ions is related to the growth of the yttria crystallites and changes in dopant coordination.

17:20 Oral

Optical oxygen sensor for safe operation in hazardous areas

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Optical oxygen sensor based on luminescence properties of ZrO_2:Eu nanoparticles was invented in Laboratory of Nanostructures of IHPP. Nanopowder with particle size around 10 nm, synthesized in Laboratory, was compressed into thin pallet and irradiated with ultraviolet (250 nm) or blue (405 nm) LED. Experiments were done in different gas atmospheres. We registered that intensity of Europium luminescence spectra depended on the oxygen partial pressure in sample environment: increased when O_2 content decreased. The phenomenon was reversible and repeatable and gave a rise to the work on the new-generation oxygen sensor. Moreover, in our prototype device all electrical elements were eliminated from measurement area: low energy excitation light as well as luminescence signal are transmitted by optical fiber. Such construction make it possible to build an intrinsically safe and explosion-proof industrial sensor for work in hazardous environment, with flammable gases or dust, found in e.g. petrochemical refineries or mines. The future commercial version of the oxygen nano-sensor, built on the basis of the present laboratory model, will meet the requirements of the Directive ATEX.

Discussion

Discussion and questions to all speakers
Wednesday afternoon, 15 October, 17:30
Chair: Giancarlo Cravotto

Poster session, joint with symp. B

Wednesday afternoon, 15 October, 17:40

transfer to down-town

Wednesday evening, 15 October, 18:30

Thursday, 16 October

Registration and poster fixing

Thursday morning, 16 October, 8:00

Session 5

Innovative industrial research in advanced materials and nanotechnology in Poland
Thursday morning, 16 October, 9:00
Chair: Cristina Leonelli

9:00 Oral

Opening of the Thursday Sessions of NanoPl 2014

Witold Łojkowski

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9:10 Oral

Nano-inks for printing electric circuits for microelectronics technology

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The ink-jet is non-contact technique for production very complicate electronic patterns like conducting points, lines and even 3D structures for electronic applications. There are several important components of the ink-jet printing technology for electronic: the printing system, the conductive ink material and substrates.

The greatest challenge is the ink formulation, because these inks have to meet strict physicochemical properties: viscosity, surface tension, adhesion to a substrate, sintering temperature, etc. The most important is low viscosity and very homogeneous structure like molecular fluid with conductive nanosized particles. This type of fluids must be stable for a long time to avoid sedimentation during printing process and to achieve optimal performance and reliability of the printing system and obtain the best printed pattern.

Amepox Microelectronics developed two inks: high sintering temperature (230^{o}C) with particles size 3-8 nm (Fig. 1a) - trade name **AX JP-6n** and low sintering temperature (150^{o}C with particles size 50-60 nm (Fig. 1b) - trade name **AX JP-60n**.

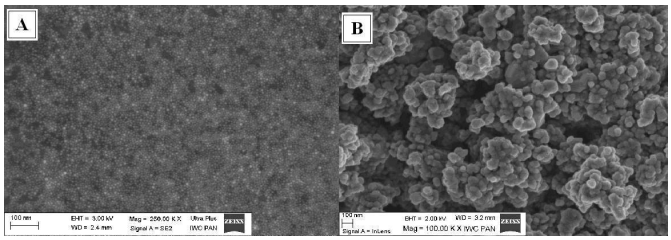

Fig .1 A – Nanosilver 3-8 nm, B – Nanosilver 50-60nm

The Inks have very high and stable electrical conductivity near value of pure silver. Inks natures (connected with molecular fluid properties) are perfectly homogeneous – fully uniform silver concentration in whole ink volume. This is also reason for very good repeatability of resistance and dispensed shapes: dots, lines, etc. The main technical parameters of the inks you can see in the Table 1.

Table1. Technical parameters of inks.

Ink	AX JP – 6n	AX JP – 60n
Consistency	Very low viscous ink	Very low viscous ink
Colour	Dark brown to black	Dark green to gray
Percentage of silver (inside ready paste)	40 – 60 %	20 % (with possibility up to 40%)
Viscosity	7.5 – 10.5 mPas (*)	5 – 6.5 mPas (*)
Thixotropy index (1/10 rpm)	~ 1.0	~ 1.0
Surface tension value	28.5 – 32.5 dynes/cm	~35 dynes/cm
Recommended curing & sintering conditions in convection oven	(220 – 230) °C – 60 min.	150 °C – 60 min.
Specific gravity	1,1-1,3 g/cm^3	0,8-1,0 g/cm^3
Eletrical resistivity	(4-6) x 10^{-6} Ωcm	5 x 10^{-6} Ωcm
Storage	2 months in refrigerator in temp. less 15°C (do not keep it in temp. less 5°C)	2 months in refrigerator in temp. less 15°C (do not keep it in temp. less 5°C)

(*) - Brookfield LVDVII + CP; 100 rpm; 20°C.

9:20 Oral

Real Time Characterisation of nanoMaterials

Roman Pielaszek

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9:30 Oral

Application of nanomaterials in chemical products

Stanisław Myszor[2], Jerzy Peszke[1,2]

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Every year comes on the market of products containing nanomaterials in its composition. One of the fastest growing markets in the medical and pharmaceutical market. Smart drugs, targeted therapies,

polymeric tissue implants - these are just some of the examples of nanostructures. One of the largest areas of applications of nanotechnology is the market disinfectants. The flagship product of nanotechnology in this area are nanoparticles of silver and copper, which have a very strong biocidal effect. In our presentation we present the applicability of these nanostructures for surface protection against the development of pathogens.

9:40 Oral

Air cleaning concrete - innovative TX Active technology in practical applications

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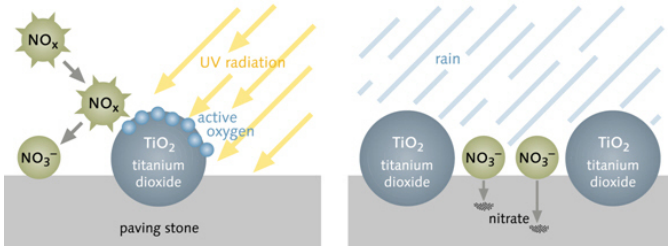
TX Active technology uses a photocatalyzer in concrete construction materials that accelerates the natural process of impurities decomposition. Only the sunlight is needed for activation of this process (UV-A radiation). Nanometric titanium dioxide (TiO2) is used as the photocatalyzer. Foundations of this technology provide also the requirements for the final construction materials needed to obtain the international TX Active trademark - reserved for the photocatalytically active materials that passed the certification process.

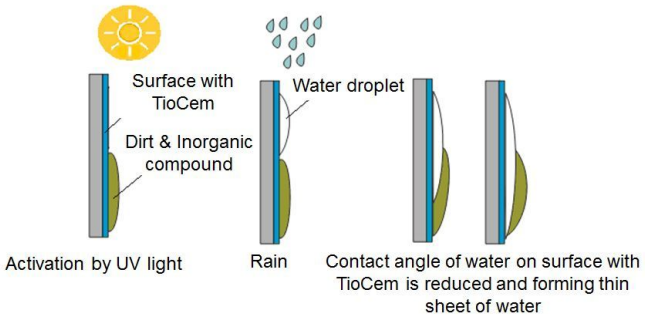
Picture 1. Logo of TX Active technology

Cement with trade name "TioCem" is used for the production of the photo-catalytically active concrete construction materials. This cement meets all requirements of the TX Active technology and is used in the following products: concrete bricks, roofing tiles, elevation panels, acoustic screens, etc. Because the TioCem cement is present in the concrete, such products have the specific properties. Their surfaces can efficiently reduce the impurities contained in the air, e.g. nitrous oxides (NOx), sulfur oxides (SO2), carbon oxide (CO), volatile organic compounds (VOC, such as benzene, toluene, formaldehydes) and many others. There is the significant fact that the purification process is long lasting and renewable during the whole "life" of the concrete product, because the photocatalyzer responsible for this property is not consumed during the purification process.



Picture 2. Diagram of nitrous oxides (NOx) reduction process through the photo-catalytically active surfaces of the concrete that contains the TioCem cement.

Efficiency of the reduction of nitrous oxides (NOx) by the specific product that uses TioCem cement is examined according to UNI 11247:2007 standard. Aside from the gas impurities reduction, the products based on the TioCem cement have also the self-cleaning properties. They allow to maintain the fresh (like new) look of the product for a long period of time, because the photocatalyzer can easily oxidize the impurities collected on the concrete element, such as greases, oils, bird droppings, etc.



Picture 3. High hygroscopic properties of the nanocrystalic titanium dioxide (TiO2) - self-cleaning of the concrete surface that contains TioCem cement.

On the vertical concrete surfaces, an additional self-cleaning effect is visible resulted from the reduction of the photocatalyzer humidification angle that decreases almost to zero. Thanks to this, the rain drops create thin water film on the concrete surface that facilitates reduction of impurities.

9:50 Oral

Microwave reactors for nanoparticle's synthesis

Andrzej Majcher[1], Jacek Wojnarowicz[2], Tadeusz Chudoba[2], Adam Mazurkiewicz, Jan Wiejak, Jan Przybylski, Witold Łojkowski[2,3]

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10:00 Invited Oral

Development of New Products in Synthos S.A.

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The Research and Development activity of the Synthos Group is focused on the development of three strategic areas: synthetic rubber, expandable polystyrene, dispersions and adhesives.

In the last three years Synthos successfully introduced numbers of products in all strategic areas. Presentation will include the most important of them: polybutadiene rubbers based on a neodymium catalyst (NdBR), expandable polystyrene with improved insulations properties and adhesives for wood in all classes of water resistance.

In 2015 Synthos will start production of solution styrene-butadiene rubber (SSBR) and low-cis polybutadiene rubber (BR) for tire application. The SSBR and NdBR grades are applied to high performance tire applications. Tires based on these rubbers have lower rolling resistance and better wet grip.

Presentation will also include information on the major projects run by Synthos R&D Department:

a) development of new functionalized grades of SSBR and NdBR (in-chain, chain-end) to improve silica filler dispersion in rubber matrix, which enables further improving of mentioned tire properties

b) development of two new production process for butadiene, one through direct fermentation of biomass, second by catalytic conversion of ethanol.

DISCUSSION and questions to all speakers

Thursday morning, 16 October, 10:20

Chair: Cristina Leonelli

Coffee & Posters & Exhibition

Thursday morning, 16 October, 10:30

Session 6

Strategy for innovation in advanced materials and nanotechnology

Thursday morning, 16 October, 11:00

Chair: Witold Lojkowski

11:00 Keynote

Developments in Nanotechnologies and Advanced Materials: a European perspective and innovation strategy

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Europe's advance in Nanotechnologies and Advanced Materials (NAM) in the last seven years was impressive. The driver behind such high level of research, innovation, investment and regulatory activity is the world-wide acknowledgement that these enabling technologies have great potential in addressing the main societal challenges that humanity is facing today such as, climate change, ageing and preventive health care, green manufacturing, resource efficiency.

Integration and convergence with other Key Enabling Technologies such as advanced manufacturing and a greater emphasis on innovation issues including value chain considerations are the features characterizing their progress. First attempts were directed to the designing and engineering of pilot lines for production of nanomaterials, components and devices. Nano-scale phenomena were utilised in increasing the performance of materials and their processing. Advanced material structures and systems were developed "fit-for-application". Safety, health, and the environment received special attention attempting to quantify societal impacts of nano developments.

Ethical, regulatory matters were reviewed and the legal framework wasplaced in an international context through partnerships and in cooperation with international organisations such as OECD, ISO, CEN, and UN. Support actions were funded to coordinate developments across Europe as well as internationally.

The next years, up to 2020 are critical. The expectations of high economic and social impact that nanotechnologies and advanced materials might bring can only be realized through an integrated strategy that is part of business and economic growth. Achieving such impact requires a total rethink of the legal and social governance framework under which production, markets, users, consumers and environment interact.

Current economic analyses of market trends recognises the synergistic and complementary role of nanotechnology in relation with the other Key Enabling Technologies (KET) such as micro-/nanoelectronics, , photonics, advanced materials, industrial biotechnology and their contribution to solving societal challenges. The commission's recent communication ‘A European strategy for Key Enabling Technologies – A bridge to growth and jobs’ (COM (2012341,outlines a single strategy for KETs, one of which is Nanotechnologies, to allow maximum exploitation of the EU’s potential in competitive markets.In Horizon 2020, the Commission's multi-annual framework programme, future developments (up to 2020) would target nano-system and/or advanced material applications whose complexity and high-level specifications demand strategic, multidisciplinary and integrated efforts. From concept to market, interdisciplinary teams need to work in a networked environment to overcome infrastructure and knowledge barriers and leverage investment. Building-up value networks and alliances for the different application need to be based on realistic future revenue streams. Business models have to be developed that attract venture capital and other forms of financing promoting industrialization.

Enabling such an innovation strategy to have an impact would require concerted effort supported by a world-class, knowledge-based regulatory regime accompanied by international standards. Supporting invention and innovation would be accompanied by societal and consumer engagements, based on an open and transparent system of proactive benefit and risk governance. Finally, economic progress would only be possible through the creation of a favourable world-class educational and skilling system that delivers a knowledgeable

and well-trained workforce.

The first year of implementation under Horizon 2020 is already bringing in encouraging results both in terms of participation and thematic coverage.

11:20 Keynote

Financing of industry relevant projects in Poland

Krzysztof J. Kurzydlowski

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11:40 Keynote

Strategic Programmes - Tools for Financing Research and Development in Poland

Jerzy Kątcki

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Adopted by the Council of Ministers the National Research Programme (NRP) sets the goals and guidelines for the science & technology and innovation policy of the state. In this document seven strategic, interdisciplinary R&D directions are defined. They are fundamental for the National Centre for Research and Development (NCRD). Guided by NRP the Council of the NCRD prepares drafts of strategic R&D programmes. The final draft of the strategic R&D programme is presented for approval to the minister responsible for science. Next, the approved programme is implemented in NCRD. In this short talk, the way of reaching the final version of the strategic R&D programme will be discusses.

Discussion and questions to all speakers

Thursday morning, 16 October, 11:50
Chair: Witold Lojkowski

Begin of the Ministers Panel

Thursday afternoon, 16 October, 12:00

Lunch & Posters & Exhibition

Thursday afternoon, 16 October, 12:01

Session 7

Applied nanotechnology
Thursday afternoon, 16 October, 14:00
Chair: Ludwika Lipinska

14:00 Keynote

Successful Market Introduction of Multi-Functional Nano-Coatings

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Artificial Nanotechnology is a broad cross-section technology, which includes both physical and chemical technologies. Within the chemical nanotechnology - coatings with additional functionalities are important. Special effects that are expected from nanotechnology, for example are a viewing angle-dependent color impression, plasmons/luminescence effects and - as a function of temparature - switchable thermochromic effects. To formulate such systems, the nano-agents must be coupled with suitable polymers, which also have to meet high requirements in terms of liability, optimized surface hardness in combination with sufficient elasticity.

The aim of such product-oriented research, such as the Fraunhofer Society at the Fraunhofer Institute for Chemical Technology (ICT) in Pfinztal (Germany) operates, is ultimately - having achieved sophisticated results, process stability, quality assurance and homologation - to organize and establish production technology in order to introduce these systems into the market. This is possible only in cooperation with other partners that are already anchored in these markets with the capability to cover marketing and distribution aspects completely.

The paper uses the example of wire coatings, which are applied in the form of woven wire mesh as facade cladding in architectural applications and points out all steps - starting from research and development - up to successful product market introduction.

14:20 Keynote

Enabling technologies for nanomaterial grafting and coating

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The so called “enabling technologies” such as microwaves, ultrasound, ball milling, hydrodynamic and cavitational reactors, atmospheric plasma, dramatically changed the way we think of material grafting and coating. Besides an efficient heat and mass transfer, these techniques may generate high-energy micro-environments and hot spots that strongly enhance reactivity and reaction rates even at low temperature [1,2]. Although combined irradiation with these energy sources entails technical and safety considerations, sequential or simultaneous treatments can easily be performed on a lab and pilot scale with unexpected synergistic effects [3-5]. This has been described when dielectric heating is associated with the large amount of energy released in cavitational collapse, causing particle fragmentation and molecular excitation [6,7]. The cavitation-based

mechanical effects, arise from shear forces, microjets and shock waves that occur outside the bubble, resulting in profound physical changes when solids or metals are present. These changes include improved mass transfer, particle size reduction, surface erosion and cleaning, and are often accompanied by changes in particle properties. Likewise, mechanochemistry can also generate radicals via the breaking of weak bonds and under extreme surface plasma conditions where covalent crystals are cracked by mechanical impact [8]. We experimented all these enabling technologies to graft carbon based nanomaterial [9-11], silica particles and fabric surfaces [12] with cyclodextrin derivatives [13]. Relationship and synergy among different energy sources for nanomaterial grafting and coating, may help to fully understand their features and great potential, highlighting, whenever possible, comparative aspects.

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14:40 Invited Oral

Superhard carbon coatings for an improved energy efficiency

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Rising prices for fossil fuels as well as the increasing effects of the climate change due to the emission of greenhouse gases reveal the necessity of saving energy. Within Europe especially the individual transport is in the focus of the public awareness. Therefore, the European commission has set ambitious goals in order to reduce the energy consumption and as a consequence the emission of CO_2.

Low friction coatings have an enormous potential in saving energy. According to a recent study it is estimated that low friction coatings can save within the next ten years about 117 billion liters of fuel per year. This corresponds to savings of 290 million tons of CO_2 emission.

Carbon based coatings – named as DLC coatings –are especially well suited for low friction coatings. In particular hydrogen-free tetrahedral amorphous carbon (ta-C) coatings are of great interest due to their extraordinary low wear properties. In addition they show excellent low friction properties and especially in combination with specific lubricants the so-called super low friction effect.

For the deposition of ta-C coatings PVD methods have to be applied instead of CVD methods as it is the case for conventional DLC coatings. We have developed a deposition method which is based on a pulsed arc steered by a laser (Laser-Arc). This allows us to use large cathodes resulting in a high long-term stability. Furthermore, the carbon plasma source can be combined with a filtering unit removing almost all droplets and particles, which usually are characteristic for an arc process. The resulting Laser-Arc source allows for the deposition of smooth and virtually defect-free ta-C coatings with a competitive deposition rate.

Due to its excellent technical parameters the Laser-Arc technology is increasingly used for industrial purposes especially in the automotive industry. Examples of applications as well as details of the deposited coatings will be given.

15:00 Invited Oral

Ultra clean concentrated nanometals muster-butches in custom carrier liquides

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A social importance of nano suspensions of the oligodynamic metals (Ag, Cu, Au) on the base of various carrier liquids is to replace dangerous and highly allergic chemical disinfectant agents as chlorine, dichlorizocianic acid, triclosan, peroxyacetic acid, evaporated aldehydes, and other environmental unfriendly chemical agents.

The Non-organic nanoadditives –metallic nanoparticles - are widely used for end-product consumer's properties modification for example for wide range of consumer products modification to upgrade end products properties without changing their traditional manufacturing technologies. That is why the "muster-butch" approach - the way of capsulation of the active ingredients (for example metal nanoparticles) in concentrated form in liquid media widely used as ingredients in thousand end products (glycerin, oils, glucose, polyssacharids, etc is the perspective way of wide commercialization of such products.

JV Marketing of Superhard Material proposes the high-tech solution in the production processes for customised healthy, green and safe liquid nano products easily implantable to wide range of traditional

consumer products to modify and upgrade home disinfectants, paints, packing materials, plastics for food preparing or nanoadditives used for air, water conditioning, and contact surfaces disinfection in public places etc.

Nanoadditives manufactures and supplies under two trade marks: “Silver Shield 1000” and “Nanoagent+”. They are concentrated ultra clean nanometals particles (or their blend) dispersed in various carrier liquids which are the actual ingredients of end products variety. The advantages of this nanoadditives (or concentrated muster-butches) are:

· easy way of modification of end-products by simple mixing with nanoadditives non changing their traditional productive technologies

· ultra clean nano metals additives - no traces of any waist chemical reagents or by-products [1]

· controlled metal nano particles size and distribution [2]

T|his master-butches are water soluble or water non-soluble depending of the property of carrier liquid. For example Silver Shield 1000 (see specifications on fig 1) is concentrated nanosilver dispersion in food glycerin which could be used as water solution to obtain desirable concentration. Depending on this solution concentration it serves like nanodisinfectant for contact surfaces eliminating bacteria, viruses, fungus (see table on fig 2) and can be supplied like self-sufficient disinfectant (see product on fig 3). At the same time concentrated nanoproduct Silver Shield 1000 can be added to liquid and hard soap, shampoo, tooth paste or dozens of personnel care products to upgrade their bactericidal properties. The same products is easily implantable to natural porous dry matrix to create low release nanoadditives for paints, hermetic sealing, lacquer etc.

From the other side nanoadditives Nanoagent+ are concentrated dispersions of various nanometals - Silver, Gold, Platinum, Copper – in natural or artificial oils, polysaccharide, plastics. This nanoadditiave could be widely used for example for anti-aged nanomodified cosmetics products which are the safest alternative to botulinum injectiones and provides guaranteed noticeable results due to deep penetrating ability and strengthening of collagen layers. Exisiting nanogold and nanoplatinum cosmetic products lines are extremely expensive due to non productive chemical processes of nanoparticles synthesis. Innovative nanoadditives produce by one-step in plasma technology in vacuum are economically competitive with the best world known of nanoadditives in this area of application.

JV Marketing of Superhard Material can offer to potential customers various way of collaboration starting from manufacturing of custom nanometal muster-butches in chosen carrier liquids to creating JV for modified end-products manufacturing or selling the license for innovative technology and productive industrial unit for nanoadditives manufacturing.

Literature

1. Eco-clean technology plasma nanodispersion of electrically conductive materials with one-step cycle of concentrated nanosuspension manufacturing in a wide range of liquid media / M.V. Novikov, L.D. Kisterska, V.V. Sadokhin et all // Powder metallurgy. - 2012. - № 1/2. - P. 34 – 45

2. Physico-chemical properties of metal nanoparticles controll in process of of colloidal solutions preparation by ion plasma sputtering. / V.V. Sadokhin, O.B. Loginova, L.D. Kisterska / / Perspective of science, Russia - 2013. - №11 (50) - P. 104 - 107.

15:10 Oral

Development of disperse system nanoprocesses using a new supersonic rotor mill

Zbigniew Najzarek[1], Remigiusz Petrich, Janusz Wełnowski[2]

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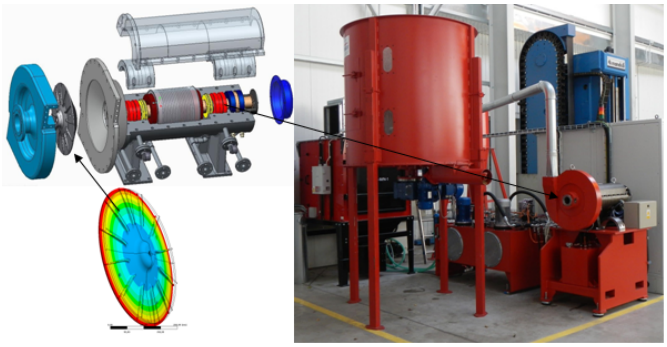
Micro- and nano-disperse systems , particularly aerosols, liquid and solid suspensions, would help increase the added value of end products in many industrial sectors or, on the other hand, they may increase the adverse environmental impact with human health risk. The above effects, beneficial and harmful, reach a maximum in the disperse system processes involving nanoparticulate reagents. Currently, processes involving these nano-disperse systems are carried out intensively and extensively. The nanomilling process has been proven to be a robust technique for generating and processing of nanoparticle suspensions and emulsions.



The high speed rotor mill with supersonic capability from Hydrapress Engineering

However at present, all the known method for preparation of micro- and nanodisperse systems, including the above nanomilling, are limited in terms of applicability, Additionally, they are very slow, complex, and consume large amount of energy, because they use devices, e.g. the known mills, ineffective for the processes at the nanoscale. In addition, such milling changes the structure and hence properties of the nano-reactants in an uncontrolled manner.

In our previous work, a new high-speed centrifugal mill was presented, which efficiently deagglomerate

nanomaterials such as carbon black and micro silica, up to primary nanoparticles [1].

Consequently, the purpose of this work was to develop the micro- and nanodisperse systems by the nanomilling process using the new high speed rotor mill with supersonic capability (*Figure)* [2]. As a result, the mill was adjusted to the function of a new disperse system generator capable to developing the disperse systems from a variety of materials. Thus, the mill can be used to production reactive dispersions for green synthesis of nanomaterials. Moreover, the mill can be used for generating aerosol standards for performance

testing of nanoparticulate control devices.

Advantages of the present nanomilling technique in relation to the state of the art lies in the fact that it is at least 1000 times faster, energy efficient, simple, and waste-free.

Presented mill and method of nanomilling have no equivalent in the state of the art.

Using this method, one may receive composite nanomaterials as membranes, catalysts, fuel, pharmaceuticals and cosmetics, as well as lead research towards reducing emission of diesel engines and power plants.

References

[1] Z. Najzarek, Proc. IX Konrf. Technologie bezodpadowe, Międzyzdroje, 385.

[2] H. Holka, J. Wełnowski, Inżynieria i Aparatura Chemiczna 51 (2012) 53

Discussion and questions to all speakers

Thursday afternoon, 16 October, 15:25
Chair: Ludwika Lipinska

Coffee & Posters & Exhibition

Thursday afternoon, 16 October, 15:40

Session 8

Carbon and graphene
Thursday afternoon, 16 October, 16:10
Chair: Andreas Lesson

16:10 Keynote

From the mine to the tops: Graphene oxide – the mysterious derivative of flake graphene. Properties and applications

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The properties of graphene will be characterized and various methods of its preparation will be briefly described. Among the "top down" techniques the chemical methods are considered as most promising for mass production of flake graphene dedicated to a very wide range of applications, including composites. There are many ways to achieve effective, "wet" exfoliation of graphite. Among them the oxidation-reduction method is the closest to an industrial scale. In addition, the graphene oxide the intermediate product has very interesting properties and many potential applications. It shows a strong luminescence, after decorating with metal nanoparticles is gaining catalytic properties. It is an excellent material for the construction of various types of sensors. It has outstanding adsorption properties, provides an excellent platform for the deposition of various types of molecules e.g. biological. The second presented method is liquid phase exfoliation of graphite in organic solvents by means of ultrasounds. Both methods have advantages and limitations. The method of liquid phase exfoliation gives flakes with very small number of defects, but thicker consisting of several layers of carbon. Furthermore, the efficiency of this method is still low, it usually uses expensive chemical reagents. Oxidation-reduction method is very efficient and allows the receiving monatomic carbon layers, which are characterized by a very high transparency. Its a limitation is the difficulty in obtaining complete reduction, which results in decreasing of the electrical conductivity of reduced graphene oxide. Wet methods are ideal for the preparation of loose graphene powders of high specific surface area, thin films on various substrates and graphene paper. The most important directions of applications of flake graphene will be shown on the basis of market research "The Global Market of Graphene (Forecast from 2010 to 2020)."

16:30 Oral

Biocarbon mechanochemical activation

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At present, biocarbon (biocoal, charcoal, torriiefied biomass, as well as hydrochar) can be produced efficiently and economically by heat treatment (i.e. via pyrolysis, carbonization or torrefication) as well as by hydrothermal (HTC) or vapothermal (VPC) carbonization of the lignocellulosic biomass, such as: wood, sawdust, straw, peat, crop residues and other waste plant residues, municipal and industrial waste, moses, and related substrates. In particular, straw as the abundant and renewable agricultural residue that are not useful in obtaining food constitute an important source of precursors for the biocarbon production especially after pre-treatment in a new mill introduced here.. Biocarbon thus produced has no heavy metals, contains almost no sulfur, very little nitrogen, low ash, and carbon dioxide emission from such source is not counted as the greenhouse gas emission. Because storage and transportation are safe here, functional infrastructures were established to deliver biocarbon from producers to consumers which promote its use. The carbonization processing were performed within a wide temperature range, in a controlled gas atmosphere, under a controlled pressure, and with the optimal presence of a catalyst. Therefore, the structure and properties of the biocarbon can be varied widely and tailored to the specific requirement of each application. The biocarbons are generally composed of non-carbonized and carbonized organic matters and their relative fractions are affected by the above processing conditions. The structure varies from amorphous to amorphous-crystalline, and the biocarbons represent essentially a nanocomposite made up of amorphous carbon, graphite nanocrystallites and graphene layers. Moreover, as-produced biocarbon can maintain the biomorphic pore microstructure of the lignocellulosic substrate which efficiently increases its performance in various applications. However, so far received biocarbons consist of poorly structured agglomerates of biocarbon-derived nanoparticles. The properties of these agglomerates are not improved in environments of the well-known mills and mechanochemical reactors. In addition, such conducted

mechanochemical processes run at the nanoscale reactants slowly and generallny inefficiently. Instead, they cause the destruction of the above mentioned microstructure of technologically preferred reactants. On the other hand, nanoparticulete materials that can be prepared from conventional chemical reactants via the carbothermal processing as well as carbon nanotube fibers that were able to isolate from the environment of the carbothermal processing of the lignocellulosic substrate showed very favorable performances. Therefore, the introduction here of a new, efficient mechanochemical processing has essential importance. Apart from the pore structure, the functional groups on the biocarbon surface affect significantly the biocarbon properties However, the known physical and chemical activatiopn methods used so far for the case of biocarbon does not allow control of such changes in its properties. Known methods do not allow even an effective micronization of the biocarbon in spite of these facts, that such micronization enables optimization of the majoraty of biocarbon applications and runs much easier than micronization of the starting biomass. According to the literature [V. Soloiu, et al.., Energy 36(2011)4353-4371], the most efficient micronization of biocarbon led .to polidisperse size distribution with average size of particie 10.5 mm and significant fractions of larger particles over 50 mm and up to 200 mm which limits such microparticle application range, e.g. as the diesel engine fuel. This micronization was carried out in a laboratory scale multi-step slow batch process of biocarbon slurries from selected wood.. Comparatively, in this work micronization proceed in the solid phase, which allowed efficient mechanochemical activation of as-formed microparticles, as a continuous, well-controlled process on an industrial scale at a rate about 1 kg/s. The literature average size of particle 10.5mm was attained here far from the maximal performance of the new supersonic rotor mill [J.Wełnowski, et al.., Patent application (2013)], within the monodisperse size distribution, without the fraction of larger particles. Such process of efficient micronization with mechanochemical activation was carried out for biocarbons prepared from the rye straw as well as from a number of representative and renewable lignocellulosic waste. These waste were pyrolysed according to the literature method [C. Schmedt, G. Bogdanow, EP 2565255(2013)]. As a result, this wok introduces a new method of micronization and mechanochemical activation of biocarbon. This method is characterized by a very high efficiency. Such activated microparticles can improve the quality of biocarbon-derived products, especially electrochemical, sorptive, composite, catalytic, as well as fuel, chemical reagents , and fertilizers. This method is currently being extended for the nanoprocessing purposes

Discussion and questions to all speakers
Thursday afternoon, 16 October, 16:40
Chair: Andreas Lesson

Poster Session A & B
Thursday afternoon, 16 October, 16:50

Transfer to down- town
Thursday evening, 16 October, 18:00

LED illumination show
Thursday evening, 16 October, 19:00

DINNER
Thursday evening, 16 October, 19:30

End of the day
Thursday night, 16 October, 22:00

Friday, 17 October

REGISTRATION
Friday morning, 17 October, 8:30

Joning Symp. B activities
Friday morning, 17 October, 9:00

Posters

Wednesday, 15 October

POSTERS A & B

Poster's fixing to be displayed 3 days
Wednesday morning, 15 October, 11:00

11:00	Poster	26

Laboratory of Scanning Electron Microscopy and X- ray Microanalysis at the Kielce University of Technology as research and industry support

Justyna M. Kasińska, Piotr Furmańczyk, Krzysztof Antoszewski

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The development of microscopy is a direct result of advances in the technology of materials that have been observed for the last several dozen years. Relatively low resolving power of light microscopes contributed to the design of electron microscopic methods which enable the researchers to view nano- and sub-nano-sized objects. Modern transmission electron microscopes can easily resolve details of 0.05 nm.

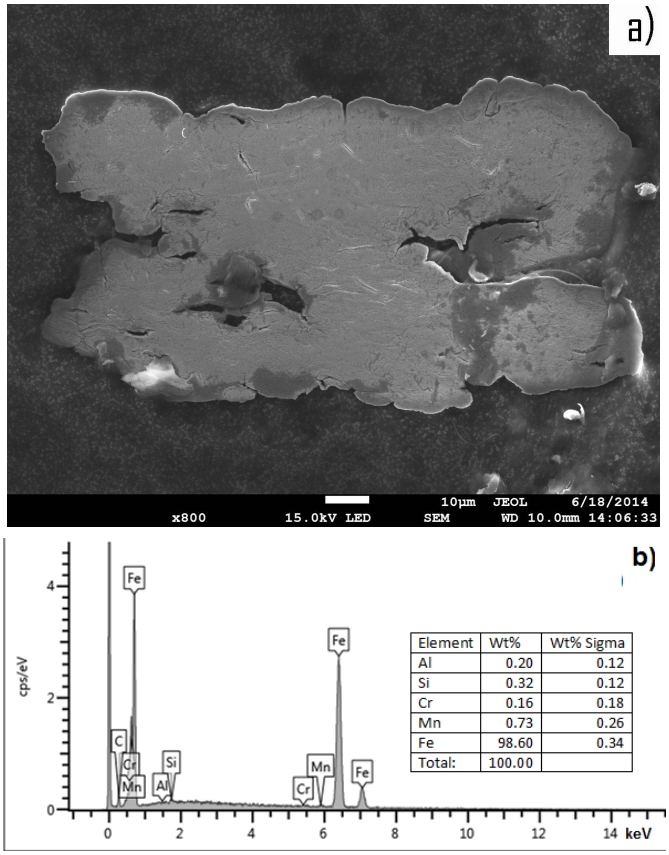
Introduction

Today, the resolving power of scanning microscopes is approximately 1nm. The resolution of the produced image is directly related to the wavelength of electrons, which is a function of accelerating voltage used (the higher voltage the shorter wavelength and thus the better resolution), and to the diameter of the beam. One of the advantages of scanning electron microscopes is that they allow observations of certain surfaces without the need for them to be processed

to produce a suitable sample. Kielce University of Technology purchased a modern JEOL JSM 7100F- scanning microscope in the framework of the project called “Developing research resources for specialized labs at public universities in the Świętokrzyskie region” co-financed from the European Fund for Regional Development under the “Innovative Economy” Operational Programme. The resolving power of the JEOL JSM 7100F is 1.2 nm, which allows the user to observe nanostructures. The observations at the Laboratory of Scanning Electron Microscopy and X-ray Microanalysis range from those with 25-fold magnification to 100 000x working magnification used to view objects with dimensions less than 20 nm.

Contracted services. Representative SEM images.

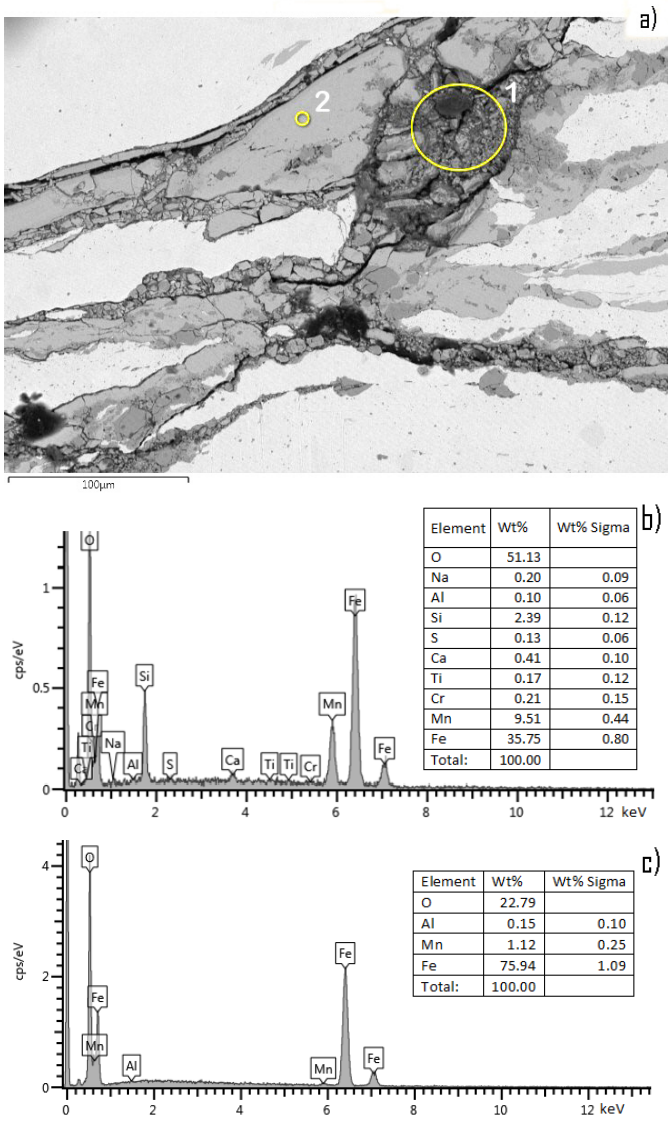
1. Identification of contamination in the elements of parts of machinery. The analysis allows determining the chemical composition of the contamination.



Element	Wt%	Wt% Sigma
Al	0.20	0.12
Si	0.32	0.12
Cr	0.16	0.18
Mn	0.73	0.26
Fe	98.60	0.34
Total:	100.00	

Fig.1. Analysis of the chemical composition of the contamination; a) image of the contamination at 800x; b) EDS spectrum of the contamination.

2. Identification of non-metallic inclusions in a rolled member. The research led to the elimination of inclusions from the finished rolled members.

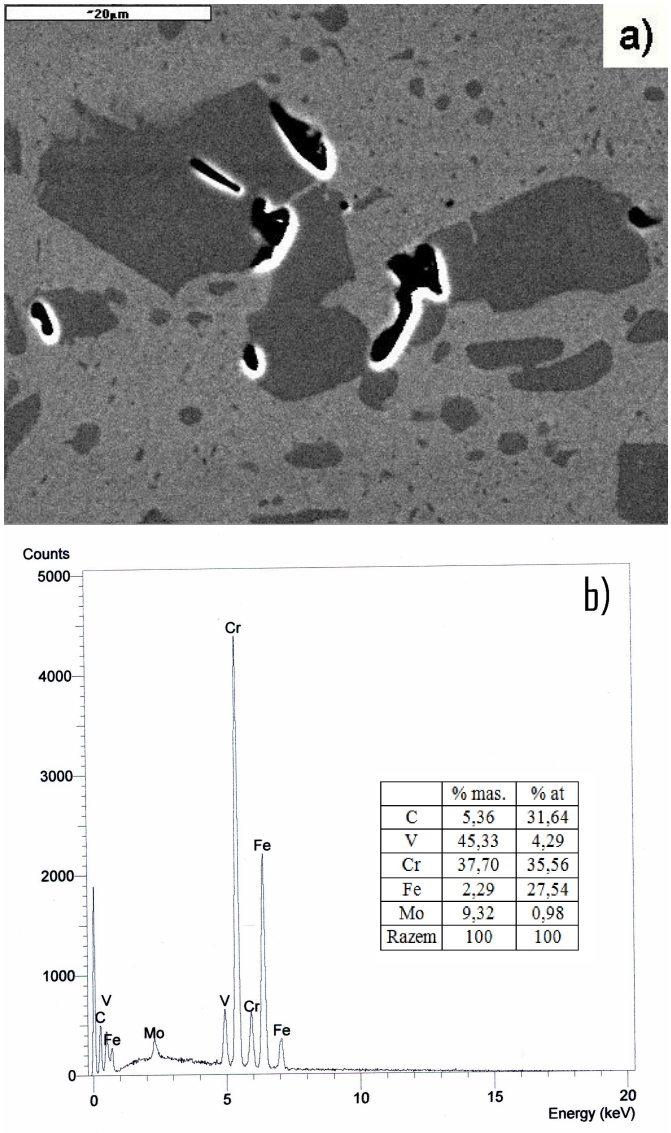


Element	Wt%	Wt% Sigma
O	51.13	
Na	0.20	0.09
Al	0.10	0.06
Si	2.39	0.12
S	0.13	0.06
Ca	0.41	0.10
Ti	0.17	0.12
Cr	0.21	0.15
Mn	9.51	0.44
Fe	35.75	0.80
Total:	100.00	

Element	Wt%	Wt% Sigma
O	22.79	
Al	0.15	0.10
Mn	1.12	0.25
Fe	75.94	1.09
Total:	100.00	

Fig.2. Analysis of non-metallic inclusion; a) non-metallic inclusion at 400x; b) EDS spectrum and chemical composition from the region marked as 1 in Fig. 2a; c) EDS spectrum and chemical composition from the region marked as 2 in Fig. 2a (EDS)

3. Identification of the causes of metallurgical flaws-related cracks in the matrix. Observations of microstructure and analyses of the matrix chemical composition allowed the detection of metallurgical defects, which were the major factor in initiating the crack.



	% mas.	% at
C	5,36	31,64
V	45,33	4,29
Cr	37,70	35,56
Fe	2,29	27,54
Mo	9,32	0,98
Razem	100	100

Fig. 3. Analysis of the inclusion; a) inclusion in the structure of the matrix material, unetched, 2 000x; b) EDS spectrum and the chemical composition of the inclusion in Fig. 3a.

Research outcome, examples

1. Study of carbon-nickel nanolayers produced using physical vapour deposition (PVD) and the layers modified in the chemical deposition (CVD) process. The carbon-nickel layer on the sample is several hundred nm thick and consists mainly of fullerene C60 with an addition of nickel crystallites, Fig.4. In the process of chemical vapour deposition, carbon nanotubes form on the nickel crystallite embryos, Fig. 5. The layers investigated were produced at the Tele&Radio Research Institute, Warsaw by he research team headed by Prof. Elżbieta Czerwosz.

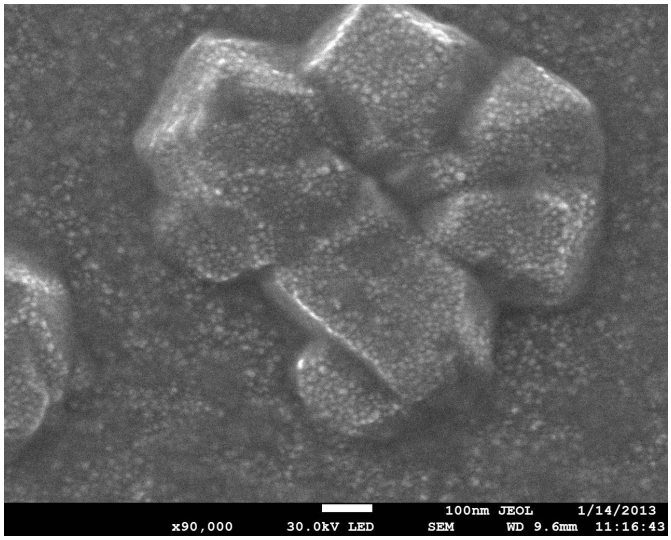


Fig 4. Carbon-nickel nanolayer prepared using physical vapour deposition (PVD) at 90 000x.

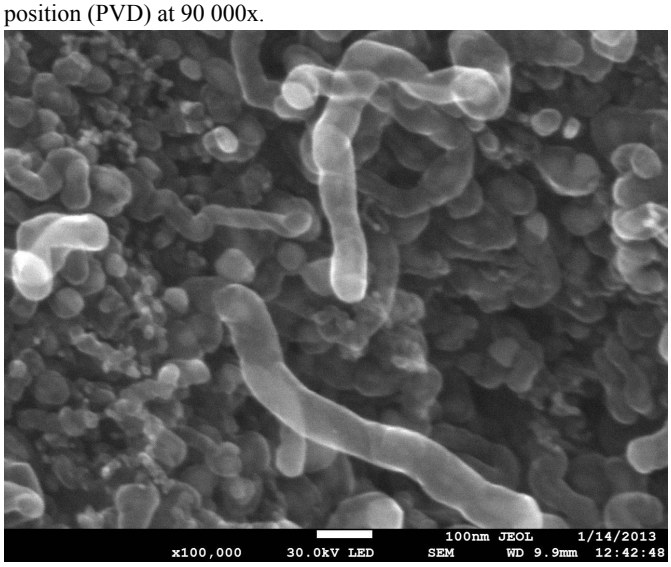


Fig. 5. Carbon-nickel nanolayer modified using chemical vapour deposition (CVD) – nanotubes at 100 000x.

2. Study of the morphology of Al_2O_3-13TiO_2 nanostructured powder grains used in plasma spraying.

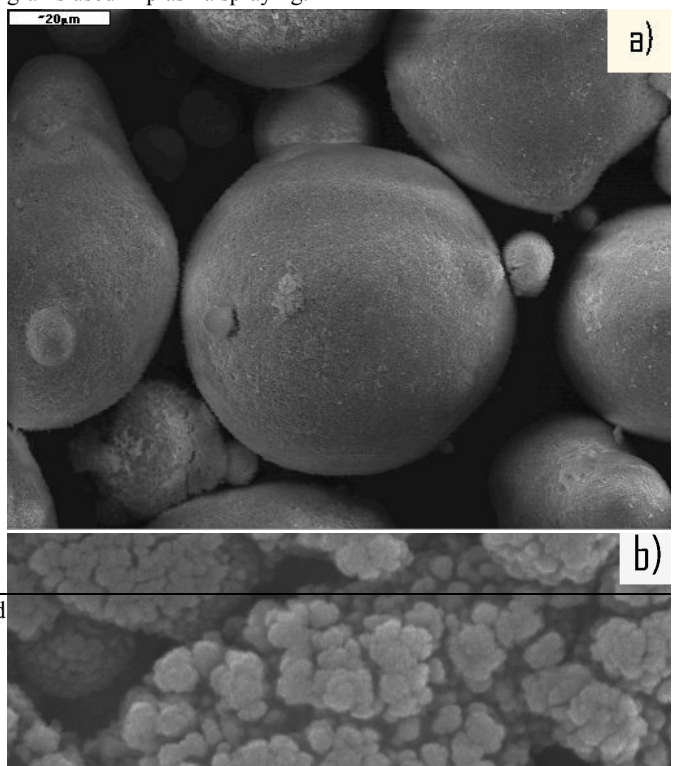


Fig. 6. Al_2O_3-13TiO_2 nanostructured powder grain; a) image of a powder grain at 2 000x; b) image of the grain surface at 100 000x.

3. Testing biological samples. Dry samples are coated with a conductive layer of gold or carbon prior to being observed. Figure 7 shows a seed of Xylanche himalaica, a very rare plant from Fujiyama, Japan, parasitic on Rhododendron species 3000 m above sea level.

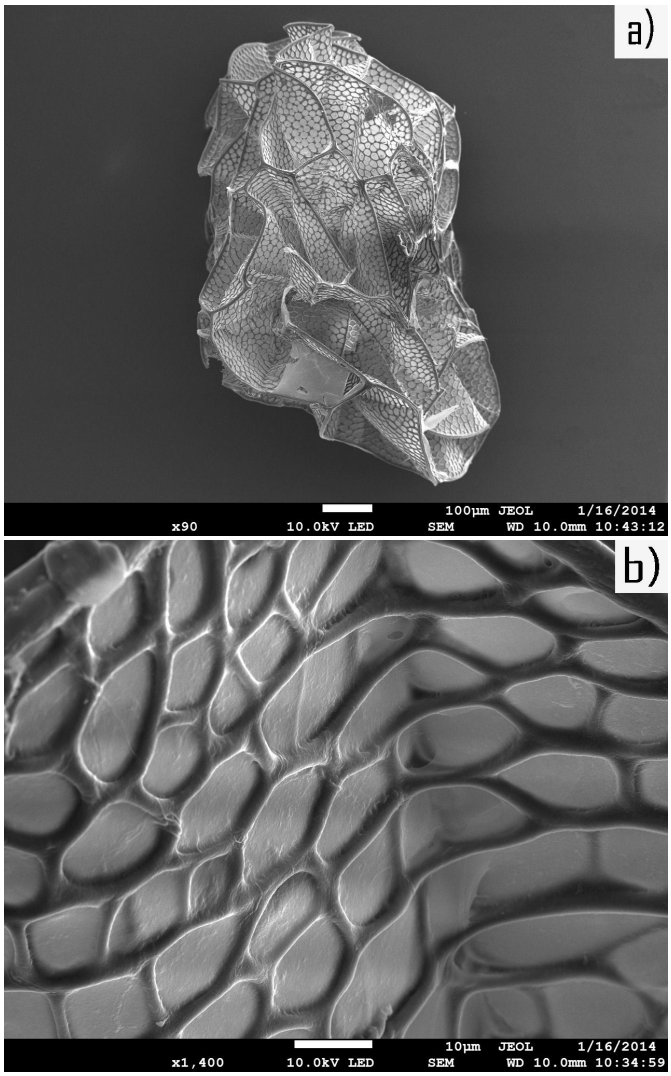


Fig. 7. A seed of Xylanche himalaica (Orobanchaceae) a) general image at 90x b) image of a seed wall at 1400x.

Summary

The Laboratory of Scanning Electron Microscopy X-ray Microanalysis at Kielce University of Technology provides analytical, research and developmental support for academic and industrial organizations. Typical applications include:

- product quality control and the analysis of flaws in the form of cracks, non-metallic inclusions, corrosion, etc.;

- evaluation of plastic forming, thermal and thermal-chemical treatment samples; material identification;

- preparation of test specimens;

- Vickers/micro and macro hardness measurements; - Investigations of non-metallic samples including ceramics, concrete and biological specimens.

11:00	Poster	27

Możliwość oceny struktury geometrycznej powierzchni powłok diamentopodobnych za pomocą wybranych metod

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Zapewnienie trwałości maszyn i urządzeń oraz niezawodności ich działania, wymaga zwiększenia odporności na zużycie materiału, z którego wytworzone są poszczególne części poprzez zastosowanie cienkich powłok o korzystnych właściwościach mechanicznych, tribologicznych i korozyjnych.

We współczesnej technologii znaczącą rolę odgrywają materiały bazujące na węglu , który może krystalizować w diamentowe lub grafitowe formy. Przez pojęcie powłoki diamentopodobne DLC (*diamond-like carbon coatings*) rozumie się ogromną liczbę amorficznych, w większości przypadków uwodornionych, cienkowarstwowych materiałów o różnych właściwościach zależnych od metody i warunków ich otrzymywania.

Ważnym elementem podczas kompleksowych badań wpływu cienkich powłok na właściwości systemów tribologicznych są pomiary struktury geometrycznej powierzchni prowadzone na różnych etapach analiz obejmujące:

v Pomiary grubości powłok

v Ocenę struktury geometrycznej powierzchni z naniesioną powłoką

v Obrazowanie rysy uzyskanej w trakcie badań adhezji metodą zarysowania (scratch test)

v Ocena struktury geometrycznej powierzchni elementów węzła tarcia (kulka –tarcza) po badaniach tribologicznych

Ze względu na to, że stan struktury geometrycznej powierzchni jest jednym z ważniejszych czynników decydujących o właściwościach mających wpływ na funkcjonowanie elementów takich jak zdolności ślizgowe, smarujące, odporność na ścieranie, wytrzymałość zmęczeniowa, przewodnictwo cieplne i elektryczne, odporność na korozję, szczelność i sztywność połączeń następuje stały rozwój metod i przyrządów służących do pomiarów przestrzennych zarysów nierówności powierzchni.

Najważniejszymi metodami służącymi do pomiaru stereometrii powierzchni są: profilometria stykowa z ostrzem odwzorowującym, interferometria z przesunięciem fazy, interferometria skaningowa koherentna, mikroskopia konfokalna, mikroskopia konfokalna chromatyczna, mikroskopia holograficzna, mikroskopia tunelowa skaningowa oraz mikroskopia sił atomowych.

Każda z tych metod charakteryzuje się innym zakresem możliwości pomiarowych wynikającymi z rozdzielczości w poszczególnych osiach, zakresami pomiarowymi, możliwościami penetracji nierówności uzależnionymi od ich geometrii czy oddziaływaniem na powierzchnię mierzonego przedmiotu.

Spośród wielu metod pomiarów stereometrii powierzchni do oceny powłok DLC zastosowano następujące metody: profilometrię stykową, koherentną interferometrię korelacyjną i mikroskopię sił atomowych.

11:00	Poster	28

Zaawansowana Charakteryzacja Nanocząstek

Jacek Wojnarowicz, Agnieszka Opalińska, Sylwia Kuśnieruk, Elżbieta Pietrzykowska, Tadeusz Chudoba, Anna Swiderska - Sroda, Adam M. Presz, Jan Mizeracki

Instytut Wysokich Ciśnień PAN (IWC), Sokołowska 29/37, Warszawa 01-142, Poland

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Kierunki badawcze Laboratorium Nanostruktur IWC PAN skoncentrowane są na charakterystyce nanomateriałów oraz możliwości zastosowania ich w medycynie, optyce, optoelektronice farmacji i kosmetyce.

Poprawna charakterystyka nanometrycznych materiałów jest niezbędna w celu określenia właściwości materiałów. Prace w tych obszarach wymagają nowoczesnego zaawansowanego sprzętu pomiarowego. Dzięki współfinansowaniu z europejskiego projektu CePT (Centre for Preclinical Research and Technology) laboratorium zakupiło takie urządzenia jak:

- analizator rozkładu wielkości cząstek oraz potencjału zeta z automatycznym systemem titracyjnym MPT-2 Zetasizer Nano ZS, Malvern
- analizator rozkładu wielkości cząstek NS500, NanoSight
- skaningowym mikroskopem elektronowym z grupy "Ultra-High-Resolution Imaging" ULTRA plus firmy Zeiss, Mikroskop jest wyposażony także w system mikroanalizy EDS firmy Bruker mod. Quantax 400 z ultraszybkim (do 300 kconts/s) detektorem o rozdzielczości energetycznej 127eV i powierzchni czynnej 30mm2 umoliwiającym dedekcję od pierwistka boru
- system do badania stabilności temperaturowej TGA/DSC (NETZSCH, STA 449 E1 JUPITER) sprzężony z systemem spektrometrii masowej (NETZSCH, Gas Analitycal System QMS 403C) oraz spektroskopem FTIR (BRUKER, System TENSOR27)
- analizator lepkości DV-II+Pro BROOKFIELD
- analizator napięcia powierzchniowego Sinterface, model BPA-1P
- analizator stabilności zawiesiń i emulsji Turbiscan Lab, Formulaction
- reaktor Mettled Toledo, OptiMax™
- Multi-parameter Instrument ProLab 2000, SI Analytics, pomiar pH, przewodności, stężenie jonów wapnia

Oprócz tego laboratorium posiada takie urządzenia badawcze jak:

- proszkowy dyfraktometr rentgenowski: Panalytical, model X'Pert PRO and Bruker, model D8
- analizatory gęstości, piknometr helowy Micromeritics AccuPyc II, model 1340 oraz Micromeritics AccuPyc, model 1330
- analizator powierzchni właściwej Micromeritics AccuPyc, model Gemini 2360
- skaningowym mikroskopem elektronowym Mikroskop Zeiss z emisją polową i kolumną Gemini z grupy „high resolution low energy", z systemem mikroanalizy EDS firmy Oxford, model Link ISIS 200 z detektorem Si-Li,

Laboratorium Nanostruktur IWC PAN zajmuję się charakterystyką nanomateriałów otrzymanych przez nas zespół, jak również świadczy usługi badawcze takie jak analiza gęstości, powierzchni właściwej, morfologii, składu fazowego, termograwitetrii nanomateriałów, napięcia powierzchniowego, lepkości, stabilności oraz wielkości nanocząstek w roztworach koloidalnych i zawiesinach.

11:00	Poster	29

Microwave Solvothermal Technology (MSS) for nanoparticle synthesis

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Microwave hydrothermal synthesis is one of hydrothermal processes that are used for the procurement of nanopowders with controlled chemical content and morphology characterised by minor differences in the size of the grains.

Most microwave hydrothermal synthesis reactions still take place in laboratory in modified microwave ovens. Commercial devices for microwave thermal syntheses can be divided into large closed vessel reactors, stop-flow reactors and flow reactors. Main drawbacks of existing solutions are as follows: low efficiency (in the case of large vessel reactors and stop-flow reactors), low flow of the substrate and the product of particularly thick suspensions in flow reactors, relatively low pressure and temperature of operation, the possibility of product contamination due to the contact with metal parts of reactor.

The response to these problems is the MSS2 reactor (fig. 1,2) – the icon of cooperation between the Institute for Sustainable Technologies and the Institute of High Pressure Physics.

Fig1. View of the MSS2 reactor.

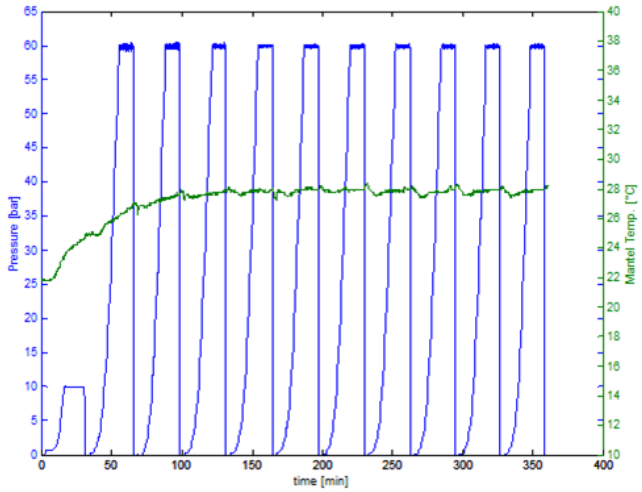

Fig.2. Course of process variables for cyclical recurrence of technological recipe realisation.

The reactor has a unique design of a process chamber, which used in conjunction with a batch control system allows a highly efficient production of nanopowders to be obtained.

The MSS2 reactor and its previous version MSS1 can be used for the scale-up production of nanomaterials characterised by a high commercial potential, and can be applied particularly in electronic, optoelectonic, pharmaceutical, chemical, cosmetic, ceramic and machine industries.

The main areas of development of these devices, and simultaneously, cooperation with scientific partners and commercial companies include the following: improvements of reactors (new measurement systems, an intelligent reactor), design of new reactors (higher pressure, multi closed vessel), design of a technological line, mass production of new materials.

11:00 Poster 30

Microwave Solvothermal Synthesis of Nano Zinc Oxide Nanoparticles

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Laboratory Nanostructures 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:

(CH3CO2) 2Zn → 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 solvo- and 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.

11:00 Poster 31

Microwave conductivity of ZnO:Co and ZnO:Cu thin films with nano-size metallic Co/Cu inclusions.

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ZnO is a very interesting material for range of applications in microelectronic, optoelectronic and photovoltaic devices. For example, it can be used as a transparent conductive oxide (TCO) film in blue light emitters, in UV light sensors and in solar cells [1]. In addition, ZnO doped with transition metal (TM – Co, Cu) ions is intensively studied for spintronics applications [2]. So the research on high-quality (Zn,TM)O alloy systems is becoming fairly important. Several reports suggest the important role of intrinsic defects [3,4], metal accumulations (Co clustering [5]), presence of uncompensated spins at surfaces of Co-rich regions [6], which may be of nm sizes and thus difficult to detect.

The presence of metallic TM inclusions is not visible in standard conductivity measurements, so in the present work we used the microwave conductivity (AC) method. This method is highly sensitive

to such small inclusions. Therefore, AC measurements allow us to investigate uniformity of TM-distribution in (Zn,TM)O films, since the presence of metal inclusions results in large discrepancy between DC and AC conductivity.

In the present paper we demonstrate direct correlation between sample uniformity, Co/Cu concentration, growth parameters and AC conductivity of our films.

The project was financed by the National Science Centre granted based on the number of decision DEC-2013/09/N/ST5/00896.

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11:00 Poster 32

The effect of annealing on properties of Europium doped ZnO nanopowders obtained by a microwave hydrothermal method

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Despite the concentrated research of ZnO, this material still has several new opportunities for biological and medical applications as luminescence markers and drug delivery systems, and optoelectronic applications as a phosphor material.

Several systems are studied for above applications. Lately, interest focused on properties of rare-earth (RE) doped nanoparticles. This relates to their attractive light emission properties – mainly due to observation sharp atomic-like 4f-4f emission lines.

The main limitation for a wider use of RE doped materials is difficulty of 4f-4f excitation

upon a host lattice excitation. This is why 4f-5d or charge transfer excitations are used,

limiting selection of host materials.

In some cases this problem can be avoided by using codoped oxide nanoparticles. In the

present work we report that ZnO nanoparticles doped with selected RE ions, such as: Pr, Tb,Eu and codoped with Al show efficient 4f-4f emission excited upon host excitation. This makes this system very attractive for different above-mentioned applications. Detail results are shown for Eu doped nanoparticles. Based on photoluminescence investigations the chromaticity diagrams are also calculated.

In this work we analyze optical properties of ZnO:10%Eu nanopowders obtained by a hydrothermal method. Effects of annealing on CL and PL spectra is investigated. The observed CL and PL changes are related to the reduction of OH-groups on the surface of annealed samples. Typical Eu 3+ emissions, related to $^5D_0$ to $^7F_1$, $^7F_2$, $^7F_3$ and $^7F_4$, transitions, are detected for ZnO:10%Eu nanopowder only after annealing at 750^{o}C. Chromaticity parameters of the emission are calculated.

The research was partially supported by the National Science Centre project 20/0139/N/ST3/04189

11:00 Poster 33

$Li_4Ti_5O_{12}$/CNT as an anode material for LiBs - structural, morphological and electrochemical studies

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One of the promising material to replace graphitic anodes in lithium secondary batteries is lithium titanium oxide – $Li_4Ti_5O_{12}$ (LTO). It has good structural stability, with an almost negligible volume change during the Li^+ insertion and extraction processes, which suggests theoretically unlimited cycle life. $Li_4Ti_5O_{12}$ features a flat operating potential of about 1.55 V vs. Li/Li$^+$, which is higher than the reduction potential of the most electrolyte solvents – preventing SEI formation and metallic lithium plating on the electrode's surface. These characteristics make that $Li_4Ti_5O_{12}$ is excellent material for anode in lithium-ion batteries with high safety, long life and reliability.

The major disadvantage of $Li_4Ti_5O_{12}$ is its poor rate capability - mainly due to its low electronic conductivity and poor lithium ions diffusivity. The conductivity of lithium-titanium oxide can be greatly improved by various surface modifications, cation doping or preparing this material in the nanocrystalline form. In this work we present the surface modification of LTO using carbon nanotubes (CNT).

The three-step solid state synthesis including ball-milling process of nanocrystalline lithium titanium oxide ($Li_4Ti_5O_{12}$) of spinel structure and surface modification of LTO grains by a new low temperature method will be presented.

All the synthesized materials have been characterized by several methods: XRD, Raman spectroscopy, SEM, TEM. The electrochemical performance of LTO/n-CNT composite powders were examined by chronopotentiometry in three electrode Swagelok cells. The res-

ults of all measurements will be presented at the Conference.

Acknowledgments

This work was supported The National Science Centre through the research grant DEC-2011/03/N/ST5/04389.

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11:00 Poster 34

Novel synthesis of olivine lithium metal phosphates $LiMPO_4$ (M = Fe, Mn, Ni, Co) - analysis and studies.

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It will be presented for the first time the new route of chemical synthesis of lithium metal phosphates - $LiMPO_4$ (where M = Fe, Mn, Co, Ni) of olivine structure. All materials have higher working potential (4 - 5 V vs. Li/Li^+) than commercially used lithium cobalt ($LiCoO_2$) or nickel oxides ($LiNiO_2$) as a positive electrode in lithium-ion batteries. The olivine structure (crystallize in the orthorombic space group Pnma) is stable than structure which represents the layered oxides, due to strong covalent bonds between oxygen ions and P^{5+} resulting in PO_4^{3-} tetrahedral polyanions. The so-obtained powders were characterized by several complementary methods, including structural, morphological and electrochemical studies.

11:00 Poster 35

The influence of CeO_2 on electrochemical performance of LiMn2O4.

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This work is focused on analysis of the influence of cerium dioxide (CeO_2) on electrochemical performance of lithium manganese oxide ($LiMn_2O_4$, LMO). Firstly, pure stoichiometric nanopowder of $LiMn2O_4$ was prepared by modified sol-gel method using lithium and manganese salts. Secondly, the low temperature method (LTM) was used to modify the grain surface of pristine lithium manganese oxide powder. The LMO/n-CeO_2 powders were analyzed by: XRD, Raman spectroscopy, SEM, TEM. The galvanostatic charge/discharge tests of the pristine and CeO_2 modified $LiMn_2O_4$ cathode materials were conducted in the potential range: 3.5 to 4.5 V vs. Li/Li^+ at room temperature. High current rates performance has been evaluated by determination of specific discharge capacity at current rates varied from 1 C to 30 C. Our work demonstrates that surface modification of LMO grains' by using 1%wt. of CeO_2 admixture improves cycling stability and capacity retention. Pristine LMO reveals 10% capacity loss after 100 cycles when discharged at 1C, while the sample modified with 1%wt. CeO_2 grains retains ca. 98% of its initial discharge capacity after 100 cycles.

Acknowledgments

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11:00 Poster 36

Kinetic studies of 4-chlorophenol adsorption on the reduced graphene oxides

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The graphene oxide was prepared by a modified Hummers method [1]. Two samples so obtained graphene oxide were chemically reduced by HI or HBr. To characterize the reduced graphene oxides, designated as rGO_{HI} and rGO_{HBr}, the nitrogen adsorption/desorption isotherms at 77.4 K, the total oxygen content, Raman spectra as well as SEM images were determined. The specific surface area (S_{BET}) and total oxygen content were 65 m^2/g and 13% for rGO_{HI}, and 35 m^2/g and 20% for rGO_{HBr}, respectively, which indicates that the type of reductant used significantly affect the properties of the resulting materials.

The adsorption properties of these materials were tested in aqueous solutions with respect to 4-chlorophenol. The adsorption equilibriums were achieved after about 60 min for the rGO_{HI} and after about 90 min for the rGO_{HBr}. For comparison, the adsorption kinetics of the 4-chlorophenol on the most commonly used adsorbents – activated carbons, was reached after approximately 6 hours [2,3]. The kinetics data were fitted well to the pseudo-second order model with the coefficient of determination (R^2) values greater than 0.99. The rate constants k_2 obtained for rGO_{HI} and rGO_{HBr} were 0.731 and 0.537 g/mmol·min, respectively. The potential use of these materials for the preparation of the SPME fibers was also investigated. The results showed that the reduced graphene oxides are promising materials for microextraction of organic contaminants from water.

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11:00 Poster 37

Anisotropy of selected properties of Al_2O_3-graphene composite

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Alumina powder (Al2O3-α, grade A16SG, 0.3-0.6 μm, ALCOA) with addition of 0.3 wt% of MgO nanopowder (Inframat) has been used as a starting material. Multilayer graphene nanoplatelets (average flake thickness <4 nm , <4 monolayers), average particle (lateral) size 1-2 μm, Cheap Tubes, USA), were used as filler for alumina based composites. The composites were sintered using Spark Plasma Sintering – SPS at 1550 °C during 10 min under 35 MPa of uniaxial pressure during the whole cycle. Sintered specimens were disk-shaped with dimensions of 20 mm in diameter and ~5 mm in thickness. Graphene participation was 0.5wt%, 1wt% and 2wt%. Selected physical and mechanical properties like: Vickers hardness with 9.8 N load, Young's modulus, Poisson's ratio measured by means of ultrasonic method, coefficient of friction for contact with a Al_2O_3 ceramic ball in ball-on-disc test and density were determined. Modulus of elasticity (Young's modulus) of the tested composites was determined by measuring the transmission velocity of longitudinal and transversal ultrasonic waves through the sample. The SEM and TEM microstructural analysis show that the applied pressure during the sintering process (SPS) leads to the orientation of the graphene phase and in consequence to anisotropy of the composite. For example, for a composite with 2 wt% graphene , the hardness HV1 in the direction of the pressing axis is 15.5±0.2, and for the perpendicular direction to the pressing axis is 16.8±0.3. The microstructure, mechanical and tribological properties of the graphene reinforced Al_2O_3 ceramic, taking into account the direction of the measurement, are presented.

11:00 Poster 38

Carbon nanotube modified aqueous commercial paints for electroconductive coats

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Especially prepared aqueous dispersion of carbon nanotubes (CNT) was introduced into commercially available paints/varnishes in small amounts (0.25-2.5 wt.% of carbon in dry mass of coat) to improve electrical, mechanical and thermal properties of final dry coats. Falling prices of CNT (<100 EUR/kg) make them attractive additive for existing commercial polymer materials. The addition of CNT in mentioned amounts to the investigated coating compositions allows

to prepare coats with electroconductive (10^2-10^4 Ohm) or antistatic (10^5-10^9 Ohm) properties. The modification of commercial water-borne coating materials using CNT allows to improve their hardness, thermal stability and does not change their glass transition temperatures.

11:00 Poster 39

Production of graphene and nanocomposites of metal / graphene

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The Institute of Precision Mechanics developed CarboTermoFluid® technology for producing graphene on the grains of powder which is called Graphene 3D^{IMP}. This technology can produce graphene on powders of metals and non-metals of different geometry and size of powder grains. Graphene formed on the copper powders can be used to manufacture: electrical contacts with high thermal and electrical conductivities, transmission cables, heat exchangers, radiators, bearings, lubricants, and pastes. Identification of the formed graphene 3D^{IMP} was performed by using Raman spectroscopy and scanning electron microscopy (SEM). Image of Cu grain coated with graphene 3D^{IMP} and Raman spectrum of graphene are shown in Fig. 1 and Fig. 2, respectively.

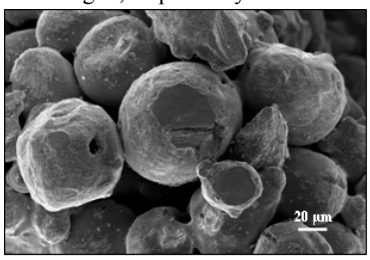

Fig. 1. Cu grain coated with graphene 3D^{IMP}

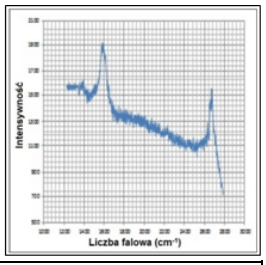

Fig. 2 Raman spectrum of Graphene 3D^{IMP}

In parallel, in the Institute of Precision Mechanics are manufactured metal/graphene nanocomposite layers by chemical and electrochemical reductions. The nanocomposite layers of Ni-P/graphene, Ni/graphene, Cu/graphene are characterized by nanocrystalline and compact structures as well as good adhesion to the substrate. The structure and properties of the metal/graphene nanocomposite layers were investigated by: scanning electron microscopy, EDS analysis, optical microscopy and the measurement of HV microhardness. Inclusion of graphene into a metal matrix significantly improved thermal and electrical conductivities as well as mechanical properties of such layers. SEM images of graphene flakes and of layer surface of Ni-P/graphene composite are shown in Fig. 3 and Fig. 4, respectively.

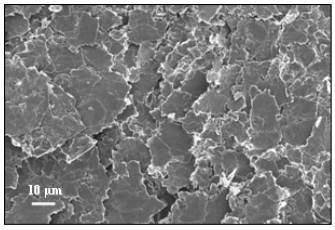

Fig. 3. SEM image of graphene flakes

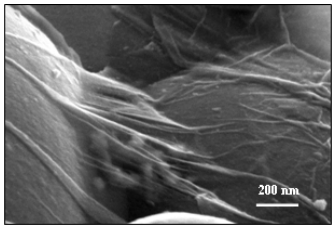

Fig. 4. Surface of the Ni-P/graphene composite layer

11:00 Poster 40

Thermal stability of SiC nanowires and their resistance to mechanical stress during after-synthesis material treatment

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Silicon carbide nanowires (SiCNWs) exhibit some unique mechanical, electrical and optical properties. Potential applications include nanosensors. blue or UV-light emitting diodes or electron emitters. They can be used as reinforcing phase in composites. While for composite reinforcement the material with no specific parameters is usually needed, electronic and optical applications require precisely purified, debundled and fractionated SiCNWs.

We investigated SiCNWs obtained by Self-propagating High-temperature Synthesis (SHS) from micrometric Si and PTFE powders. The products of such reaction include not just nanowires but also bulk SiC crystals as well as unreacted carbon and silicon. Therefore some additional preparation steps are required to obtain pure and shape-controlled SiCNWs.

SiC is known to possess excellent thermal and mechanical resistance. It was expected that mechanical treatment of SiCNWs would assist in debundling of the nanowires to allow their fractioning while annealing in the presence of oxygen would cause a controlled surface oxidation and allow for decreasing of the SiCNWs lateral size. Surprisingly SiCNWs were found to be extremely sensitive to thermal energy and mechanical stresses. Here we report on some unexpected and specific transformations that SiCNWs undergo during thermal and mechanical treatment which was applied to purify and debundle the material.

Initial thermal treatment of SiCNWs was condicted at 750ºC in air in order to burn-out free carbon and oxidise free silicon. It was followed by boiling NaOH water solution in order to remove silica. The treatment produced pure SiC material consisting of long entangled nanowires and a considerable fraction of bulky crystallites.

Rubbing and milling of SiCNWs was though to be an effective way of nanowires debundling and shortening. The material was rubbed in a mortar and / or milled in a planetary ball mill. Depending on rubbing / milling conditions, the material recrystallized partially or totally into non-fibrous bulk of irregular SiC particles. The most destructive conditions for SiC nanowires is set up when the material is shortly milled in a planetary ball mill without any liquid media – all the wires transform into irregular SiC particles. Rubbing and milling

are therefore not safe for SiC nanowires.

Annealing in the presence of oxygen was expected to cause limited surface oxidation of SiCNWs and possibly breaking them into shorter fragments. Annealing was conducted at 1000, 1100 and 1200ºC for 10 minutes in air. It was found that above 1000ºC the nanowires get converted into micrometric spherical entities composed of multiple SiC nanocrystals. Most probably the nanowires get dissolved in droplets of molten silica and recrystallize in a form of bulk nanocrystals.

The the present study indicates that silicon carbide nanowires are metastable objects which tend to change their morphology under conditions at which SiC is usually believed to be stable. Any harsh post-synthesis treatment appears to be destructive for them and the key to obtaining the of the SiCNWs of desired morphology lies in the synthesis process itself.

11:00	Poster	41

A novel green route for the fast combustion synthesis of silicon carbide nanofibers

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It is well known that nanomaterials offer many unique and variety properties arousing great interest of researchers and give hope to the many potential applications. Silicon carbide also belongs to this type of material [1-4]. In particular, the 1-D dimensional form (nanofibers, nanowires, nanotubes) is most promising, mainly for a good electrical and mechanical properties [5-8]. There have been many attempts to produce a 1-D SiC, for example:

- synthesis from the elements [9,10];
- carbothermic reduction [11];
- chemical vapor deposition [12];
- arc, microwave or induction discharge [13];
- NRW use as template for the growth [14];
- laser ablation [15];
- "hot filaments" method [16].

The above-mentioned methods and techniques of production of 1-D SiC are, however, multi-step approaches while energy and time-consuming factors makes them at the same time unattractive in the process scale-up and industrial use. Thus, the combustion synthesis (CS), which is a new way to obtain silicon carbide nanofibres is economically justified and privileged.

Combustion synthesis is a well-known method [17] for the preparation of useful compounds of carbon, such as nano SiC, activated carbon, [18] and magnetic carbon encapsulated [19]. The high temperature and pressure of very fast redox reaction provide opportunities to process materials with a high melting point. Furthermore, these reactions are thermally autogenous and usually lead to the formation of nano-sized products. Basically, there is no restriction on the choice of appropriate reagents, it is important, however, to connect a strong oxidizer and reducer [20]. From the past experience it can be concluded that by careful selection of starting reactants the combustion leads to the desired product, but in most cases the mechanism of the process is still not clearly defined. In addition, the proposed synthesis method is autothermal, which means that after the initiation the reaction is carried out without energy needs. This makes such processes economically very attractive.

The aim of this study is to demonstrate that SiC nanofibers can be one-pot produced in fast and single step synthesis using waste materials as substrates and to optimize the conditions in order to maximize the main product - nanofibers SiC.

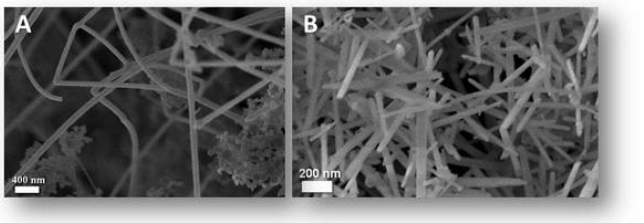

Fig. 1. Fragments of discarded or waste silicon solar panels (A); the experimental setup after the process (Si from waste panels and waste PTFE powder, 1 MPa, air atmosphere) (B)

Lowering the cost of synthesis of nanomaterials is extremely important for the further development of nanotechnology, which is often limited by the high price of their precursors. So far in the process of nanofibers SiC production we used pure commercial reagents, which are relatively expensive. Thus, the attempt to use cheap or waste reactants is obviously justified. While there have been many attempts to re-use silicon from discarded or waste silicon solar panels there are currently, however, no rational methods for their disposal. Obviously, in the near future this problem can become a major barrier to the development of photovoltaics [21]. We also carried out the exploratory experiments using domestic waste technical PTFE (Teflon) powder.

To carry out successful syntheses we used silicon material resulting from grinding waste photovoltaic panels. The powder mixture is prepared from the stoichiometric amount of the reactants: 36% wt. Si and 64% wt. of PTFE. The synthesis was performed in an air atmosphere (initial pressure of 1 MPa) in the system presented in Fig. 1. [22]. The obtained products were almost identical to the ones obtained in previous tests with the commercial reactants. The have a 'spongy' morphology, demonstrating a significant content of fibrous structures. 'The microscopic observation confirmed the presence of SiC nanofibers (SEM - Fig. 2.). Further investigation in search of the most favorable process conditions were carried out in CO and CO2 atmosphere. We analyzed the morphology and composition of the reaction products from all tests, including the diameter of nanofibers obtained. The performed CS was monitored opto-electronically to efficiently track the progress of the reaction and to evaluate the velocity of propagation of a combustion wave.

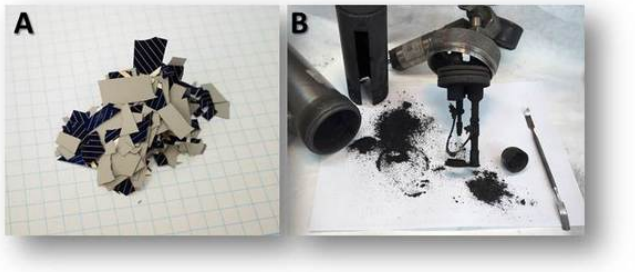

Fig.2. Silicon carbide nanofibers produced during combustion synthesis (starting reactants: silicon from waste solar panels and waste PTFE powder, 1 MPa, CO2 atmosphere), (A) raw products, (B) products after purification

The performed studies indicate that silicon from the ground silicon solar panels efficiently reacts during the combustion synthesis with waste PTFE, and the reaction products contain SiC. Thus, a substantial reduction in the cost of the starting reactants is achieved. A scale-up of the CS will be a further step in minimizing the costs associated with the synthesis. The production of a larger amount of material will be extremely important for consideration of different applications. Thus, due to the low time-and energy-consumption, and simple design of the reactor the products can be produced in more than gram quantities.

The research has been supported by the NCN grant No. UMO-2011/03/B/ST5/03256 and UMO-2012/05/B/ST5/00709.

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11:00 Poster 43

A Polythiophene derivative with pendant viologen: In situ ESR/UV-Vis-NIR spectroelectrochemical study

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Recently, considerable research has been developed in modifying the electronic properties of conducting polymers mainly by attaching specific functional groups to the polymeric chain, so called third generation of polymers. These efforts can result in additional redox activity, modified electronic properties, molecular recognition or electrochromism. Viologen is such a functional group which can give additional redox properties to the polymeric backbone, besides the electrical conductivity. The chemical or electrochemical reduction of cyanopyridines to viologens is well known. In this work, we have synthesized a thiophene monomer bearing a cyanopyridine moiety in its side chain. Its electrochemical polymerization results in a polythiophene film highly cross-linked by the viologen unit. In order to improve adhesion and insolubility of the polymer film, we choose ionic liquid as an electrolyte for electropolymerization. The resulting polymer film shows electrochemical activity from both viologen and polythiophene moieties. *In-situ* spectroelectrochemical studies of the polymer shows an improved multicolored electrochromism with coloring characteristics both of the viologen and the polythiophene backbone.

11:00 Poster 44

Wear behaviour 3D AFM roughness of sintered nanomaterials produced by hot isostatic pressing (HIP)

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We propose an experimental study of the surface to dry and analysis of the evolution parameters roughness. The simplified model was proposed to predict the metrological parameters in the contact area of the deformed surface. The model is based on the analysis of the topography 3D of the deformed surface. The hot isostatic pressing (HIP) is the only process that will develop fully dense samples, From Fe,Cr,Mo,Ni,Ti,W powder. This sample is hot pressed at 1500°C under 150Mpa of argon pressure. Moreover, the grain size of the consolidated samples was analyzed by SEM, ABSD and optical microscopy. Study aims to characterize the topography of sintered materials obtained by wear tests. Therefore it is interesting initially in the evolution of wear for the loads applied and to characterize the different roughness emerging from 3D AFM observations. Experimental and theoretical research on the topography changes during dry contact deformation was carried out, providing results that demonstrate the persistent nature of roughness asperities even

under high loading when bulk plastic deformation appears. Most theoretical investigations of the problem have been based on a simplified model neglecting the statistical distribution of asperities on the real surface. The test used and the 3D measurement of the surface topography to study the friction behavior. The mechanism of contact of a rigid plane with a rough surface in the presence of a lubricant is different than in the case of dry contact. The topography of the samples was measured both in initial undeformed and in the deformed state after removal of the load. In these states, however, a change of the shape of the samples when compared to the initial state was observed. Thus, prior to the determination of roughness parameters of the deformed surfaces, their curvature was removed using a filtration procedure. The essential differences in surface topography of samples loaded in dry condition are confirmed in the analysis of roughness parameter evolution. The following 3D parameters were considered: In the unloaded state, flattened asperities can be observed on the deformed surface Fig 1.

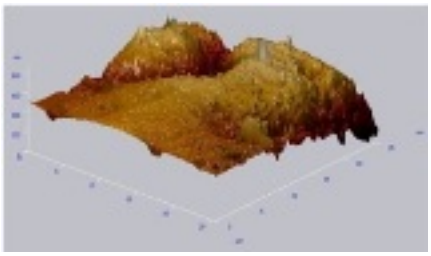

Figure 1: measurement 3D of surface topography in order to investigate its frictional behaviour.

The real contact area corresponding to the maximal load attained in the surface compression experiment can be identified from measurement of the deformed roughness after unloading. The identification of the real contact area was carried out using a special algorithm based on single profile analysis. The single, randomly selected profiles, i.e., their coordinate's xi, zi, were extracted from the measured topography of the deformed surface. It should be noted that the profiles obtained in this way have a common reference level Fig 2. The selected profiles also have the same direction, which, in the case of anisotropic surfaces (turning, grinding) should be perpendicular to the direction of the movement of the machining tool.

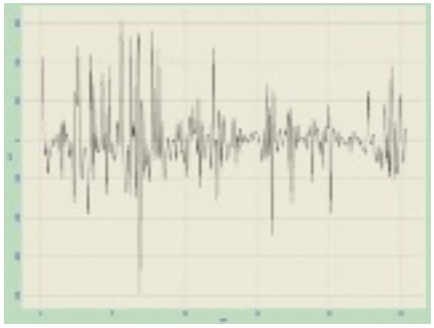

Figure 2: Profile corresponding to real area

The proposed model was applied to analyze a wear of four kinds of rough surfaces. The predicted values were compared with experimental results Table 1.

Table 1*: Roughness parameters of the deformed surfaces of the samples figure 1.*

Max	Min	Peak-to-peak, Ry	Ten point height, Rz
577,339 nm	430,073 nm	147,266 nm	506,714 nm
Average	Average Roughness, Ra	Root-mean-square, Rq	Dispersion
510,674 nm	28,7048 nm	511,831	34,3946 nm
Surface skewness, Rsk	Coefficient of kurtosis, Rka	Entropy	Redundance
-0,157453	-0,696865	7,88768	-0,0936811
-0,157453	-0,696865	7,88768	-0,0936811

The wear and surface roughness based on the parameters of dry friction tests were measured. This study suggested the optimal parameters of chemical composition, and analysis of the effects of alloying elements on surface roughness and wear in the process dry friction tests.

11:00 Poster 45

Multifunctional coatings of niobium carbide (NbC) for industrial applications

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1. *Adam Mickiewicz University, NanoBioMedical Centre (CNBM), Umultowska 85, Poznań 61-614, Poland* **2.** *CIC biomaGUNE (BIOMAGUNE), Edificio Empresarial C Paseo Miramón 18, San Sebastian 20009, Spain* **3.** *CIC nanoGUNE, Avenida de Tolosa 76, San Sebastian 20018, Spain* **4.** *Centro de Investigacion en Quimica Aplicada, Blvd. Enrique Reyna Hermosillo, Saltillo 33456, Mexico*

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Nb-C nanocomposite thin films have been traditionally deposited at high temperatures and applied bias voltage, based on idea that crystallinity and nanohardness were directly related to such parameters. In our study, we have deposited for the first time, nanocomposite thin films of Nb-C, with carbon content ranging from 0 to ~100 at.%. Films were deposited at room temperature by non-reactive magnetron sputtering from pure Nb and C targets without applying bias voltage or additional temperature to the substrates. Samples show a maximum hardness of 23 GPa for the film with ~8-10% percent of the amorphous phase. The same film showed a four-point probe electrical conductivity of $2.2x10^6$ S/m at 22ºC and superelastic behavior, with more than 80% of elastic recovery. The fact that the substrates do not need to be heated to obtain relatively hard, elastic and electrically conductive nanocomposite structures opens the possibility for a wide range of applications for temperature sensitive flexible substrates such as polymers and plastics. Biocompatibility studies of the NbC films and corrosion studies show strong cellular

biocompatibility and corrosion resistant properties.

ACKNOWLEDGMENTS

L.E. Coy thanks the financial support from the National Cen-tre for Research and Development under research grant "Nanomaterials and their application to biomedicine", contract number PBS1/A9/13/2012. We thank Layza A. Arizmendi and Gilberto Hurtado at CIQA for assistance with TEM imaging and for electrical measurements.

11:00 Poster 46

Analysis of electro-optical parameters of InAs/GaSb superlattice infrared detectors

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High requirements set upon infrared photodetectors stimulate the development of both more sophisticated materials and device structures. An example of the former is type-II broken gap InAs/GaSb superlattice proposed for the first time by Esaki, which is promising for infrared sensing systems. Mainly, due to its electro-optical properties, especially absorption edge, which can be easily tunable by changing the thickness of InAs and/or GaSb layers constituting a single period of superlattice. Unfortunately due to their complex nature none of the commercially available software packages are able to properly simulate both heterostructures and devices based on this material system.

In this paper various numerical methods are combined to determine parameters of photodiodes as close to experimental ones as possible. At first, the tight binding algorithm in the sps* basis was implemented to calculate the energy dispersion as a function of the k vector. Spin-orbit coupling was then introduced, which allowed a significant improvement in precision of determined effective heavy and light hole masses. Additional excited s* state allowed for better adjustments of conduction band especially in the case of high symmetry points. Parameters obtained by this method were used as input data in further numerical analysis of type-II InAs/GaSb superlattice photodetectors.

Additionally analysis of dark current of a Barrier Infrared Detector (BIRD) type device was performed. Its diffusion and generation-recombination components were considered as well as band-to-band and trap-assisted tunneling ones. The former dominate in high temperature operating regime. The latter are directly related to narrow bandgap of absorber material and relatively high electric field across depletion region. Finally, a numerical model of dark current was discussed, and theoretical results were compared with those obtained from experiment. Uncertainties were addressed and possible directions of improvement proposed.

11:00 Poster 47

Experimental evaluation of melting point depression in AlSi/AlN nanomultilayer system

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The aim of the study was to evaluate the structural changes in AlSi/AlN nanomultilayers (NML) upon annealing for low temperature joining applications. It is known that due to relatively high thermal sensitivity of nanometals, standard methods of joining, e.g. furnace brazing or welding, are not suitable as they cause e.g. grain coarsening. To overcome the limitations of conventional brazing technologies based on a bulk eutectic alloy approach, proposed research strategy aims to employ the size-dependent melting behaviors of metals confined in a nanostructured multilayer geometry. The use of such a nanoarchitectured configuration can reduce the processing temperature, thus allowing benign joining of heat sensitive nanometals. Nevertheless, before the step for application the basic understanding of the melting point depression occurred in such systems, its significance and possible mechanisms need to be deeply investigated, which is the main goal of this study.

The system investigated was produced by magnetron sputtering and consisted of Al-Si(12%) braze filler metal layers (bulk Tm=577°C) with a thickness of 4 nm alternated by aluminium nitride diffusion barrier layers with a constant thickness of 3 nm. The bilayer of AlN/AlSi was repeated 10 times on the Si substrate and covered with the final AlN layer on the top. In order to investigate the melting behavior, the system was heat treated at various temperatures and observed with the use of Scanning Electron Microscope (SEM). Further, the cross section FIB-lamellae of the as-deposited and the heat treated state directly under the droplet were cut and examined using a Scanning Transmission Electron Microscope (STEM) and Transmission Electron Microscope (TEM).

The TEM observations of the as-deposited state showed that the AlSi/AlN system consists of policrystalline layers of AlN and AlSi, where the layers possess very fine structure with the grain sizes similar to the corresponding film thicknesses. The chemical analysis using EDX revealed a homogenous distribution of both Al and Si throughout the AlSi layers, which suggests that the structure is single phase. The annealing at 300°C did not cause any significant changes in the multilayer structure, while the annealing at 400°C brought about a visible phase separation within the Al-Si layers, where the supersaturated solid solution transformed into Al reach and Si reach regions.

The SEM observation of metal-containing features created on the

top of the nanomultilayer revealed their first appearance at a temperature of 400°C. The droplets had irregular shape and were randomly distributed on the surface. The chemical analysis on the cross section of a droplet showed that it consisted of Si and Al indicating that the droplets formed as a result of liquid metal outflow from the nanomultilayer. The STEM observations of the central region under the droplet revealed that the NML was partially damaged and became thinner. Some of the AlN layers were deformed and created bumps due to the accumulation of the AlSi alloy under them, while others were broken and removed and their fragments were embedded in a solidified AlSi droplet above the system.

Presented results demonstrate that the melting point depression in AlSi/AlN NML system reached the value of 177°C. The mechanism responsible for melting at depressed temperature consisted of two stages. In the first stage, at a temperature of 400°C phase separation within the AlSi layers occured. This led to the creation of privileged sites at the interface between AlSi and AlN layers, where the liquid Al-Si alloy was cumulated. In the second stage, the upper AlN layers became bent and beyond their bending strength, AlN layers started to deform and break, which allowed liquid metal to freely outflow and create a two-phase flower-like structures on the NML surface.

11:00	Poster	48

Joining ultrafine grained aluminium by friction stir welding - processing, microstructure and mechanical properties

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Nowadays different processes are used to improve mechanical properties of materials. In metallic materials, grain size refinement down to nanoscale is one of the most efficient strengthening mechanisms, as predicted by Hall-Petch relationship. Such microstructure refinement can be obtained in several ways, among others by severe plastic deformation (SPD). Although a tremendous progress has been made in the development of SPD methods, the main drawback is the restriction in billet dimensions. The most common shape – rods, are manufacturing with diameter about few or sometimes over a dozen millimetres. Incremental ECAP is a novel tool to manufacture plates with ultrafine grained structure. Possible sizes of plates are promising for future applications, e.g. in automotive industry. Another issue related to ultrafine grained materials is joining without losing their properties governed by the nanoscale structure. Traditional methods cause grain coarsening which is highly unwanted.

In this work plates from Al 1050 after Incremental ECAP were joined using Friction Stir Welding. The quality of joints was determined using microscopic observations. Also, the structure of joints and base materials was investigated by light microscopy and transmission electron microscopy. Mechanical properties were measured by microhardness and tensile tests. To investigate mechanical properties like yield strength and tensile strength mini samples were used. Samples were separately cut from the joints and initial materials as well. It allowed to investigate the differences in both areas.

The results revealed that joints zone are characterized by lower values of microhardness and tensile properties compared to base materials. Structure investigation showed changes in grain sizes caused by joining process.

11:00	Poster	49

Microstructured surfaces and their practical applications in heat exchange devices

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The paper involves developing practical aspects of the using enhanced surfaces in heat transfer research when two types of devices are applied. The first one is the heat exchanger with the minichannel furnished with enhanced heating surface. The second type includes prototype solar collectors with the developed surface of absorber's pipes or smooth pipes covered by the absorber plate, with an external developed surface. The author's Polish patent No. A1 396579 *"A structure for boiling heat transfer enhancement"* was used in described heat transfer devices construction.

Heat transfer in small channels has been studied intensively over the last few years, especially as regards the application in cooling electronic components. Mini heat exchangers are used to provide higher cooling capability for new technologies. It means reducing their sizes and costs, while the consumed power is identical. Owing to the change of the state which accompanies flow boiling in small channels, it is feasible to meet contradictory needs simultaneously, i.e. obtain a heat flux as large as possible at small temperature difference between the heating surface and the saturated liquid while retaining small dimensions of heat transfer systems. The use of microstructured surfaces allows additional intensification of the process. The series of studies pursued at the Kielce University of Technology includes research on flow boiling heat transfer in a cooling fluid flow along the minichannel with plain or microstructured heating wall and various orientations. The results were described in numerous publications.

The essential part of the experimental stand is the test section with a rectangular minichannel 1 mm deep, 40 mm wide and 360 mm long. The heating element for FC-72 flowing in the minichannel was the thin alloy foil designated as Haynes-230. There is a microstructure on the side of the foil which comes into contact with fluid in the channel. Two types of microstructured heating surfaces: one with micro-recesses distributed evenly, and another with mini-recesses distributed unevenly, were used. The micro-recesses were performed by laser drilling. The diameter of the single micro-recess is usually 10 mm, its depth is 3 mm. 5÷7 mm high layers of melted metal deposit annularly around the recesses, forming structures that can be named as "craters". Micro-recesses are evenly distributed every 100

mm in both axes. The mini-recesses were obtained by spark erosion. The melted metal foil and electrode material, a few mm high, reaching locally 5 mm, accumulate around the recesses. The depth of the cavity craters is usually below 1 mm. It is possible to observe both surfaces of the channel through glass panes. One pane allows observing changes in the temperature distribution on the plain side of the foil thanks to the liquid crystal thermography. The latter one allows observing the two-phase flow patterns on the microstructured foil side. The final results are presented as local heat transfer coefficients.

The observations have confirmed experimentally that boiling incipience occurs in lower heat flux supplied to the enhanced foil which constitutes a heating surface of the minichannel in comparison to results from the studies on similar minichannels employing the plain foil. Thus, the heating surfaces with the proposed arrangement of recesses make it possible to provide a large number of nucleation sites. It was found that prototype solar collectors with recesses formed on the surface of the absorber's pipes have higher energy efficiency in comparison with the collectors with plain absorbers. To sum up, the analysis of all results of the discussed studies indicates that the application of enhanced surfaces allows achieving effective heat transfer.

11:00 Poster 50

Modification of the carbon nanotubes by electroless deposition of Ni-P for anisotropic composites manufacturing

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The motivation of the studies was to investigate the relationship between the properties of carbon nanotubes CNTs and their modification with metallic coating on the possibility of obtaining improved epoxy resins with aligned nanotubes for the exploitation of their enhanced electrical and mechanical properties. In order to obtain improved directional conductivity we tried to arrange nanotubes in polymer matrix using magnetic field.

Magnetic properties of nanotubes were modified by ferromagnetic layer of Ni-P. Carbon nanotubes surface was prepared for Ni-P electroless deposition. Multiwall nanotubes CN3100 from Nanocyl® were used. The Ni-P coating on the purified CNTs grew along the nanotubes while on the untreated CNTs grains of Ni-P formed agglomerates at the surface. Depending on the process conditions, nickel content varied in the range of 30-60%wt. The solution reaction (pH=8.5) and the basic composition of the solution led to the deposition of Ni-P coatings with a low phosphorus content of 1.5-3.0%wt..

11:00 Poster 51

Application of Hot Isostatic Pressing in densification of Nd:YAG ($Nd_{0.03}Y_{1.97}Al_5O_{12}$) nanopowder

Magdalena E. Gizowska, Krzysztof Perkowski, Marcin N. Osuchowski, Irena K. Witosławska, Izabela Kobus, Gustaw Konopka, Paulina Tymowicz-Grzyb, Adam Witek

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Yttrium aluminum garnet doped with neodymium (Nd:YAG) is a commonly used material for solid state lasers. However, neodymium doped yttrium-aluminum garnet is mainly fabricated by melt-growth process. Polycrystalline YAG materials obtained by ceramic techniques path are an attractive alternative to the single crystal. YAG ceramics offer many advantages, including easy preparation, scalability to large size. Additionally, ceramic technology makes it easier to incorporate several dopant ions into the YAG structure compared to single crystal grown from the melt.

Nano PL 2014, Symposium B

Programme

Wednesday, 15 October

POSTER

Poster's fixing to be displayed 3 days
Wednesday morning, 15 October, 11:00

Joining Symposium A events
Wednesday morning, 15 October, 11:30

Joint poster session with Symp. A
Wednesday afternoon, 15 October, 17:40

Thursday, 16 October

REGISTRATION
Thursday morning, 16 October, 8:30

Joining symp. A activities
Thursday morning, 16 October, 9:15

Joint poster session with symp. A
Thursday afternoon, 16 October, 16:30

Friday, 17 October

REGISTRATION and Poster Fixing
Friday morning, 17 October, 8:00

Session 9

Nanotechnology and bio-nanotechnology for society
Friday morning, 17 October, 9:00
Chair: Robert Bucki

9:00 Oral

Opening of Nano-Bio-Med session

Witold Łojkowski

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9:05 Keynote

How nanotechnology already affects daily life

Alex Dommann

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Nanotechnology allows to develop new materials, additives and coatings with tailor-made functional capabilities to solve a large number of existing technological problems. Empa has established a broad field of nanotechnologies with applications-oriented exploitations of nanoscale effects for different kind of applications like building technologies, textile industry and heath applications. The risks for mankind and the environment involving this key technology are also being studied at Empa.

9:35 Keynote

Solutions and problems in the diagnosis and treatment of cancer

Artur Kowalik

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Each year 8 million people die from cancer and it is predicted that 13.2 million patients will die in 2030. 90% of deaths caused by systemic disease. Systemic cancer spread means formation of metastasis where secondary tumours arise from cells originating from the primary tumour, and these include circulating cancer cells (CTC). Thus, disseminated cells are the subject of numerous studies and abiding interest of pharmaceutical and biotechnological companies.

Currently, diagnosis of cancers is performed in an invasive way. Obtaining diagnostic material is carried out with painful biopsy. Sometimes amount of the material thus obtained is insufficient for the complete diagnosis. These situations necessitates resampling material from the patient. In addition, radiological methods currently used are very expensive and inaccurate. It follows that the need is to search for new and more sensitive diagnostics and monitoring based on non-invasive or vanishingly invasive example for example based on small blood sample. This gives the possibility of regular monitoring of treatment and improve quality of patients life. Compliance with these requirements is necessary to conduct a personalized treatment of cancer.

High hopes are associated with nanotechnology and techniques lab-on-chip to solve the above problems. Currently, advanced work in the use of nanotechnology in the diagnosis and treatment of cancer resulting in the first drug doxorubicin containing liposome. This formula increases the efficacy of the drug while reducing side effects.

At present the nanomedicine cabinet consist of wide array of constructs: graphene oxide, quantum dots, gold nanoparticles, silica, liposomes, DNA nanotrains and many many more.

Using nanotechnology we can solve many existing problems concerning caner treatment and diagnostics described above. Americans a few years earlier saw the contact force of the physical sciences to the biology of the cancer cell giving rise at the National Institutes of Health 12 multidisciplinary research teams http://physics.cancer.gov/. Holycross Cancer Center currently established a multidisciplinary team involved in the projects in various fields of technical and biomedical sciences. We have close cooperation with leading technical and physical institutes from Poland. Together we are working on the problems connected with enrichment and detection of circulating tumor cells and usage of nanoparticles to cancer detection and treatment.

We are open and welcome cooperation in research field of cancer treatment and diagnostics.

9:55 Invited Oral

Liquid Nanometals Dispersions For Polymers Modification and Biocatalist of Microbial Fuel Cells

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Application of Nano Metals additives for modification of end products to upgrade their consumer's properties is the main trend now in multiple industrial fields. Despite substantial concerns about their ecological integration the Nano Metals additives were widely used for upgrading antibacterial properties of medical equipment and disposable materials treatment, self cleaning paints and plastics for contact surfaces, antibacterial sport and military clothing, food packaging and processing, antifouling treatment of underwater ship parts, upgrading of electro conductive properties of electronic ink and dozen of other industrial applications. The Nanometals additives are the largest group of whole nanomaterials market which was conservatively estimated to grow from 31,574 tons in 2010 to 44, 267 tons in 2016 [1].The most spectacular market volume grow was demonstrated for Nano Silver applications, specifically in the field of food packaging: from $410 mln in 2010 to $5800 mln in 2012 due to substantial food shelf life prolongation via upgrading of packaging antibacterial properties[2].

Most industrially produced metal nanoparticles are dry powders, so they require sequential processes of colloid chemistry to convert them into liquid solutions: wetting by carrier liquids, breaking agglomerates by different techniques and additional chemical for stabilizing them in suspension. To solve this problem the researchers worldwide are working on new technologies of cheap nanoadditive mass production, mostly on liquid-based dispersion or colloidal solutions, which may be added by simple mixing to consumer end products without radical change of their traditional manufacturing processes. The stability of the active non organic nano particles and the method of their delivery (nano metals payload) to the end products are the key problems to be resolved for nanoadditives wide practical application. That is why the "muster-butch" approach - the way of encapsulation of the active ingredients (metal nanoparticles) in concentrated form in liquid media, storing them in stable condition and then mixing with end product– is the most perspective way of wide commercialization of metal nanoparticles.

The Joint Venture "Marketing of Superhard Materials" – spin-off company from V.Bakul Superhard Materials Institute NAS Ukraine – is introducing a new combined "dry-wet" technology of stable concentrated metal nanoparticles dispersions ("muster-butches") manufacturing by physical plasma dispersion in vacuum with one-step technological cycle of implanting them in various liquid media placed to the same vacuum chamber. Core competence of proposed technology is manufacturing of effective highly concentrated nano dispersions of multiple metals as Ag, Au, Pt, Pd, Fe, Cu etc. in various carrier liquids which are the most common ingredients of the modern household or technical end products. So, these liquid nano dispersions can be easily mixed with end products without changing of traditional manufacturing processes for upgrading their functional properties.

As hundred of end products consist food Glycerin as technological ingredient, the concentrated nanometals additives in Glycerin were developed. One of these nano additives is a new product "Silver Shield-1000". It is the concentrated (100 mg/liter) stable muster–butch of Ultra clean Nano Silver particles with controlled size distribution 25-50 nm (about 70% of nano particles). The semi-industrial pilot unit with the productivity of 6 ton per year (one shift regimen) is capable to supply the customers with substantial volume of concentrated nano Silver dispersion. The physical-chemical properties of nano metals dispersions (master-butches) are well controlled and repeatable, which is supported by multiple studies of independent laboratories in USA, Germany, China and Israel. The stability of concentrated nanometal dispersions is proved for not less then 12 months. The whole process of manufacturing is provided in vacuum chamber and has no pollutions to environment. The metal nano particles are ultra clean and have no oxidation due to implantation in liquid media in vacuum right after their dispersions by plasma jet. The use of Silver nanoparticles in proposed low dosage are safe to people and pets (they are toxic only to one-cell bacteria) which is proved by special toxicological passport issued by professional researchers from the Institute of Toxicology and Eco-Hygiene of Ministry of Public Health of Ukraine. Efficiency of the product "Silver Shield-1000" in comparison with colloidal Silver made by the best US manufacturer is proved by special studies and economic advantages is shown in Table 1. This muster-butch can be simply mixed in required low quantity with hospital soap, shampoo, tooth paste and dozens of personal hygiene products. For low release it

can be added to water based paint or used for impregnation of surgical plasters or disposable masks or napkins.

Table 1. Comparative properties of colloidal Silver made in USA

with new nanoproduct "Silver Shield-1000"

Product Name & PPM on Label	PPM (from Lab Report)	Particle Surface Area cm^2/mL	Price cents/mL
Sovereign Silver 10 ppm	9.71	**0.217**	**50,70**
Ultra Pure Colloidal Silver 35 ppm	16.5	**0.225**	**30,35**
Argentyn 23 - 23 ppm	15.7	**0.355**	**50,70**
American Biotech Labs - ASAP 22 ppm	22.3	**0.587**	**69,20**
ElectraClear Colloidal Silver 10 ppm	26.4	**0.662**	**20,74**
«Silver Shield-1000» * 100ppm	100,0	**0.679**	**12,50**

*It shall be stressed that "Silver Shield-1000" is nano Silver dispersion (not ionic) in Glycerin (not in water, so it has evidently more expensive media) but the price and the properties are comparable with the best analog of US product [3].

The proposed combined "wet-dry" plasma dispersion technology and semi-industrial unit allows to manufacture the tailored masterbatches of such nano metals as Au, Pt, Pd, Cu, Fe, Mo, Ti, etc dispersed in Natural or Synthetic oils, Monomers, Polysaccharides, Petroleum derivatives, etc. The ready to use liquid dispersions are easily mixable with end products. For example, the nano Silver (or nano Copper) could be loaded to monomers for manufacturing of self sterilized food packaging films and biopolymer materials. The respective nanoadditives could be supplied by JV"MSM" in industrial volume.

The new and perspective application field is using of metal nano additives for the sustainable energy production from biodegradable and reduced compounds. Microbial fuel cells (MFCs) technologies are a promising approach to wastewater treatment as the treatment process can become a method of capturing energy in the form of electricity or hydrogen gas and removing wastes simultenuously. The substrates used in MFCs range from carbohydrates (e.g. glucose, sucrose, cellulose, starch), alcohols (e.g. ethanol, methanol), amino acids, proteins and even inorganic components such as sulfides or acid mine drainages. Evidently the type of bacteria and substrate fed to MFC is a subject of intensive research all over the world. Ukrainian researchers studied the impact of substrate on the structure and composition of the microbial community and have proposed the respective types of bacteria for MFC with effective wastewater treatment and increased power generation. Scientists from I. Franco Lviv University found that the multifold intensification of electricity produced by MFC can be reached by using nano Fe dispersions (supplied by JV"MSM") which is responsible to intensify oxidation [5].

So, the new combined "dry-wet" technology allows to produce in industrial volume the stable concentrated Metal Nanoparticles dispersions ("muster-butches") in liquid ingredients which is a common part of dozens end products. Also these dispersions can be used as the catalysts for new energy sources. It is paving a way for commercialization of nanometals additives and open an opportunity to compete at the forefront of the additive manufacturing revolution, which in the long term will lead into entire new production and consumption paradigms.

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Hiligsmann [a], Styphanie D. Lambert [b], Benoot Heinrichs [b], at all

a Centre Wallon de Biologie Industrielle (CWBI), Dйpartement des Sciences de la Vie, B40, Universitй de Liиge,B-4000 Liиge, Belgium

b Laboratoire de Ganie Chimique, B6a, University de Liege, B-4000 Liege, Belgium

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Fuel Cell,p.14-20, Oleksandr Bilyy, Oresta Vasyliv and Svitlana Hnatush, Lviv, 2014.

10:10 Oral

Sonochemical coating technology as a universal method of surface modification and its application

Bartosz Woźniak[1], Julia M. Rogowska-Tylman[2]

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Ultrasonic waves have many more applications than we can realise.

In most of the cases we are using them for surface cleaning or dispersing of suspensions. Unexpected application of ultrasounds is coating of various materials with nanoparticles. It is possible due to cavitation phenomena. When cavitation bubble growth and collapse, shock waves which give high velocity for nanoparticles are generated. These nanoparticles are deposited on the surface and forming strong chemical and physical bond.

Ultrasound technology allows to create homogeneous layer on the titanium, ceramics or polymer surface. Process of coating is short time and temperature can be adjusted to the needs and the type of material and this method does not damage the surface. Nanolayers can be applied for UV protection, antibacterial coating or bone regeneration implants.

The speech summarizes the influence of coating with nanoparticles and possibilities of application in industry.

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DISCUSSION and questions to all speakers

Friday morning, 17 October, 10:20
Chair: Robert Bucki

COFFEE

Friday morning, 17 October, 10:30

Session 11

Regenerative Nanomedicine
Friday morning, 17 October, 11:00
Chair: Marek Godlewski

11:00 Oral

Bioactive GoHAP nanoparticles and their applications in medical implants technology

Elżbieta Pietrzykowska[1], Tadeusz Chudoba[2], Jacek Wojnarowicz[2], Sylwia Kuśnieruk[3], Witold Łojkowski[1,2]

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Hydroxyapatite (HAp) is a major inorganic component of human hard tissues, such as bones and teeth, and its content determines their microstructures and physical properties. It is one of few materials that are classed as bioactive, meaning that it will support bone ingrowth and osseointegration when used in orthopaedic, dental and maxillofacial applications.

The Institute of High Pressure Physics of the Polish Academy of Sciences (IHPP) has developed technologies of the nanohydroxyapatite powder synthesis (called GoHAP), obtained by microwave reactor and the high pressure consolidation technology for ceramic material. Those techniques aim to obtain materials close to the natural structure and mechanical properties of the bone tissues. Obtained GoHAP ideally mimics the natural hydroxyapatite found in human body. The prepared powder has plate and crystalline structure and is characterized by grain size in the range 5-25 nm (it can be controlled); specific surface area is 237m2/g,. The resorbable powder was investigated according to norm ISO 10993-14.

11:10 Invited Oral

Zebrafish - an animal model for high-throughput screening in nano labs

Małgorzata Wiweger

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Nanotechnologies bring new possibilities but also raise many questions concerning potential benefits and risks. In order to justify the answer *in vivo* testing usually is required. Yet, experimentation on animals is an issue with regards to multiple aspects such as: ethics, costs and methodology. Non-animal models are being developed, but for now, they only partially reflect the complexity of life organisms. Therefore, laboratory animals still have to be used however mice, rats and other mammals can be replaced by evolutionary less developed vertebrates that are still relevant as a model for human diseases but also are suitable for high-throughput screenings.

Zebrafish (*Danio rerio*) are a small fish (up to 5cm) of the Cyprinidae (carp) family. They have been described already in 1822 by Hamilton, but introduced as a model only in the 70's of the twentieth century. In comparison with other laboratory animals zebrafish has many advantages. Fish husbandry requires less effort and costs less than husbandry of mice, rats or any other mammals. Development of zebrafish is relatively fast - they become sexually mature in 2-4 months. On average, 100 highly synchronized in their development eggs can be obtained weekly from one pair of fish and since zebrafish are oviparous, eggs can be collected without harming the parents. Embryos begin to form the vital organs already in 24 hours post fertilization. Also the innate immune system starts to function at this time. After 5 days post fertilization (dpf), the zebrafish larvae reach about 3 mm in length, have fully functional body and are able of independent feeding. Although zebrafish genome is about half size of the human one, 70% of human genes have their equivalent in fish and mutations in the homologous genes leads to similar phenotypes (Howe K. *et al*., 2013, Nature 496: 498-503; Wiweger M. *et al.,* 2012, PLoS One 7:e29734).

Most of the experiments are performed on zebrafish embryos or larvae i.e. stages which are not classified as protected animal. During that time zebrafish can be can be kept in a Petri dish or a multi-well plate and tested substances can be added directly into the water in which the fish are kept. This eliminates the need of making injection and simplifies all procedures. Characteristics of zebrafish (small size, transparency), impressive genetic/ technique tool box with many characterized mutant and transgenic lines and easiness of work with this model allow for long-term (up to a few days) and simultaneous studies in the same organism of various events e.g.: parallel observations of the behaviour of macrophages, neutrophils and tumour

cells (He S. *et al*., 2012, J Pathol. 227:431-445) or observations of the behaviour of osteoblasts upon bacterial infection (model for skeletal tuberculosis, unpublished data). This helps to reduce the number of animals that are needed to carry out experiments. High fecundity, synchronized development and easiness in maintaining and treating embryos make this fish particularly suitable for automated high-throughput screens (Spaink H. *et al*., 2013, Methods. 15:246-54 and Letamendia A. *et al*., 2012, PLoS One). All this characteristic makes zebrafish an excellent model for *in vitro* and *in vivo* studies . For illustration see work of Park H.-G. and Yeo M.-K. (Molecular and Cellular Toxicology, 2013, 9: 375-383) or by van Manen E.H. and colleagues (Odontology, 2014, 102:14-21) where properties of nanomaterials have been tested during bone and dental regeneration.

Zebrafish Core Facility (ZCF) at the International Institute of Molecular and Cell Biology (IIMCB) is a licensed breeding facility that was established in 2012 as a base for Fishing Targets and Medicines (FishMed; http://fishmed.iimcb.gov.pl) – a coordination and support actions financed by the 7th Framework Program of the European Union. Since 2014, ZCF invites new internal and external projects and collaborations. We offer: (i) various lines of *Danio rerio*, (ii) help with handling the zebrafish model (fish husbandry, experiments planning and conducting, teaching various techniques), and (iii) provide bench space in fully equipped zebrafish laboratory. For more information visit http://www.iimcb.gov.pl/zebrafish-core-facility.html or contact us at: aquarium@iimcb.gov.pl; Zebrafish Core Facility, IIMCB, 4 Ks. Trojdena Street, 02-109 Warsaw, Poland.

11:25 Keynote

Artificial bone – from idea and invention to commercialization

Grażyna Ginalska, Anna Belcarz

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The key point in commercialization of new product is an innovative idea, competitive in relation to available products. It should meet the expectations of final users, entering the niche market. Surgeons seek for new bone-replacing materials of enhanced surgical handiness, facilitating the manipulations during implantation procedures. This reason is one of the crucial ones for development of composite materials. During the last years, the focus on ceramics has been extended to polymers, either as hydrogels or as polymer composites.

Elastic biphasic bone replacing composite, the property of Medical Inventi Co, belongs to such innovative inventions. In vitro and in vivo studies confirmed its surgical handiness, bioactivity and lack of toxicity, ability to repair bone defects in animals. All these properties makes elastic composite promising in a segment of biomaterials for bone repair. The composite and process of its production were patented in Poland and applied for patenting in EPO procedure in 2009-2010.

The idea of the composite commercialization appeared in inventors' brains. Two women-inventors decided to go beyond their traditional role of academic researcher. Therefore, they publicized their achievements in mass-media. Information concerning the elastic composite evoked the growing interest of potential investors. After several months, the concept of spin-off company appeared as a result of numerous arrangements. In consequence, Medical Inventi, a spin-off limited company, was created in May 2011. Its structure primarily included: two inventors (President and member of Supervisory Board), Medical University of Lublin and private investor. Patents for bioactive composite were contributed in kind to the Company. The initial funds allowed to perform some clinical experiments, concerning implantation of the biomaterial (stomatological or orthopaedic cases) to human patients. The experiments confirmed good results of healing of bone defects filled with elastic composite which was awarded in 2013 on Brussels Innova Exhibition (WIPO award, Grand Prix of Europe France Inventors and gold medal with mention). These promising information initiated the efforts to capitalize Medical Inventi Company. In the first step, some private funds were introduced into the company structure. Next, the company seeks for venture and private funds in amount sufficient for certification and registration of the product in Office for Registration of Medicinal Products, Medical Devices and Biocidal Products. Also, it is planned to launch the low-scale production of bioactive composite in South-Eastern Poland. For this purpose, MI limited company will be soon transformed to a joint-stock company.

Medical Inventi is focused on certification and commercialization of elastic bone-replacing composite. However, its efforts concern also other biomedical products. For this reason, it seeks for partners in a field of scientific studies. It is also open to a cooperation with industrial companies, interested in the expansion of its business activity.

11:45 Invited Oral

Medical applications of electrospun nanofibers

Tomasz Kowalczyk

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Electrospinning is relatively simple and inexpensive method for preparation of polymer nanomaterials with great potential of use in regenerative medicine and drug delivery systems. Such obtained materials were tested as a temporary scaffold for growing cells that reconstitute functional tissues and organs. The membranes made of nanofibers have been used for in vivo experiments on reconstruction of the ureter and urinary bladder in an animal model - rat. Mesenchymal stem cells colonized the nanomaterial and proliferated on it better than on the acellular collagen matrix. The implant showed a high degree of neovascularization. Nanomaterial was hemostatic when used in kidney-sparing surgery procedures. used as a component of that after staining Fluorescently stained nanofibers made of serum albumin served as pH indicator of micrometer size. Wound dressings made of membrane containing albumin nanofibers had anti-adhesive properties and supported skin implants. Highly innovative prospect for the nanofibers is production of drug delivery systems. Release of antioxidant and protein growth factors was examined in vitro for the membranes made of electrospun nanofibers. Nanofiber membranes used in neurology as an internal anticicatrisation dressing protected cerebral cortex and against neurode-

generation. Nanofibers releasing sodium glutamate were used to create an animal model of Lou Gehring's Disease.

Discussion and questions to all speakers

Friday afternoon, 17 October, 12:00
Chair: Marek Godlewski

LUNCH & POSTERS & FAIR

Friday afternoon, 17 October, 12:10

Session 12

Nano-things against big problems
Friday afternoon, 17 October, 13:00
Chair: Ewa Stępień

13:00 Invited Oral

Small things against powerful bugs: functionalized nanoparticles as a new antibiotics against drug resistant bacteria.

Robert Bucki[1,2], Katarzyna Niemirowicz[1], Urszula Surel[1], Paul Savage[3]

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The rapid development of nanotechnology in the past decade is rising hope that different emerge health problem that human face today, such as increasing number of bacterial resistant infections might be solve with nanostructure designed at atomic level. Iron oxide magnetic nanoparticles and their ceragenins-shell derivatives (MNP-CSA-13) represent one example of such material that despite its small size has the ability to recognize and kill different pathogenic bacteria strains. In this study, we provide evidence that MNP-CSA-13 nanosystem is stable in different body fluids and might be use to elimante multidrug resistant clinical strains *of Pseudomonasaeruginosa*. Additionaly, the bactericidal activity of MNP or MNP-CSA13 is associated with ability to prevent bacteria biofilm formation. Covalent attachment of CSA-13 to the MNP surface increases its biocompatibility by reducing its otherwise relatively high toxicity towards host cells. MNP and their ceragenins-shell derivatives represent a great promise in developing new methods to fight bacterial infections.

This work was financially supported by the National Science Centre, Poland, Grants , UMO-2012/07/B/NZ6/03504 (to RB.) and UMO-2012/05/N/NZ7/00534 (to KN.)

13:15 Invited Oral

Optical and magnetic markers for intraoperative localization of the sentinel lymph node in cancer treatment

Geneviève Pourroy[1], Julien Jouhnnaud[1], Antonio Garofalo[1], Delphine Felder-Flesch[1], Hervé Simon[2], Laurent Mengus[2], Jacques Chambron[2], Kaveh Mohamadabadi[3], Christophe Coillot[4], Christophe Goze-Bac[4]

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13:30 Invited Oral

Nanoparticles for medical imaging

Marek Godlewski[1,3], Jarosław Kaszewski[1,2], Ewelina A. Wolska[1], Michał Godlewski[2,4]

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The first generation of biomarkers, used in biology and medicine as fluorescence labels, is based on organic dyes. In addition to many advantageous properties, these markers show also some disadvantageous ones. The most important are fluorescence bleaching and relatively wide excitation and emission bands. These limitations promoted search for alternative systems. Biomarkers based on quantum dots (e.g. CdS or CdSe) were investigated as the second generation of fluorescence labels. Unfortunately, these markers show also several disadvantageous properties, such as use of toxic cadmium, fluorescence blinking, energy migration, etc.

In the talk properties of a new generation of biomarkers will be discussed. They are based on biocompatible nanoparticles (NPs) of oxides (ZnO, $ZnAl_2O_4$ and ZrO_2) activated with rare earth (RE) ions. RE ions are used because of their sharp atomic-like emission independent of host properties and a size of NPs.

Our tests of NPs biocompatibility indicate that use of relatively large-size NPs (say in the range of 50 nm) allows their detection as foreign objects in the body. Tests on adult mice prove that NPs can be introduced via gastric gavage (IG), which is followed by an intestinal uptake. NPs transport to the variety of tissues and organs (liver, spleen, pancreas and kidney) is detected. Importantly, NPs are recognized as foreign objects and are then removed from most of the organs. Their biocompatibility is confirmed. For the ZnO NPs the metabolism of NPs and further utilization/redistribution of Zn in ion-

ic form is observed.

Acknowledgment

The research was partially supported by the European Union through grant of Innovative Economy (POIG.01.01.02-00-008/08) and by the NCN grant 2012/05/E/NZ4/02994.

13:45 Oral

The use of magnetic nanoparticles as an effective separator and diagnostic of Candida albicans.

Katarzyna Niemirowicz[1], Urszula Surel[1], Izabela Święcicka[2], Agnieszka Z. Wilczewska[3], Karolina H. Markiewicz[3], Alina Kułakowska[4], Zbigniew Namiot[5], Beata Szynaka[6], Halina Car[7], Robert Bucki[1,8]

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Severe fungal infections have significantly contributed to the increasing morbidity and mortality of immunocompromised patients in need of broad-spectrum antibiotic therapy. Therefore, the increasing number of resistant fungal infections creates a constant need for the development of new therapeutic and diagnostic methods. In this study, we show that iron oxide magnetic nanoparticles and their core-shell derivatives possess high affinity to membrane of fungal cells. Additionally, we provide evidence that these nanostructures offer an opportunity to quickly capture, eliminate, and detect presence of *Candida albicans* as well as to separate these pathogens from different body fluids.

This work was financially supported by the National Science Centre, Poland, Grants UMO-2012/05/N/NZ7/00534 (to KN.), UMO-2012/07/B/NZ6/03504 (to RB.)

13:55 Oral

Practical test of biocidal properties of nanosilver.

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Introduction:

Releasing nanoparticles from products and materials causes great concern connected with a potential risk for man and environment. Increase of nanoparticles concentration, including nanosilver, in domestic sewage is caused by more and more frequent use of products containing nanoparticles in everyday life (fabrics, cosmetics, cleaning supplies, paints...). An influence of released nanoparticles on efficiency of wastewater treatment by activated sludge method is particularly important but little known issue. Biocidal action of nanosilver on activated sludge biocenosis could reduce wastewater treatment efficiency and make negative consequence for health.

Materials and methods:

The purpose of the studies was to prepare a new methodology to analyze the influence of silver nanoparticles on the beta hemolytic Gram-positive bacteria that exist in activated sludge and to investigate biocidal activity of nanosilver. The studies were conducted for various concentrations of nanosilver and different times of its contact with a suspension of beta hemolytic bacteria. The effect of influence of nanoparticles on live organisms depends on the properties of nanoparticles, and these change with the surrounding medium and within time. Among many properties of nanoparticles, zeta potential and the size of particles are considered to be of decisive importance in respect of biological effects.

Bacteria were isolated from activated sludge collected from residential wastewater treatment plant by spread-plate method on medium COLUMBIA (bioMerieux). A colloidal nanosilver solution received by the physical method was used in the experiment. The size of particles, their concentration and zeta potential (Zetasizer 3000, Nanosight NS500) were determined in the nanosilver solutions.

Results:

The results obtained indicate that nanosilver in solution forms a solution with heterogeneous structure containing nanoparticles and their agglomerates. Together with a change of colloid concentration its properties also modify – the size of particles and zeta potential are changing. Because of aging of the colloidal nanosilver solutions their characteristics and biocidal properties change. The experiments showed that nanosilver shows biocidal activity against beta hemolytic bacteria isolated from activated sludge. It was found that there exists a relation between the nanosilver dose and the time of contact with bacteria. The higher the volume of nanosilver dose is, the more effective it is and the shorter is the time required to obtain the biocidal effect.

Discussion:

The conducted studies have shown the usefulness of the test based on the viability of beta hemolytic bacteria in the screening assessment of potential biocidal properties of nanosilver. The developed method is characterized by low costs, simplicity in conducting and rapidity of determination of nanosilver biocidal effectiveness. The method, after some modifications, may be also applied in effectiveness tests of other substances toxically acting on microorganisms.

14:05 Oral

Wykorzystanie cytometru przepływowego Apogee A50Micro w ocenie ilościowej i jakościowej mikrocząstek biologicznych

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Mikrocząstki - geneza i biologia

Krążące mikrocząstki (MP) to są pęcherzyki pochodzenia komórkowego obserwowane we wszystkich płynach ustrojowych, począwszy od krwi, płynu mózgowo-rdzeniowego, moczu, śliny, mleka oraz płynów jam ciała. Definicja mikrocząstek opiera się głównie o kryterium wielkości, wskazując na obiekty wytwarzane z pęcherzyków na powierzchni błony komórkowej (ang. *vesiculation*) przez ich „złuszczanie" (ang. *exfoliation*) lub „wypychanie" (ang. *shedding*, *bubbling*). Średnicę określa się między 0,1 a 1,5 mm, czyli więcej niż średnica egzosomów (obserwowanych w próbkach pochodzących z hodowli komórkowych), a mniej niż średnica małych płytek – trombocytów (obserwowanych w próbkach osocza).

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MP są wytwarzane w odpowiedzi na czynniki stresogenne, takie jak niedotlenienie, uraz mechaniczny???' ??? lub w wyniku reakcji zapalnej.??? Utrata asymetrii błony komórkowej towarzysząca uwalnianiu MP ma też związek z aktywacją szlaku apoptotycznego w komórce???, jednak w przypadku komórek śródbłonka niemal zawsze jest ona wyrazem dysfunkcji??? lub szeroko pojętej aktywacji.???'??? Są liczne doniesienia wskazujące na udział MP w procesach regeneracyjnych, np. w rewaskularyzacji.???' ??? MP działają jak „transmitery" przenoszące aktywne biologicznie endogenne związki substancje???, które biorą udział w procesie adhezji i chemotaksji??? oraz angiogenezy.??? W chorobach układu sercowo-naczyniowego, takie jak choroba niedokrwienna serca, zawał, udar, kardiomiopatia, nadciśnienie, a także cukrzyca i choroba zakrzepowa mikrocząstki są uważane za markery toczących się procesów patofizjologicznych lub pobudzenia. ???'???'???'??? Mimo wielu dowodów na udział MP w procesach fizjologicznych oraz w odpowiedzi patofizjologiczne bodźce, rola ich nie jest dobrze poznana, a mechanizmy regulujące tworzenie MP są intensywnie badane na modelach *in vivo* i *in vitro.*

Metody badawcze stosowane do oceny funkcji biologicznej i charakterystyki mikrocząstek

Duży postęp, jaki się dokonał w o ostatnich latach, szczególnie w technikach opartych o metody mikroskopowe i cytometryczne, pozwolił również na wprowadzenie nowych narzędzi do badań nad rolą MP oraz oceną ich budowy i funkcji.

Szczególne rozwój techniki cytometrii przepływowej, w kierunku zwiększenia rozdzielczości tej metody umożliwia charakterystykę tak małych obiektów jak cząstki o średnicy poniżej 100 nm. Dostępne na rynku cytometry przepływowe o wysokiej rozdzielczości, jakim jest Apogee A50-Micro (Apogee Flow Systems Ltd) mogą różnicować obiekty o średnicy nawet 20 nm, czyli można przy użyciu tego urządzenia analizować nie tylko MP, ale również egzosomy. Przy czym obiekty te można analizować zarówno ilościowo (liczba MP) jak i jakościowo (ocena średnicy obiektu oraz specyficznych antygenów powierzchniowych). Urządzenia te są one dostosowane do metod rutynowo wykorzystywanych w cytometrii przepływowej, t.j. oznaczanie antygenów powierzchniowych za pomocą przeciwciał znakowanych fluorescencyjnie. Zastosowanie w aparacie Apogee A50-Micro 3 laserów wzbudzenia, pozwala wykonanie jednoczasowo analizy 9-kolorowej , czyli umożliwia szczegółową charakterystykę ilościową i jakościową antygenów znakowanych takimi barwnikami jak:

1/ laser niebieski 488 nm (odczyt: FITC, PE, ECD, PC5, PC5.5, PC7, Alexa Fluor 488, FLMA, PI, 7-AAD, GFP, YFP, Ds-RedFP),

2/ laser czerwony 635 nm (odczyt: APC, APC-Cy7, Alexa Fluor 647, Alexa 700, APC Alexa 700, PE Alexa Fluor 700

3/ laser fioletowy 405 nm (odczyt: DAPI, Hoechst, PacificBlue, PacificOrange, KromeOrange)

Można zatem dokładnie określić pochodzenie MP, czy są produkowane przez płytki krwi, czy komórki śródbłonka, oraz ich stan wzbudzenia (apoptoza, dysfunkcja, itp.). Z punktu widzenia zastosowania Apogee A50-Micro do badań nad MP, wysoka rozdzielczość tego urządzenia oraz szerokie spektrum analizy, ma to istotne znaczenia przy poszukiwaniu nowych biomarkerów, charakterystycznych dla dysfunkcji śródbłonka i płytek.

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Discussion and questions to all speakers
Friday afternoon, 17 October, 14:15
Chair: Ewa Stępień

DISCUSSION

Conclusions from the conference and farewell
Friday afternoon, 17 October, 14:25
Chair: Witold Łojkowski

Farewell
Friday afternoon, 17 October, 14:40

Posters

Wednesday, 15 October

POSTER

Poster's fixing to be displayed 3 days
Wednesday morning, 15 October, 11:00

11:00 Poster 1

Synthesis of multiwalled carbon nanotube-polymer nanohybrids and their internalization to biological systems

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Multiwalled carbon nanotubes (MWCNTs) have many potential applications in both, medicine and biology. They can be used either as tissue scaffolds and biomarkers for cellular imaging or drug delivery systems and a basis for biosensors. The MWCNT polymer wraping helps to integrate them into several substance classes or biological systems and make them perfectly dispersed in organic solvents. The CNTs wrapped in polymers are used for the integration into various substance classes or biological systems and enable their dispersibility in organic solvents.

In the present studies, we have focused on the functionalization protocol of multi-walled carbon nanotubes (MWCNTs) by polyethylene glycol (PEG) [1] with three selected molecular weights: 400, 2 000 and 100 000. All three polymers were covalently attached to the surface of previously oxidized carbon nanotubes [2] and the water-soluble PEG-MWCNT hybrids were successfully formed. The nanostructures were characterized by means of Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR) and Raman spectroscopy. MWCNTs and PEG-MWCNT complexes were internalized into HeLa cells. The cells were analyzed by confocal microscopy and In-cell Analyzer after selected organelles staining. The investigation of PEG-ylated nanostructures treated cells has revealed the higher viability and unchanged morphology in comparison with non functionalized CNT samples.

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Acknowledgements

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11:00 Poster 2

Preparation of antimicrobial nanoparticles containing chitosan

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In recent years, the number of diseases caused by microbiological and nosocomial infection increased, which led to intensive research on new materials for medical and hygiene products.The fibrous materials used so far for medical and hygienic applications, which gained antimicrobial properties, were usually obtained using a treatment with salts and/or nanoparticles of silver. Currently it is common to depart from the use of silver in dressings materials, and therefore and interest in research and technology development based on the use of alternative renewable materials has increased.

In this work modern fibrous cellulose products modicied by chitosan nanoparticles were developed. For the modification of cellulose fibers a useful form of chitosan - microcrystalline chitosan (MCCh) and a complex od chitosan/alginate of nanometric dimension were developed. A method of applying chitosan nanoparticles on fibrous cellulose products was elaborated. The modified products obtained were subjected to the assesment of their antimicrobial activity as well as physico-mechanical and sorption properties.

Tests were carried out within the project No. NCBiR/ ERA-NET-MATERA/01/2011 "Advanced Cellulose Materials - AdvanCellMat"

11:00 Poster 3

Enviromental syndorme - the case of non-communicable diseases induced by nanoparticulate matter

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Environmental Syndrome (ES) is the universally basic, global interdisciplinary new terminology communication tool presented and en-

dorsed by 2012 Geneva, Congress of European Academy of Allergy and Clinical Immunology (EAACI) [1]. The aims of ES etymology is to create harmonized, constructive, credible, common assertive nomenclature for clinical expression of impending environmental disturbances.

The immunological system response to hostile and disturbing agents is the most sophisticated, ultra sensory signalling entity to environmental degradation. The immuno-response parameters, humoral and cellular, detects instantly indicators of inducting factors comprising nanoparticulate inhalants (pollutants), ingestants and contactants (i .e. nickel). However, different people on different continents, will show different symptoms from the same aberrant. In this respect an independent quality surveillance, on separate geographical locations is an exclusive guarantee to collect credible, comparable data. The nanoparticulate matter is of a special concern as the amount and heterogeneity of hazardous nanoparticles are growing limitless and exponentially in human environmental surrounding. Nanomaterials are able to cross biological membranes and access cells, tissues and organs that larger-sized particles normally cannot; they can gain access to the blood stream via inhalation or ingestion and also can penetrate the skin [2].

In order to elucidate immunoresponsive nature of nanoobjects we propose to use a combination of X-ray spectrometry techniques based on total reflection phenomenon [3,4]. Suggested methodology is providing elemental detection limits in femtogram range and is also very discriminating in the accurate description of nanoobjects [5]. Thus nanoparticlulate matter can be measured determining its elemental composition, size and density in order to allow comparison of this comprehensive data with the immunoresponse of nanoparticles. Such a methodology will create a new clinical characteristics of nanoparticulate related ES as a credible attempt to collect constellations of a variety of symptoms, at given geographical location. Effective analyses of signalling immunotoxicity biomarkers will allow to selectively discriminate of ecologically threatening contaminants. As yet, unpredictable clinical manifestations of nanoparticulate disturbances, will culminate in establishment of locally specific ecoprotective measurements.

Acknowledgements:

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11:00	Poster	4

Lactoferrin as a potential nanodrug preventing neonates sepsis

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Lactoferrin (formerly known as lactotransferrin) is a glycoprotein, and a member of a transferrin family, thus belonging to those proteins capable of binding and transferring Fe^{3+} ions. There are three forms of lactoferrin according to its iron saturation: apolactoferrin (iron free), monoferric form (one ferric iron), and hololactoferrin (binds two Fe^{3+}). Lactoferrin is widely represented in various secretory fluids, such as milk, saliva, tears, and nasal secretions. Lactoferrin is one of the components of the immune system of the body; it has antimicrobial activity (bacteriocide, fungicide) and is part of the innate defence, mainly at mucoses. In particular, lactoferrin provides antibacterial activity to human infants. [1,2]

Bovine lactoferrin (Lf) supplementation has been implied in prevention of late-onset sepsis in preterm very low birth weight neonates [3]. However, little attention has been paid to iron saturation level of lactoferrin preparations used in reported studies. This seems alarming as there is evidence for Lf serving as iron donor for pathogen species. Biological impact of various lactoferrin species is still poorly understood and requires further research. Our goal is to differentiate effects mediated by apo- and hololactoferrin on both microorganisms and intestinal epithelial cells function. Also, we endeavour to obtain lactoferrin saturated with manganese ions (MnLf). It seems plausible that MnLf might tip the balance of intestinal microbial ecology in favour of probiotic species that require manganese for their growth.

Here, we present comprehensive method for quantification of metal saturation levels of bovine lactoferrin preparations based solely on the defined ratio of absorbances. Combining spectrophotometry, ELISA and ICP-MS we plotted calibration curve between metal saturation levels and absorbance ratio [4]. Also, several methods serving to obtain various lactoferrin species (MnLf included) were inspected and evaluated. Further studies included testing of antimicrobial activity and microbial adherence assays. These results will certainly contribute to better understanding of biological activities of lactoferrin species differentially saturated with metal ions in context of bacterial translocation from intestinal lumen to peripheral blood.

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11:00 Poster 5

Nanocrystalline hydroxyapatites modified with various ions.

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Synthetic nanocrystalline hydroxyapatites, due to their high biocompatibility are widely used as bone substitutes, implant coatings and fillers in composites.

The chemical, mechanical and biological properties of hydroxyapatite may be improved by doping of additional ions into its structure.

Various substitutions, both cationic (for calcium cations) and anionic (substituting orthophosphates and structural hydroxyl groups) have been recently prepared and proved to be favorable to biological response, thermal stability or bioresorbability of bioceramic.

In our study we focus on synthesis and physicochemical analysis of nanocrystalline hydroxyapatites modified with several anions- silicates (SiO_4^{4-}), borates (BO_3^{3-}), carbonates (CO_3^{2-}), selenites (SeO_3^{2-}) and cations – strontium (Sr^{2+}), magnesium (Mg^{2+}) and manganese (Mn^{2+}).

It is known that magnesium and carbonates are the most abundant "impurities" of biological apatite. Magnesium ions have been shown to play an important role in bone remodeling and osteoblasts activity whereas carbonate ions have a great impact on solubility. Strontium stimulates preosteoblasts replication and inhibits bone resorption while borate ions have a favorable effect on mineralization process. Therefore Sr^{2+} and BO_3^{3-} enriched hydroxyapatite may be used in osteoporosis treatment. Silicates play a crucial role in bone metabolism. Used in bioceramics, it increases solubility of hydroxyapatite and improves osseointegration. Manganese ions (Mn^{2+}) are known to induce integrins in tissues (participating in intercellular adhesion) and to stabilize bioceramic in bone. Selenium may prevent carcinogenesis and inhibit the growth of tumor cells (even in bone metastasis).

We have characterized the influence of incorporation of these ions on the apatites structure and physicochemical properties.

11:00 Poster 6

Microwave solvothermal synthesis of resorbable nano-HAp with controlled grain size.

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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 $270m^2/g$ 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 $20mg/dm^3$.

11:00 Poster 7

Alginate/chitosan core-shell beads with bioactive functionalities – synthesis and preliminary physicochemical characterization

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Bacterial infections still remain as one of the most serious threat to human health. The widespread use of antibiotics has created bacterial evolutionary adaptation, leading ultimately to the so-called multidrug resistance (MDR). The consequence of the increasing resistance of bacterial strains to biocidal agents is an increasing risk of chronic infections and difficulties in their treatment (*i.e.* wound infections, osteomyelitis, septic arthritis, endocarditis, *etc.*). Antibiotic therapies, in case of chronic bacterial infections, are very limited. Due to the ease of pathogens spread multidrug resistance has become a global problem[1].

Extensive efforts should be made to find effective antimicrobial agents with low toxicity to the host cell and a broad spectrum towards a wide variety of bacterial pathogens[2]. It is equally important to develop effective delivery systems that integrate existing antibiotics and/or other biocidal agents with novel drug delivery systems to promote their antimicrobial activity.

Present study concerns alginate/chitosan beads constructed of polymeric core loaded with antibiotic (*e.g.* ciprofloxacin) and polymeric shell prone for further lytic enzyme immobilization. Due to stable and uniform core-shell beads formation diverse synthetic approaches were carried out. Preliminary physicochemical characterization of obtained materials was performed: size and zeta potential were determined by dynamic light scattering technique (DLS), particles shape and morphology were investigated with scanning electron microscopy (SEM), drug loading efficiency and cumulative drug release profiles were evaluated with UV-Vis spectrophotometry.

Acknowledgements: This work was supported by Polish Foundation of Science within POMOST project "Alginate/chitosan core-shell beads with bioactive functionalities" (POMOST/2013-7/7).

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11:00 Poster 8

Biocompatible and bactericidal chitosan-silver nanocomposites

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The increasing number of Multidrug-Resistant (MRD) bacterial strains makes a need of searching innovative biomaterials exhibiting antibacterial activity very urgent???. Several desirable properties such as biocompatibility, biodegradability and non-toxicity against human cells are required. Among a number of current strategies, the synthesis of metal nanoparticles as fillers inside polymers and further bionanocomposite preparation methods draw a lot of attention???.

Present studies concern chitosan (CS) based silver nanoparticles (AgNPs) and further composites as solid films. Different variables have been analyzed in order to optimize the bactericidal properties of silver nanoparticles embedded in chitosan films. The main challenge was to achieve a complete bactericidal effect against antibiotic-resistant, biofilm forming Gram-negative (*Pseudomonas aeruginosa, Eschierichia coli*) and Gram-positive (*Staphylococcus aureus*) bacterial strains with simultaneous low (or even lack) cytotoxicity towards mammalian cell lines.

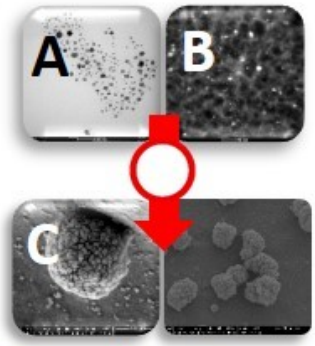

Fig.1. AgNPs (A) and resulting CS-AgNPs nanocomposites (B) exhibit bactericidal activity (C) and simultaneously are non-cytotoxic towards mammalian cell lines.

The best results were obtained for materials based on chitosan with the medium molecular weight and the highest deacetylation degree where a fast reduction of silver ions was favored. It leads to smaller nanoparticle formation and a homogenous NPs dispersion across the membrane)???.

Bactericidal effect towards Gram-negative and Gram-positive bac-

terial strains (Fig.1) and low cytotoxic effect towards human keratinocytes were obtained *in vitro*. This results make the obtained nanocomposites perfect candidates for many biomedical application including wound dressings, antimicrobial coatings on medical devices and so on.

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11:00 Poster 9

Preparation of silver sols by reduction of silver bromide nanocrystals with the use of different reducing agents

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Colloidal systems can be described as a bi- or multiphase systems, wherein the continuous phase (dispersion medium) is distinguish as well as the dispersed phase. Dimensionality of the dispersed matter is distributed in a range of several to several hundred nanometers. Colloidal systems are also known under the name of sols. Silver sols can be characterized by a specific electrical, optical and mechanical properties.

Silver sols were prepared by reduction of silver bromide nanocrystals with the use of two different reducing agents: hydrochinone and pyrocatechol. Reactions were conducted in diverse environments to determine the optimum pH. Done were solutions necessary to obtain a suspension of silver bromide nanocrystals, then a Lippmann suspension in the darkroom was made. Diagram of apparatus used in the synthesis of Lippmann suspension was presented on the Fig. 1. Photolytic reduction was performed on properly prepared samples of the photosensitive material using the aforementioned reducing agents. The evaluation of the reaction was carried out under the spectroscopy tests.

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11:00 Poster 10

Application of silver and copper nanocolloids in disinfection of explants in chrysanthemum in vitro cultures

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Nowadays mass reproduction of many species of ornamental plants, e.g. chrysanthemums, is conducted in *in vitro* cultures. Plants are propagated in specific laboratories under strictly controlled light and temperature conditions, irrespective of season and climate zone. Micropropagation is performed under sterile conditions, on an auto-

claved nutrient medium poured into glass vessels or plastic containers. Plant parts which are transferred on the medium are known as explants. Work with disinfected plant material is only possible under aseptic conditions under HEPA filtered air in laminar flow cabinets. For this reason also the initial plant material collected from the place of cultivation (garden, greenhouse) needs to be disinfected before the initiation of an *in vitro* culture and micropropagation. Ethanol, sodium or calcium hypochlorite or mercuric chloridesolutions are the most frequently used sterilizing agents. However, these chemical compounds often damage the plant tissue or are ineffective in the elimination of bacterial and fungal contaminations. Silver and copper nanocolloids also present antibacterial, antifungal and antiviral activities. The aim of this research was to develop an effective disinfection protocol of *Chrysanthemum* × *grandiflorum* /Ramat./ Kitam. 'Falco' explants during initiation of *in vitro* cultures. The explants - shoot tips were first rinsed under running water. Then they were incubated in 5% detergent solution for 5 minutes. Next they were transferred into 70% ethanol solution for 5 seconds. After that the explants were immersed in Ag, Cu or Ag+Cu nanocolloids at the concentration of 5 or 10 ppm for 5 or 10 minutes. Then they were rinsed for 5 minutes in sterile distilled water. The explants were dried on sterile paper and inoculated on the universal MS medium. During four successive weeks observation were made for the appearing of bacterial and fungal contaminations. The share of disinfected cultures in each research object was determined. For this data the Freeman-Tukey transformation was used and next the analysis of variance for a three-factor experiment was performed. Means were evaluated with the Tukey test at the significance level of 5%. The share of sterile cultures, depending on the research object, ranged from 70 to 100%. The statistical analysis did not demonstrate significant differences between the used nanocolloid type, its concentration and the time of explant disinfection on the percentage of disinfected cultures. Nevertheless, the research confirmed the usefulness of Ag and Cu nanocolloids for the elimination of bacterial and fungal contaminations in chrysanthemum *in vitro* cultures. The nanocolloids demonstrated satisfying antibacterial and antifungal activity even at low concentration and short time of disinfection. Additionally no plant tissue damage was observed.

11:00	Poster	11

Cytotoxicity assessment of herringbone carbon nanostructures dispersed in two different surfactants

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Carbon nanostructures include wild range of materials, i.e. carbon nanotubes and nanofibres. There are three forms of nanofibres, classified by the angle of the graphene platelets or layers with respect to the filament axis: stacked, herringbone and nanotubes. Due to carbon nanostructures properties (i.e. mechanical, electrical, kinetic, optical, thermal) they are commonly used in electronics, mechanics, energy storage, sensors, filling materials in nanocomposites and biomedicine. There are different reports concerning their cytotoxicity profile in biological systems. Especially in the case of herringbone structures, there is poor knowledge of biocompatibility traits. There is deep need to develop bioaspect in this field.

The goal of the study was to determine potential cytotoxic effect of herringbone nanostructures in *in vitro* study dispersed in two surfactants.

The investigation were revealed on cancer (HeLa) and normal (HEK293) cell lines. Herringbone nanoparticles in diameter 12 nm, were examined in six different concentrations (5,10,15,25,40,60 µg/ml) as well as in three different time intervals (24, 48 and 72 h). Methodology involved viability test (Trypan blue staining) and WST-1 proliferation assay. All procedures were conducted with two groups of herringbone nanostructures: dispersed in surfactant Nanosperse AQ (NaAQ) (NanoLab Inc) and dispersed in surfactant Pluronic P-123 (Sigma).

The investigations show, that there is no significant cytotoxic effect in analyzed range of nanoparticles' concentrations in both biological schemes (HEK293 and HeLa cells lines) as well as both surfactants. These results lead to the conclusion, that herringbone carbon nanostructures represent biocompatibility profile with different surfactants, which enable their use in biomedicine.

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11:00	Poster	12

Electrospun nanocomposites from PHBV for bone tissue engineering

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Filling the defects which are caused by fractures, injuries or surgical interventions after tumor resections are of the most importance in today's regenerative medicine. One of the promising technologies for scaffold fabrication is electrospinning. This method allows for micro and nanofibers preparation from polymer solutions with the use of electrostatic forces[1]. Electrospun nanostructure scaffolds composed of biodegradable polymer systems could not only mimic a structure of native Extracellular Matrix (ECM), but also work as a carrier for control release of drugs into injured site.

The aim of this study was development and characterization of nanofibrous mats from novel biodegradable polymer, extracted from biomass, poly-3-hydroxybutyrate-co-3-hydroxyvalerate (PHBV) blended with poly-3-hydroxybutyrate (PHB) and Bovine Serum Albumin (BSA). Polymeric composites based on polyhy-

droxyalkanoates have drawn considerable attention in recent years as a scaffolding material in tissue engineering and regenerative medicine due to their degradation ratio and non-toxic product hydrolysis (R)-3-hydrobutyric acid which serves as a normal constituent of human blood. BSA was used as a model protein. Cytocompatibility of the prepared mats was evaluated. The topography and morphology of the obtained nanofibers mats was analaysed using a Scanning Electron Microscopy (SEM). The wettability of the mats was measured by water contact angle. Mechanical properties were investigated using a mechanical tester. Release study of BSA were evaluated using spectrophotometer UV-Vis.

The process parameters of electrospinning were optimized in order to produce uniform fibers from pure polymer and polymeric composites. The obtained fiber diameter decreased with addition of PHB. The fibers diameters were up to 500 nm. Our results present the effects of PHB and BSA addition to PHBV polymer blends and its influence to Human Mesenchymal Stem Cells (hMSC) response.

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11:00 Poster 13

TiO_2-based nanoparticles as photosensitizers in photodynamic therapy

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Titanium dioxide is well-known material mainly due to its interesting photocatalytical activity. Due to properties such as strong light absorption in UV range, low toxicity, biocompatibility and chemical stability, TiO_2 is widely used not only in the field of photochemistry, but also in biomedicine [1]. Particular focus is on its photocatalytical properties that can greatly benefit the photodynamic therapy (PDT) in cancer treatment, by acting as a photosensitizer (PS) and/or as carrier of other photosensitizing molecules or biologically active molecules (e.g. antibodies). The predominant role of TiO_2 NPs in PDT is a generation of Reactive Oxygen Species (ROS) upon light exposure and with the contribution of the molecular oxygen, which exhibit cytotoxicity towards cell located in their vicinity [2].

In order to maximize photosensitizing properties, nanocrystalline particles of TiO_2 doped with acceptor Fe^{3+} ions (1-10 wt. %) have been prepared with the sol-gel method. The synthesis procedure included modification with polyethylene glycol (PEG400) after nanoparticles formation. The morphology and phase composition of as-prepared NPs have been investigated with XRD and TEM. According to results, TiO_2 NPs, crystallize in the predominant anatase phase over the rutile and brookite trace contribution, what has been also evidenced with Raman spectroscopy. UV-Vis studies have shown significant changes of the band gap structure and light absorption properties upon Fe-doping and PEG modification. The cellular response to as-prepared NPs treatment have been studied in vitro on cervical cancer cells (HeLa) and normal fibroblasts (Detroit 551) with Live/Dead (Calcein/Ethidium homodimer-1) fluorescent assay. In turn, the photo-induced cytotoxic activity of HeLa in comparison to normal fibroblasts have been investigated with WST-1 assay using near-visible (405 nm) light irradiation. Finally, the cellular uptake of TiO_2 NPs and their effect on the cell viability and morphology have been observed with fluorescence and confocal microscopy using fluorescent labeling method with FITC dye (fluorescein isothiocyanate) and specific cross-staining of mitochondria (Mito-Tracker green), lysosomes (Lyso-Tracker red) and nuclei (Hoechst).

Acknowledgements

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11:00 Poster 14

Surface Plasmon Resonance and gold nanoparticles based detection of miRNA-210

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MircoRNAs (miRNA) are short single stranded molecules which control genes expression by degradation of mRNA or by inhibition of translation. The Role of miRNA in many pathological states makes them a potential candidate for biomarkers. Despite many research results that sustain the correlation between the changes of expression of miRNA and the progress of pathological state, there are many challenges that have to be overcome for the development of method for miRNA detection. The most characteristic feature of miRNA is their short length and high sequence similarity. Those factors may cause many problems in hybridization based detection methods. For this reasons a development of new methods of detection that are sensitive and reliable is essential.

Surface Plasmon Resonance (SPR) based diagnostic methods are characterized by short time needed for obtaining an result, are

sensitive, and don't require labeling of molecules. In this work we present a new method for detection based on SPR in which after the hybridization of miRNA on the sensor surface with the PNA probe the 3' and 5' ends or miRNA are enzymatically and chemically modified with the use of poly(A) tail or biotin. After the functionalization of miRNA the detection stage is carried out by gold nanoparticles functionalized with poly(T) or streptavidin.

The analysis shows that the use of gold nanoparticles that enhance the SPR signal and specific PNA probes, it is possible to detect miRNA in subnanomolar concentrations. The results show that the sensitivity and versatility of this method of detection can be used for the development of diagnostic methods that don't require amplification of genetic material.

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11:00 Poster 15

Cell-penetrating peptides as nanocarriers for drug, gene and contrast agents delivery into human cells.

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Cell-penetrating peptides are short, positively charged (at neutral pH) peptides possessing the ability to penetrate into the cell. This property, combined with formation of complexes with other molecules, allows to use them as transporters crossing the biological membrane barrier. By means of covalent or electrostatic interactions cell-penetrating peptides bind to molecules and are efficiently internalized into the cell.

The aim of the study was comparison of two CPP peptides, C6M1 and HR9 as intracellular transporters. Both peptides non-covalently interact with quantum dots ($CuInS_2$/ZnS) and DNA (pEGFP-N1 plasmid DNA) forming stable CPP/QD and CPP/DNA complexes which are capable of entering human cells. The internalization studies revealed that FITC-labeled CPP peptides are distributed evenly throughout the cytosol, mainly close to nuclear membrane. The transporting efficiency of CPP carriers of active biomolecules was proved with the internalization of plasmid and observed expression of GFP protein.
In addition it was proven that HR9/QD complex in contrast to C6M1/QD, can effectively enter human cells. Finally, the cytotoxic activity of peptides and prepared complexes were studied by WST-1 assay on normal and cancer cells, while microscopic analysis of cell structures has allowed an assessment of the physiological condition of the cell.

Provided results on the intracellular localization and the ability to transport functional molecules is essential from the point of view of further development and application the proposed system for therapy and medical diagnostics.

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11:00 Poster 16

Wykorzystanie mikromacierzy w ocenie prozapalnych i proangiogennych cytokin w patomechanizmie retinopatii cukrzycowej

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Wstęp:

Polskie Towarzystwo Diabetologiczne definiuje cukrzycę jako grupę chorób metabolicznych charakteryzującą się hiperglikemią wynikającą z defektu wydzielania i/lub działania insuliny. Przewlekła hiperglikemia wiąże się z uszkodzeniem, zaburzeniem czynności i niewydolnością różnych narządów, zwłaszcza oczu, nerek, nerwów, serca i naczyń krwionośnych.

Cukrzyca to pierwsza niezakaźna choroba uznana przez ONZ za epidemię XXI wieku. Szacuje się, że w 2035 roku liczba chorych sięgnie 592 milionów.

W Polsce na cukrzycę typu pierwszego i drugiego (cukrzyca typ 1 i typ 2) cierpi około 3 mln osób, przy czym ok. 800 tys. nie wie o swojej chorobie. Jednym z poważniejszych powikłań cukrzycy jest retinopatia cukrzycowa. Jest to schorzenie związane z uszkodzeniem naczyń krwionośnych o średnicy poniżej100 mm znajdujących się w siatkówce oka. Patomechanizm tego schorzenia opiera się na zmianach w produkcji pozapalnych i proangiogennych cytokin.

Cytokiny są białkami o niskiej masie cząsteczkowej, które biorą udział w przekazywaniu informacji pomiędzy komórkami. Odgrywają one istotną role w odpowiedzi zapalnej, apoptozie, wzroście komórek i ich różnicowaniu. Ich nośnikami mogą być mikrocząstki, które mają postać pęcherzyków o średnicy od 0,1-1 µm. Mikrocząstki powstają z błony komórek mających bezpośredni kontakt z krwią, jak na przykład komórki śródbłonka i płytki krwi. W oznaczeniu cytokin wykorzystano mikromacierze do oznaczania białek. Wykonane one są z opłaszczonych przeciwciałami membran, które pozwalają na bardzo szeroką ocenę zależności międzykomórkowych. Służą one do przesiewowego oznaczenia wielu cytokin (n=43) i porównania poziomu ich ekspresji w próbce o objętości 100 mL. W klasycznej metodzie ELISA przy użyciu takiej samej ilości materiału można uzyskać informacje o stężeniu tylko jednego parametru. Wybrana metoda pozwala przy bardzo niewielkiej ilości materiału biologicznego określić kluczowe czyn-

niki i mechanizmy chorobowe oraz biomarkerkery, związane z sygnalizacją cytokin.

Cel badania:

Prowadzone badanie ma na celu ustalenie, które z prozapalnych i proangiogennych cytokin przenoszone są przez mikrocząstki. Istotne jest również określenie jakie zmiany zachodzą w ich profilu u osób chorujących na cukrzyce powikłaną retinopatią. Informacje te mają kluczowe znaczenie dla wyjaśnienia i zrozumienia patomechanizmu retinopatii cukrzycowej.

Metody:

Nasze badanie objęło 12 pacjentów w tym 5 kobiet i 7 mężczyzn. Za kryterium podziału wybrano poziom hemoglobiny glikowanej (HbA1C). Zgodnie z wytycznymi Polskiego Towarzystwa Diabetologicznego na 2013 rok wartość HbA1C na poziomie 7% jest wartością graniczną. Osoby u których poziom hemoglobiny glikowanej przekracza 7% traktowane są jako pacjenci z nieuregulowaną cukrzycą (UCD). Pozostali pacjenci wchodzą w skład grupy z cukrzycą wyrównaną (CD). Trzecią wyodrębniona grupą są osoby nie chorujące na cukrzycę (C). Do każdej z grup zaklasyfikowano po cztery osoby.

Badanym materiałem było osocze otrzymane z krwi pacjentów pobranej na antykoagulant cytrynianowy (0,109 M). W celu oddzielania osocza od elementów morfotycznych krew pełna była wirowana dwukrotnie przez 15 min przy prędkości kątowej rotora 2500 G. Otrzymane osocze, w którym znajdują się substancje białkowe m.in. cytokiny w stanie wolnym jak i związane z mikrocząsteczkami, porcjowano po 300 µl. Chcąc wyodrębnić mikrocząstki powtórnie zwirować rozporcjowane próbki przez 90 min przy prędkości 16 000 G. Mikrocząstki po wirowaniu zagęszczają się w dolnej frakcji próbki.

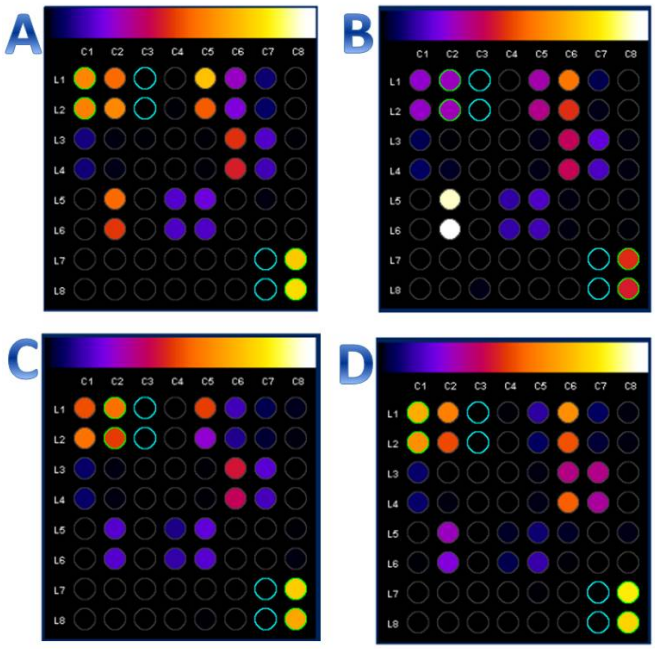
Oznaczenie prozapalnych i proangiogennych cytokin wykonano zarówno w osoczu z mikrocząsteczkami jak i w osoczu ich pozbawionym. Badanie przeprowadzono wykorzystując zestaw mikromacierzy do oznaczania białek firmy Rybiotech serii C. Zestaw ten ma postać mikromacierzy nitrocelulozowych o wymiarach 2x3 cm i umożliwia jednoczesne oznaczenie 43 różnych białek min. czynników wzrostu, proteaz i rozpuszczalnych receptorów. Test ten jest zmodyfikowaną metodą Western blot. Na błonie z nitrocelulozy umieszczone są przeciwciała wychwytujące skierowane przeciwko różnym cytokinom. Oznaczenie opiera się o zasadę wiązania przeciwciała z antygenem. Metoda ta charakteryzuje się wysoką czułości (1pg/ml dla niektórych białek) oraz niską nieprecyzją rzędu 5%. Test ten jest również bardzo swoisty dzięki zastosowaniu wyselekcjonowanych przeciwciał. Detekcja odbywa się na zasadzie pomiaru chemiluminescencji substratu rozkładanego przez związany z przeciwciałem enzym peroksydazę chrzanową. Do detekcji wykorzystano zapis cyfrowy obrazu, uzyskany przy użyciu systemu EC3 Bioimaging firmy UVP z oprogramowaniem Vision Works LS. Do opracowania wyników wykorzystano program ImageJ.

Wyniki:

Oznaczenia cytokin wykonywano równolegle dla próbek z mikrocząsteczkami oraz dla osocza pozbawionego mikrocząstek. W materiałach poddanych analizie wykryto zwiększoną obecność angiostatyny i RANTES (chemokina β) na mikrocząstkach (Rys. A i B) w stosunku do osocza pozbawionego mikrocząstek (Rys. C i D). Różnica była istotna statystyczni i wynosiła odpowiednio (p=0,03 i p=0,007).

Przeanalizowano różnice pomiędzy próbkami pochodzącymi od chorych na cukrzycę, a próbkami pobranymi od osób zdrowych. Zaobserwowano, że w materiale zawierającym mikrocząstki u chorych z cukrzycą (UCD, CD), znajdują się zwiększone ilości rozpuszczalnego receptora dla naczyniowego śródbłonkowego czynnika wzrostu typ 3 (VEGFR3) (p=0,004) w przeciwieństwie do grupy kontrolnej. Natomiast analiza osocza pozbawionego mikrocząstek pokazała, że w próbkach pochodzących od chorych na cukrzycę obecne są zwiększone stężenia zasadowego czynnika wzrostu fibroblastów (bFGF) (p=0,03) oraz śródbłonkowego czynnika wzrostu typ 2 (VEGF2) (p=0,01).

W grupie UCD dodatkowo zauważono jeszcze na mikrocząstkach zwiększone stężenia innych cytokin, takich jak: TNFα (czynnik martwicy guza α) GRO (onkogenny czynnik stymulujący wzrost α) oraz inhibitorów metaloproteinaz 1 i 2 (TIMP1, TIMP 2) (p<0.05).



Wnioski:

Wykazano, że mikromacierze celulozowe są przydatne do przesiewowych półilościowych oznaczeń czynników proangiogennych (w tym cytokin) w osoczu chorych na cukrzycę. Mimo, że stężenia oznaczanych czynników mieszczą się w zakresach nano i piko molowych, ich czułość pozwala na wykrycie różnic między ilością tych czynników na mikrocząstkach i w osoczu pozbawionym mikrocząstek.

11:00 Poster 17

The role of natural capping agents in the production of gold nanoparticles by direct current atmospheric pressure glow microdicharge generated in the contact with flowing liquid cathode

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Nanoparticles are the structures in which at least one of the dimensions is less than 100 nm [1]. The enormous interest in the practical application of metallic nanoparticles raises no less interest in their methods of synthesis, allowing the preparation of controlled nanostructures with the given size and shape. In view of catalytic and therapeutic properties [2], the greatest interest is observed for gold nanoparticles (AuNPs).

Gold nanoparticles (AuNPs) were prepared by newly plasma method, i.e., direct current atmospheric pressure glow microdischarge (dc-μAPGD), generated between miniature flow argon microjet and flowing liquid cathode, using chloroauric acid ($HAuCl_4$) as AuNPs precursor. The plasma reactor was presented on the Fig. 1. The addition of natural capping agents such as: gelatine and fructose to solution containing gold precursor onto AuNPs production were investigated. The optical properties, morphology and size of AuNPs were determined. The Localized Surface Plasmon Resonance (LSPR) band was observed in the UV/Vis absorption spectrum for different concentrations of the precursor in initial solution. The position of the LSPR band indicated production of AuNPs. The size of AuNPs was obtained using dynamic light scattering (DLS) method.

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11:00 Poster 18

Magnetic field assisted nucleic acid delivery into primary fibroblasts

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Magnetic nanoparticles (MNPs) are increasingly used in many biomedical applications including nuclei acids delivery. The aim of this study was to explore the potential of the plasmid DNA delivery to the primary fibroblasts using magnetic transfection complexes and gradient magnetic field, an approach known as magnetofection. The MNPs tested for size, shape, crystalline composition and magnetic behavior displayed suitable physicochemical properties for their potential use in magnetofection method. Magnetic transfection complexes formed by the self-assembly of MNPs, plasmid DNA and an enhancer allowed for rapid and efficient plasmid DNA delivery into primary mouse embryonic fibroblasts and porcine fetal fibroblasts upon application of an inhomogeneous magnetic field. Efficient transfection of these cells is important for the generation of induced pluripotent cells or cell transdifferentiation and the generation of transgenic animals. Magnetofection of the porcine and mouse fibroblasts with optimal magnetic lipoplexes resulted in improvement of the transfection efficiency in terms of the luciferase reporter gene expression and in the percent of the transfected cells as compared to lipofection. Magnetic cell labeling further increased the transfection efficacy. Specific labeling of the cell surface receptors of the mouse fibroblasts with magnetic nanoparticles, both in the adherent state and in suspension, resulted in 2-4-fold enhancement of transgene-expressing cells. Non-specific cell labeling had no effect on the efficacy of the reporter expression, despite the acquisition of similar magnetic moments per cell. In contrast to the mouse fibroblasts, in porcine fibroblasts, specific magnetic labeling of the cell surface receptors inhibited internalization and transfection efficacy. We suggest that magnetic labeling of cell-surface receptors in combination with magnetic field (nanomagnetic activation) can affect the receptor-mediated internalization of delivery vectors and be used to control nucleic acid delivery to cells.

11:00 Poster 19

SPIO doxorubicne nanocomplex as MRI contrast agent

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Superparamagnetic iron oxide nanoparticles (SPIO) are widely used in medicine as MRI contrast agents and plays important role in drug delivery systems. Doxorubicine is known as important medicine in tumor therapy. Nanocomplexes of superparamamgnetic iron oxide/ doxorubicine (SPIO/DOX) have ability to accumulate in the tumor area, so they are well suited to use in monitoring of tumor treatment by magnetic resonance imaging (MRI).

Dispersion of SPIO/DOX complexes in water and saline were investigated using Nuclear Magnetic Resonance (NMR) techniques: relaxation and imaging. Studies of proton relaxation have been performed at three magnetic fields intensities 0.4T, 4.7T and 9.4T. Relaxation times, T1 and T2 have been measured (using Inversion-recovery and CPMG pulse sequences, respectively) for samples of different concentration (mM) of investigated nanoparticles in water

and physiological saline. Relaxivity parameters r1 and r2 were calculated for measured dispersion series for each value of magnetic field.

In all studied values of transverse relaxivity (r2) are significantly higher than longitudinal relaxivities (r1). Furthermore, values of r2 are comparable in aqueous and physiological saline solutions across the series and persist above 100 mM-1 sec-1. Also it should be noted that values of r1 increase with decreasing magnetic field. Obtained results allow to conclude that SPIO/DOX can be classified as effective T2 contrast agent in wide range of magnetic fields commonly used in medical diagnosis. Aforementioned complex can also be used as T1 contrast agent in human diagnosis up to magnetic fields of 1T.

Series of T2-weighted MRI images of solution of SPIO/DOX injected into agarose gel were obtained at 9.4T using Fast Spin Echo sequence (TR = 2s, TE = 20ms). The successive images were used as a monitor of contrast area changes in function of time after injection. Results confirmed that investigated particles are efficient T2 contrast agent and have ability to easily spreading by diffusion in environment with tissue-like density.

Acknowledgements
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11:00	Poster	21

Oxygen Consumption Assay as a new tool for analysis of cellular respiration and mitochondrial function

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Oxygen Consumption Assay [HS Method] is a highly flexible 96 or 384-well fluorescence plate reader-based approach, for the direct, real-time analysis of cellular respiration and mitochondrial function. The easy-to-use MitoXpress® Xtra assay allows measurement of extracellular oxygen consumption rates (OCR) with whole cell populations (both adherent and suspension cells), isolated mitochondria, permeabilised cells and a wide range of 3D cultures including: tissues, small organisms, spheroids, scaffolds and matrixes. The assay is also suitable for measurement of isolated enzymes, bacteria, yeasts and moulds. In this assay, MitoXpress® Xtra is quenched by O2 through molecular collision, and thus the amount of fluorescence signal is inversely proportional to the amount of extracellular O_2 in the sample. Rates of oxygen consumption are calculated from the changes in fluorescence signal over time. The reaction is non destructive and fully reversible (neither MitoXpress® Xtra nor O_2 are consumed), facilitating measurement of time courses and drug treatments. Luxcel's flexible plate reader format, allows multiparametric or multiplex combination with Luxcel's other products, as well as combining with commonly available reagents to measure glycolysis, LDH, JC-1, MMP (□), ROS, and cellular ATP. For example, MitoXpress® Xtra in combination with Luxcel's pH-Xtra® – Glycolysis Assay allows the simultaneous real-time measurement of mitochondrial respiration and glycolysis and analysis of the metabolic phenotype of cells and the shift (flux) between the two pathways under pathological states**.Plate Preparation** 3D RAFT cultures were prepared with either A549 or HepG2 cells at the indicated density in 240ul DMEM / Collagen solution on a 96-well plate. RAFT cultures were formed as per manufacturer's protocol. **Oxygen Consumption Measurements.** For oxygen consumption measurements MitoXpress®-Xtra stock was prepared in 16ml of pre-warmed DMEM and culture media was replaced in each well with 150µl of this solution. Where applicable, 1µl of compound stock (150X) was added to each well. Wells were then sealed by overlaying with 100µl pre-warmed HS mineral oil to inhibit oxygen back diffusion into the sample. This is best done using a repeater pipette. The plate was then measured kinetically on a FLUOstar Omega (BMG Labtech) for 90-120mins with ~2 minute interval exciting the probe at 380nm and measuring emission at 650nm. Ratiometric measurements were performed using the following delay and gate settings. Delay 1: 30s, Gate 1: 30s, Delay 2: 70s, Gate 2: 30s. **Extracellular Acidification Measurements.** Three hours prior to measurement the RAFT culture plate was placed in a CO2 FREE incubator at 37°C, 95% humidity, in order to remove CO_2 from the plate material. Spent media was removed and 2 wash steps were performed using the Respiration Buffer (0.5 mM KH2PO4, 0.5 mM K2HPO4, 20 mM Glucose, 4.5 g/L NaCl, 4.0 g/L KCl, 0.097 g/L MgSO4, 0.265 g/L CaCl2), finally 150µl of Respiration Buffer containing pH-Xtra probe at the recommended concentration was added to each well. The plate was then measured kinetically on a FLUOstar Omega (BMG Labtech) for 90-120mins with ~2 minute interval exciting the probe at 380nm and measuring emission at 615nm. Ratiometric measurements were performed using the following delay and gate settings. Delay 1: 100s, Gate 1: 30s, Delay 2: 300s, Gate 2: 30s.Oxygen consumption provides detailed information on mitochondrial function, specifically on the activity of the electron transport chain (ETC),while extracellular acidification (ECA) informs on glycolytic flux. Measurements are conducted on standard 96-well microtitre plates, on a fluorescence plate reader, and facilitate a deep insight into the metabolic behaviour of the 3D culture and into how metabolism is perturbed by a particular compound or environmental condition. 3D cell culture facilitates the development of complex intra-cellular interactions thereby helping to narrow the gap between in vitro and in vivo biological systems. Adoption of 3D technologies has however been limited, in part due to difficulties associated with producing reproducible 3D cultures. Difficulties can also arise due to an incompatibility with certain in vitro assay technologies.

11:00	Poster	22

Nowe zastosowanie fotocytometrii przepływowej (ImageStream X Mk II) do analizy mikrocząstek w materiale klinicznym.

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Nowe zastosowanie fotocytometrii przepływowej (ImageStream X Mk II) do analizy mikrocząstek w materiale klinicznym.

Gruszczyński Krzysztof [1], Kowalik Artur [1], Stępień Ewa[2]

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Wstęp

Mikrocząstki (MP) są fragmentami błon większości komórek eukariotycznych. Dzięki mikroskopii elektronowej ich średnicę określono pomiędzy 50 nm, a 1000 nm. Są wytwarzane w komórkach na drodze egzocytarnego *pączkowania* lub *złuszczania* fragmentów błony komórkowej, dzięki czemu składają się komponentów cytoplazmatycznych oraz fosfolipidów i białek błonowych [1]. Pochodzą głównie z megakariocytów, płytek krwi, erytrocytów, monocytów, neutrofili oraz mogą być wytwarzane przez komórki nowotworowe. Na swojej powierzchni MP posiadają powierzchniowe antygeny odzwierciedlające ich pochodzenie komórkowe [4], co umożliwia wykrywanie poszczególnych subpopulacji MP.

Zasady obrazowania przy użyciu cytometru przepływowego – ImageStream X MkII

Fotocytometr przepływowy Amnis *ImageStream X Mk II* (ISX) łączy podstawowe możliwości fenotypowania klasycznych cytometrów z zaletami szczegółowego obrazowania cyfrowego mikroskopu fluorescencyjnego, poprzez precyzyjną metodę elektronicznego śledzenia poruszających się obiektów z wieloparametrowym systemem obrazowania o wysokiej rozdzielczości. W ISX zastosowano unikalną technikę (*Time Delay Integration*), która opiera się na wykorzystaniu matrycy CCD z filtrem rozpraszającym wiązkę światła. Dzięki połączeniu metody naprowadzania fal świetlnych o odpowiedniej długości do określonych obszarów chipa CCD oraz nowatorskiemu systemowi detekcji można zebrać 1000 razy więcej danych o źródle światła jakim są obiekty poddane analizie cytometrycznej, niż w konwencjonalnych technikach. Umożliwia to zapis obrazów przy bardzo niskim poziomie światła [2].

Cel pracy

Celem pracy było wykorzystanie metody fotocytometrii przepływowej do obrazowania obiektów biologicznych (MP) o rozmiarach na pograniczu rozdzielczości optycznej tradycyjnych urządzeń pomiarowych. W tym celu przeprowadzono kalibracje urządzenia na 2 niezależnych systemach opartych o zestawy polistyrenowych monodyspersyjnych kulek kalibracyjnych.

Metody

Urządzenie. Analizę wykonywano przy użyciu metody cytometrii obrazowej drugiej generacji (ImageStream X Mk II, Amnis Corporation, Seattle, WA). Fotocytometr został wyposażony w dwie kamery CCD TDI o wysokiej rozdzielczości każda. Pozwala to na równoczesna pracę z maksymalnie 9 sondami fluorescencyjnymi oraz pozyskanie do 12 obrazów danego obiektu: obraz odzwierciedlający ziarnistości tzw. SideScatter/ Darkfield Channel, dwa obrazy światła przechodzącego (Brightfield Channel)**,** oraz do dziewięciu obrazów fluorescencji. Dostępne powiększenia 20x, 40x lub 60x pozwalają uchwycić szczegóły pojedynczych obiektów o wysokiej rozdzielczości (1200 pcs) dla setek tysięcy z nich [8]. Program wylicza zarówno intensywność jaki i położenie sond fluorescencyjnych oraz umożliwia analizę bardzo różnorodnych próbek i rzadkich subpopulacji bez konieczności wzbogacania próbki przed analizą. Udoskonalona technika przepływu umożliwia przechwytywanie ponad 95% wszystkich obiektów, a prędkość zapisu może przewyższać 1000 obiektów na sekundę.

System ISX łączy obrazy światła przechodzącego z mocą czterech laserów: niebieski laser 488 nm o mocy 200 mW, czerwony 642 nm o mocy 150 mW, fioletowy 405 nm o mocy 120 mW, oraz 70 mW laser 785 nm (*Side Scatter* - SSC). Wszystkie lasery, które były używane podczas kalibracji i eksperymentów na materiale biologicznym działały z maksymalną mocą. Obraz obiektu dzielony jest na sześć kolorów składowych poprzez unikalny element rozkładu widma (matryca CCD). ISX jest wyposażony w kilka powiększeń (20, 40, i 60x). W badaniach używano powiększenia 60x o aperturze numerycznej 0,9 z rozdzielczością obrazu około 0,3 x 0,3 μm/pixel. Ponieważ model ten posiada dwie matryce CCD (każda z sześcioma kanałami detekcji sygnału) niezbędne są dwa kanały światła przechodzącego (*BrightField* - BF). Umożliwia to koordynację przestrzenną między matrycami. Intensywność tła dla BF została ustawiona na 800 dla obydwu matryc.

Oprogramowanie. System ISX wykorzystuje oprogramowanie analityczne *IDEAS*^{TM} (Amnis Corporation, Seattle, WA), które oblicza ponad 40 cech ilościowych dla obrazu, czyli do 480 funkcji na komórkę. Cechy te mogą być wykorzystywane przez badacza do wygenerowania histogramów i wykresów korelacji, podobnie jak w klasycznej analizie danych. Zróżnicowane funkcje pozwalają identyfikować populacje na podstawie obrazów opierając się nie tylko na intensywności fluorescencji czy wielkości komórek, lecz również na kształcie i teksturze [2]. Zidentyfikowane grupy mogą być charakteryzowane z wykorzystaniem statystyk populacji np.: średnich, mediany, odchylenia standardowego oraz standardowych testów statystycznych. Prawidłowe badanie ilości MP wymaga jak największego wykluczenia zakłóceń tła. Zaletą ISX jest wykorzystana w oprogramowaniu funkcja pomijania *SpeedBead*® podczas akwizycji (mikrosfery, służące do autokalibracji urządzenia w czasie pracy) [8]. Oprogramowanie *IDEAS*^{TM} pozwala za pomocą narzędzia *Merge .cif files*, złożyć w jedną bazę zapis osobnych plików bramkowanych pomiarów, co wykorzystano przy kalibracji zestawami *Sphero*^{TM}.

Kalibratory. Analiza danych w klasycznych cytometrach przepływowych opiera się na dwóch parametrach *Forward Scatter* (FSC) oraz/lub *SSC*. W ImageStream X Mk II kanał FSC zastąpiono kanałem światła przechodzącego tzw. *Brightfield Channel*. Ma to bezpośredni związek z wyborem rodzaju kalibratora. Do kalibracji urządzenia wykorzystano 2 zestawy kulek kalibracyjnych wykorzystywanych do kalibracji ustawień cytometrów przepływowych w analizie MP:

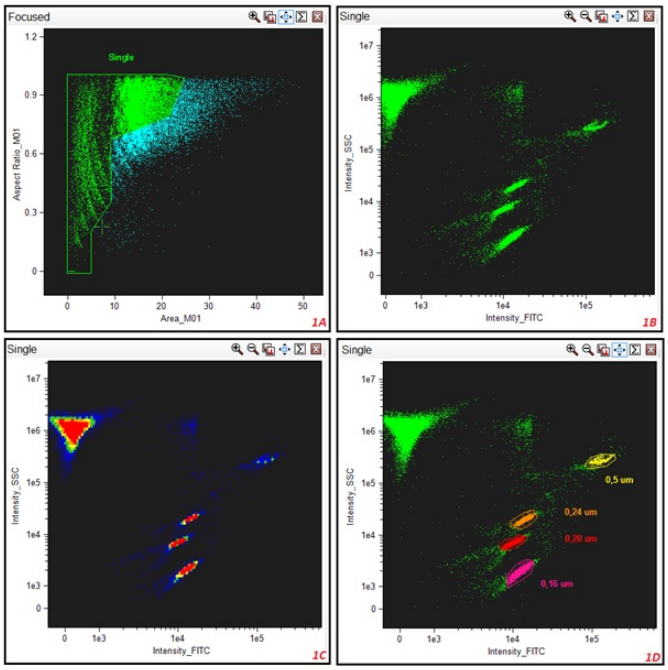
1.Zestaw *Megamix–Plus SSC* (cat no. 01077 – Biocytex, Marseille, France) dedykowany do urządzeń opierających analizy na SSC jako głównym parametrze analizy. *Megamix–PlusSSC* to mieszanina fluorescencyjnych kulek o średnicach: 160, 200, 240, oraz 500 nm, tak dobranych, aby pokrywać się z przewidywanymi rozmiarami MP (0,1 do 1 μm), wykorzystując SSC jako pochodną wielkości. Dodatkowo obiekty związane są z izotiocyjanianem fluoresceiny (FITC) – fluorochromem o długości fali emisji w przedziale 480 – 560 nm. W przypadku ISX MkII generowany jest zielony obraz fluorescencji odczytywany na kanale drugim (*Ch02*).

2.Zestaw kalibracyjny - *Sphero*^{TM} *Flow Cytometry Nano Fluorescent Size StandardKit* 0.1-0.3 μm, 0.4-0.6 μm, 0.7-0.9 μm & 1.0-1.9

µm (cat no. NFPPS-52-4K, Spherotech Inc, Lake Forest, IL): cztery oddzielne grupy kulek o średnicach: 220, 450 i 880 nm oraz 1,33 µm. Do określenia parametrów optycznych najmniejszych obiektów zastosowano kalibrator *Nano Fluorescent Size Standard* kit 0.05-0.15 µm (cat no. NFPPS-0152-5, Spherotech Inc, Lake Forest, IL) o średnicy 130 nm.

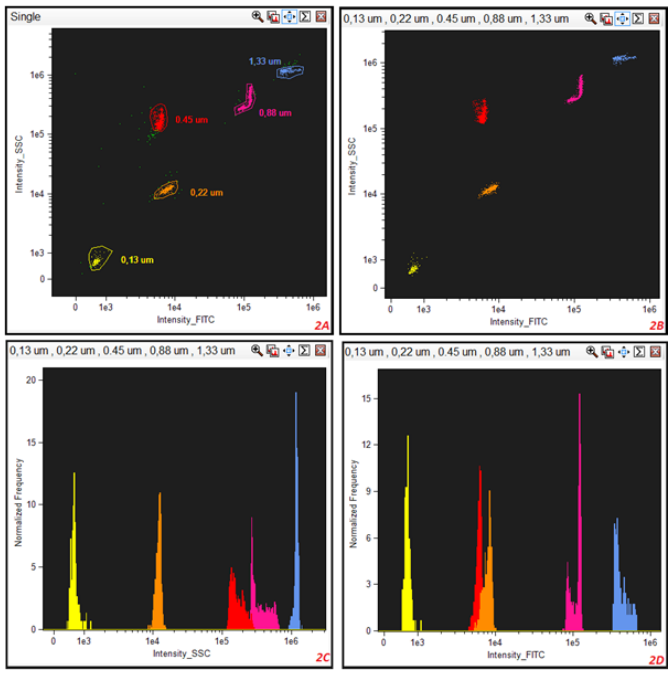
Algorytm akwizycji obrazu i analizy danych.

1. Dla zestawu *Megamix–Plus SSC* zapis prowadzono z obszaru obejmującego pojedyncze obiekty *(Single)*. Bramkę utworzono na wykresie punktowym *Area_BF* do *Aspect_Ratio_BF* (Rys. 1a). Zapisano plik zawierający 60 000 obiektów. Grupa ta posłużyła za populację wyjściową do analizy w programie *IDEAS*^{TM}. W oparciu o populację *Single* wygenerowano wykres punktowy intensywności FITC do intensywności SSC (Rys.1b). Wykorzystując odczyt zagęszczenia obiektów (Rys. 1c) oznaczono wszystkie poszukiwane populacje na tle pozostałych obiektów z bramki *Single* (Rys 1d).



Rys 1. Algorytm akwizycji danych dla zestawu kalibracyjnegoMegamix–Plus SSC do analizy mikrocząstek za pomocą fotocytometru przepływowego Amnis ImageStream X Mk II.

2.Dla zestawu *Sphero*^{TM} zapis kalibracji wykonano na identycznych ustawieniach urządzenia jak w przypadku pierwszego zestawu, a analizę przeprowadzono według tych samych procedur dla każdej populacji kulek osobno. Do analizy zebrano dane dla 5000 obiektów. Za pomocą narzędzia *Merge .cif files* złożono w jedną bazę zapis osobnych plików bramkowanych pomiarów, co przedstawiono na wykresie punktowym (Rys. 2 a,b) i histogramach pomocniczych (Rys 2 c,d).:



Rys 2. Algorytm akwizycji danych dla zestawów kalibracyjnych SpheroTM Flow Cytometry Nano Fluorescent Size Standard do analizymikrocząstek za pomocą fotocytometru przepływowego Amnis ImageStream X Mk II.

Wyniki

Standardowa procedura analizy w programie *IDEAS*^{TM} pozwala odrzucić obiekty nieostre oraz aglomeraty przeprowadzając użytkownika przez kilka prostych histogramów i wykresów punktowych. Stąd różnice między liczbą zapisanych obiektów, a rzeczywistą ilością analizowanych obiektów.

Na postawie danych ilościowych uzyskanych w wyniku akwizycji w oparciu o opisane algorytmy uzyskano procentowy rozdział wszystkich populacji kulek kalibratora Megamix–Plus SSC. Wyniki w porównaniu z wartościami referencyjnymi dostarczonymi przez producenta zaprezentowano w Tabeli 1.

	Megamix – Plus SSC	% Mgx	ImageStream X MkII	% ISX
All events	56 783	100	13 286	100
0,50 µm	4 023	7,0	536	4
0,24 µm	9 680	17,0	2516	19
0,20 µm	14 062	24,8	4114	31
0,16 µm	29 018	51,1	6120	46

Tabela 1. Porównanie liczby obiektów oraz ich udziału procentowego dla kalibratora Megamix–Plus SSC oraz cytometru ISX MkII.

Instrukcja do zestawu *Sphero*^{TM} zawiera porównanie wyników dla różnych cytometrów przepływowych [10]. Rozdział populacji jaki otrzymaliśmy na ISX jest zbliżony do danych przedstawionych w instrukcji (Tabela 2).

	Sphero - merged .cif files	%	Sphero - mixed objects	%
All events	3262	100	3616	100
1,33 µm	771	23,64	804	22,23
0,88 µm	977	29,95	828	22,90
0,45 µm	866	26,55	1572	43,47
0,22 µm	392	12,02	370	10,23
0,13 µm	256	7,85	42	1,16

Tabela 2. Porównanie liczby obiektów oraz ich udziału procentowego dla kalibratora Sphero™ oraz cytometru ISX MkII.

Wnioski.

Stale przyśpieszający rozwój technologii oferuje coraz nowsze narzędzia diagnostyczne. Nowoczesna cytometria przepływowa, a zwłaszcza system *ImageStream X Mk II*, który łączy w sobie możliwości wysoce dokładnego cytometru przepływowego z mikroskopem fluorescencyjnym umożliwia dokładniejsze zliczanie oraz analizę obiektów tak małych jak np. bakterie czy mikrocząstki. Jest to o wiele bardziej czuła metoda niż standardowa cytometria przepływowa. Poza obrazowaniem fluorescencyjnym umożliwia morfologiczną charakterystykę obiektów. Co ważniejsze, program *IDEAS™* pozwala na analizę danych, przy pomocy ogólnie znanych z klasycznych cytometrów narzędzi - np. histogramów i wykresów punktowych, co czyni analizę łatwiejszą do przeprowadzenia. System pozwala także obejrzeć dowolny obiekt, po wcześniejszym wybraniu na wykresie odpowiadającego mu punkt pomiarowego.
Zastosowanie dwóch zestawów kalibratorów miało na celu uzyskanie powtarzalnych wyników przy użyciu dwóch rożnych systemów do kalibracji urządzeń, gdzie znane są parametry obiektów. Dodatkowo, analiza populacji kulek o średnicy 130 nm dostarczyła informacji o granicy czułości urządzenia i pozwoliła na powiększenie obszaru akwizycji na korzyść obiektów mniejszych niż 150 nm. Badanie populacji 1,33 µm umożliwiło powiększenie obszaru zaczytywania mikrocząstek o obiekty zbliżone rozmiarami do górnej granicy wielkości MP.

Otrzymane w wyniku przeprowadzonej kalibracji ustawienia urządzenia ISX posłużą do przeprowadzenia analizy biologicznych MP uzyskanych z próbek klinicznych, takich jak osocze, surowica i inne płyny fizjologiczne.

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11:00 Poster 23

Wykorzystanie metody NTA (Nanoparticle Tracking Analysis) w ocenie wielkości krążących mikrocząstek osocza i innych płynów fizjologicznych: pułapki i triki

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Wstęp: Krążące mikrocząstki (MP) osocza są to mikropęcherzyki, których głównym źródłem są płytki krwi, komórki śródbłonka, makrofagi oraz inne komórki krwi. Na ich uwalnianie mają wpływ czynniki zapalne, niedotlenienie, uszkodzenia mechaniczne oraz bodźce wywołujące stymulację komórek naczyń, takie jak hiperglikemia oraz hiperhomocysteinemia. Ponadto komórki nowotworowe wytwarzają i uwalniają dużo MP, które mogą być wykrywane w krwi i innych płynach ustrojowych. Przypuszcza się, że za pośrednictwem MP komórki wymieniają między sobą "informację" w postaci uwalnianych i transportowanych na długie dystanse makromolekuł. Istotną rolą MP jest ich udział w procesach hemostazy, angiogenezy oraz organogenezy.

Rozwój diagnostyki laboratoryjnej zmierza z jednej strony do poszukiwania nowych markerów diagnostycznych, z drugiej strony do miniaturyzacji urządzeń, wprowadzania do klinicznego laboratorium diagnostycznego nowych metod, wykorzystywanych w nanotechnologii.

Do oceny ilościowej i jakościowej MP możemy wykorzystać zarówno metody molekularne i powszechnie stosowane techniki mikroskopowe, wykorzystujące zjawisko fluorescencji specyficznych barwników- etykiet dla białek lub kwasów nukleinowych (cytometria przepływowa, mikroskopia konfokalna, techniki mikromacierzy). Coraz częściej sięga się w diagnostyce laboratoryjnej po metody wykorzystywane w nanotechnologii, głównie w przemyśle chemicznym, takie jak: NTA (Nanoparticle Tracking Analysis), Z-NTA (Zeta Potential Nanoparticle Tracking Analysis), TRPS qNano (Tunable Resistive Pulse Sensing- Izon particle sizing) oraz DLS (Dynamic Light Scattering). Wszystkie metody pozwalają określić wielkość MP, agregację cząstek oraz przedstawić w sposób ilościowy rozkład wielkości MP w próbce. Jedynie metody Z-NTA oraz NTA pozwalają zmierzyć koncentrację MP w oznaczanym materiale.

Metoda NTA oraz DLS wykorzystują analizę ruchów Browna, będących nieuporządkowanymi i chaotycznymi ruchami cząstek w roztworach cieczy. Metoda NTA skupia się na analizie odległości przebytej przez cząstkę, podczas gdy DLS koncentruje się na analiz-

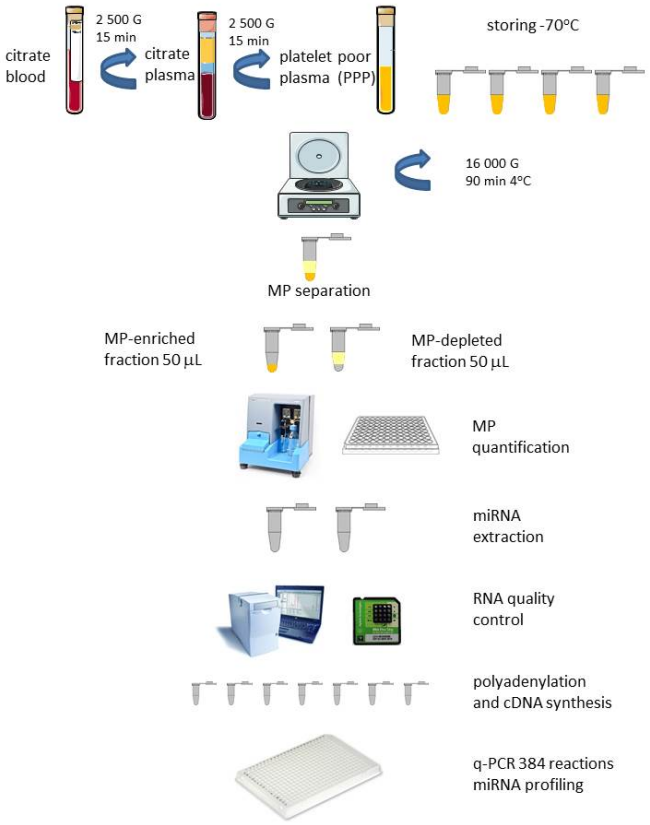
ie intensywności światła rozproszonego poprzez poruszające się MP. Nieodzownym pojęciem związanym z metodą Z-NTA oraz qNano jest zeta-potencjał, który ma zasadnicze znaczenie dla określenia stopnia wzajemnego odpychania się cząstek.

Metoda NTA mierzy cząstki z zakresu od 10-40 nm do 1-2µm. Minimalna koncentracja, którą można oznaczyć przy użyciu tej metody to około 10^6cząstek/mL, natomiast wartość maksymalna to około 10^{10}cząstek/mL. Istotną różnicą pomiędzy metodą NTA i DLS jest pomiar wielkości MP w próbkach polidyspersyjnych. Pierwsza metoda ukazuje piki przy wielkościach dominujących MP badanej mieszaniny, natomiast w DLS uzyskuje się jedynie wartość średnią. Dodatkowo metoda NTA umożliwia analizę cząstek o właściwościach fluorescencyjnych oraz pozwala wykorzystać lasery (niebieski i zielony) zdolne wzbudzić zarówno fluorofory jak i kropki kwantowe.

W alternatywnej metodzie qNano, w sposób bezpośredni mierzy się ładunek cząstek przechodzących przez nanopory, który jest proporcjonalny do objętości nanocząstki lub mikrocząstki. Wykorzystuję się zasadę pomiaru spadku potencjału **(ΔR)** po przejściu mikrocząstki o objętości **d3** przez porowatą membranę o średnicy pora **D**, gdzie **ρ** oznacza opór właściwy cieczy (elektrolitu) w jakiej cząstki są zawieszone.

$$\Delta R = \frac{4\rho d^3}{\pi D^4}$$

Cel: Założeniem przeprowadzonych badań była charakterystyka metody NTA w celu późniejszych zastosowań tej metody do oceny wielkości krążących MP osocza i innych płynów ustrojowych. Metody: Pobrane próbki krwi odwirowano dwukrotnie przez 15minut w 2500G w temperaturze pokojowej w celu uzyskania osocza ubogopłytkowego (PPP). PPP następnie odwirowano przez 90minut w temperaturze 4°C w 16000G, aby uzyskać frakcję MP. Zarówno frakcję MP jak i nadsącz zbadano przy użyciu analizatora NS500 (Nanosight Ltd, Amesbury, Wielka Brytania), w celu określenia wielkości krążących MP. W urządzeniu NS500 wyposażonym w laser niebieski o długości fali 405nm wykorzystywane było zjawisko rozproszenia światła na MP zawieszonych w medium (rozpuszczalniku) o różnych właściwościach fizykochemicznych, w temperaturze 23,3°C.



Analizowane próbki rozcieńczono 200- oraz 1000- krotnie w:

- W- woda destylowana
- S- sól fizjologiczna

- ABB- bufor do cytometrii przepływowej- Annexin V binding buffer (BD Biosciences, USA), w skład którego wchodzi 0.1M Hepes (pH 7.4), 1.4M NaCl oraz 25mM roztwór $CaCl_2$. Przed dokonaniem pomiaru ABB rozpuszczono 10-krotnie w wodzie destylowanej, zgodnie z zaleceniem producenta.

Wyniki:

1. W próbkach, w których jako rozpuszczalnik zastosowano wodę, zarówno w nadsączu jak i we frakcji MP, wykryto cząstki wielkości 100nm, tak zwane małe MP (mMP), jak również zaobserwowano duże MP (dMP) o średnicy 300-650nm. Średnia wielkość MP w nadsączu wyniosła 157,00nm, a zakres wielkości MP 20-600nm, podczas gdy średnia wielkość MP we frakcji wyniosła 175,60nm, a zakres wielkości 30-650nm.
2. Użycie soli fizjologicznej jako rozpuszczalnika spowodowało, że w obu pomiarach dMP były wykrywane. Średnia wielkość MP w nadsączu wyniosła 160,00nm, podczas gdy we frakcji MP wyniosła 146,70nm. Zakres wielkości MP w nadsączu wynosił 20-450nm, natomiast we frakcji wynosiła MP 30-500nm.
3. Wykorzystanie buforu do cytometrii spowodowało, że dokonując pomiaru można było uzyskać rezultat przeprowadzonego wirowania. Tylko we frakcji MP można było zaobserwować dMP. Średnia wielkość MP we frakcji wyniosła 110,20nm, podczas gdy w nadsączu wynosiła 89,69nm.

Wnioski: Uzyskane wyniki pokazały, że dobór rozpuszczalnika ma wpływ na uzyskane rezultaty. Właściwości użytych rozpuszczalników w sposób pośredni mogą wpłynąć na wielkość badanych MP. Jedynie zastosowanie buforu do cytometrii, który posiada właściwości sprzyjające agregacji cząstek, umożliwił uzyskanie wyników pokazujących skuteczność wirowania.

Zastosowanie wody destylowanej jako rozpuszczalnika mogło wywołać zjawisko agregacji MP bądź osmozy. Wykazano, że rozkład wielkości badanych MP był znacząco większy w odniesieniu do wyników uzyskanych przy użyciu innych rozpuszczalników.

Izotoniczny charakter soli fizjologicznej zapobiega agregacji cząstek oraz zjawisku osmozy. Mając na uwadze właściwości PBS-u oraz uzyskane wyniki, należy zastanowić się czy stosowane parametry wirowania (16000G) są wystarczające do sedymentacji dMP do frakcji MP, w celu wyeliminowania ich z nadsączu.

Zanalizowanie rozkładu wielkości MP stanowi etap poprzedzający analizę fenotypową MP. Wykorzystanie metody NTA pozwala potwierdzić skuteczność przeprowadzonego wirowania, co jest niezbędną procedurą do rozpoczęcia badań dotyczących antygenów powierzchniowych MP przy użyciu cytometrii przepływowej, oraz zbadania ekspresji białek i genów przez nie transportowanych, przy użyciu odpowiednio metody Western Blot oraz qPCR.

11:00	Poster	24

Hydrophilic multi walled carbon nanotube-Fe composites as mri contrast agents

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Carbon nanotubes (CNTs) have been extensively investigated in many fields of science since their discovery. Short, functionalised, multi-walled tubes filled with magnetic particles are suspect harmless to human beings and have been seen to be able to penetrate cellular membranes without inducing apoptosis. Multi-walled CNTs containing different amounts of iron filling were synthesised. Afterwards, various functionalisation protocols were explored and compared in order to identify the most effective for producing a highly soluble and biocompatible material. The functionalised structures were separated by length.

The properties of sorted nanohybrid structures were characterised by several techniques; scanning electron microscopy, high resolution transmission electron microscopy, Superconducting Quantum Interference Device, NMR and Magnetic Resonance Imaging. For cytotoxicity analysis of carbon nanotubes MTT, WST-1 and Neutral Red Uptake assays were performed. The cytotoxicity was evaluated by determining the viability of HeLa and human fibroblast cells after incubation in media containing MWCNTs with different Fe concentrations.

Significant correlations and enhancements in MRI contrast were found when testing the particles with different lengths, iron content and dispersion type. Our data indicated that those nanoparticles have very low or no cytotoxicit. We believe that these findings are highly promising and further research might lead to tissue-selective or externally guided, “intelligent”, MRI contrast and drug delivery agents.

11:00	Poster	25

Azopolymer nanostructures with various morphologies.

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Polymer nanomaterials are interesting topic, because of their unique electronic, optical and thermal properties. The nanostructures play a significant role in development of materials engineering, due to their potential applications, eg. systems of drug delivery, in photovoltaics or sensors. The main advantage of nanomaterials is ability to obtain specified properties by controlling the preparation process at a molecular and atomic level.

This work presents synthesis and photochromic properties of new azopolymers containing azo-derivatives of sulfamerazine. The obtained methacrylate polymers and copolymers were used to form nanostructures with various morphologies: spheres, tubes, fibers. The azo spheres were prepared by gradually adding water to the polymer solution. The azo tubes were obtained by template method. Moreover, we used the electrospinning process for fibers preparation.

The photochromic properties of obtained materials were investigated by UV-Vis spectroscopy. Based on the resulting UV-Vis spectra and the exponential equations, we estimated the rate of changes in the obtained materials occurring under illumination and the rate of the thermal relaxation in the dark. Moreover, the complex refractive index and the changes of the real part of refractive index after illumination in polymer films were determined by ellipsometry.

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Index

L

M

N

O

P

R

S

T

V

W

Y

Z