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SELF-ASSEMBLED Ge/METAL CORE/SHELL NANOPARTICLES IN ALUMINA MATRIX

Lovro Basioli[1], Vito Despoja[2], Jordi Sancho Parramon[1], Stjepko Fazinić[1], Nikolina Nekić[1], Sigrid Bernstroff[3], Goran Dražić[4], Mile Ivanda[1], Maja Buljan[1]

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Core-shell structured Ge nanoparticles (quantum dots) with metallic shells (aluminium, tantalum and titanium) in an amorphous alumina matrix were investigated. The presence of metallic shells just a few atoms in thickness has shown to radically weaken oxidation of germanium but occurrence of intermixing Ge-metal or Ge-metal-O phases is implied. Greater amounts of metal in some samples caused shells to overlap forming a slab-like structure also causing loss of self-assembly. In addition, these metallic shells dramatically influence the dielectric function and absorption coefficient. Most important absorption peak energies in investigated systems are in the relevant part of solar spectrum (1 - 4.5 eV) which suggests applications of these materials in photovoltaic devices are possible. Samples with self-assembled super-lattice were created using magnetron sputtering at 300°C. Characterization methods used are Elastic recoil detection (ERDA), Transmission electron microscopy (TEM), Grazing incidence small angle X-ray scattering (GISAXS), X-ray absorption near edge structure (XANES), Raman spectroscopy and Ellipsometry.

BETWEEN THIN FILM RESEARCH AND JOB COATING SERVICE

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A typical thin film department at an institute is interested in depostion, characterization, property interpretation, but generally without immediate industrial application. A typical job coating facility on the other hand is primarily a service with all the R&D subordinated to the needs of this service. Doing both activities in one place is relatively rare; in this paper the experience of this kind of synergy will be shown as has been in operation at the Jožef Stefan Institute for over 30 years. Thin film R&D is usually understood as starting from a purely experimental lab environment, to be step-by-step upgraded to an industrial environment and implemented in daily production at a factory. Skipping the first step, i.e. doing research in an industrial chamber, has some clear benefits, such as working in a more reallistic environment, expecially as it is driven by the industrial demands. These demands may even trigger new research which might not be the case in a standard lab environment. Two such cases will be shown: (i) a warranty claim of poor coating quality initiated a successful research of growth defects, (ii) a similar complain of multilayer coating reproducibility was followed by an extensive work on modeling of thin film growth in a multi-rotation system. However, there are also drawbacks in such an industrial-driven approach. In addition to the strict deadlines, required by the partners, perhaps the biggest obstacle is a recluctance to implement novel coatings in favor of proven solutions.

PLASMA PRE-TREATMENT INFLUENCE ON COLOR YIELD OF TEXTILE SUBSTRATE

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Due to its efficiency, digital InkJet printing technology, has demonstrated wider possibilities over the customary textile printing methods, such as excellent pattern quality, considerably little pollution, quick response to the frequent shifts on textile product market the reduce of the overall printing costs. However, there are still many open areas in the field of digital InkJet textile printing technology that requires intensive solution finding research in order to overcome the still existing barriers. By following an integrated approach the benefits of textile ink jet printing can maximized by improving the final printed image quality (including print definition), colour yield, colour fastness and the "handle" of the final textile ink jet printed product. Considerable research and developments are now taking place on printing inks composition, print head developments but also on the complex droplet/ substrate interaction and the droplet breakup process while interacting with printed medium, which is of key importance for reproducing digital colour [1, 2].

This paper describes the influence of textile surface pre-treatment using low-pressure plasma on colour spectral parameters of printed surface, which depends on the quantity of bounded pigments as well as on structure of the polymer layer on the surface of textile material. The pre-treatments is required to achieve the optimal benefits of textile ink jet printing by improving the final printed image quality (including print definition), colour yield, colour fastness and the "handle" of the final textile ink jet print. Pre-treatment of textile substrate (raw cotton and man-made cellulosic knitwear for clothes) were performed using low-pressure plasma type Nano LF-40 kHz using oxygen and argon as working gases under optimised plasma processes for fibre surface activation (power of 500W, pressure of 0.34 mbar, gas flow rate of 50 sccm and time duration of 2 minutes). Ink-jet (with Micro Piezo Head) digital textile printer, type Azon Tex Pro that prints directly on fabric, was used in experiments. For a better contribution and explanation of the results of surface analysis, SEM microscope analyse was carried out. For objective evaluation of colour, the Data Color Spectra flash SF+ 600 remission spectrophotometer was used, and for expression of colour differences the CIE 76 as first colour difference formula that related a measured to a known set of CIELAB coordinates, was performed. Printed image quality and colour fastness were analysed through Wash test and Abrasion test (using Martindale apparatus) according to standardized testing methods. Obtained results showed positive impact of oxygen/argon plasma pre-treatment with improving the total colour yield on the surface of substrate, because of better wettability and hydrophilicity of such pre-treated samples. The results showed significant differences in colour characteristics of pre-treaded textile samples in compare to untreated ones. One of the reasons of such results is that plasma pre-treatment allows deeper embedding of the printing ink polymer film into a structure of a fibre which further allows more firm and durable bonds of pigment with fibrous substrates and enlarges the active surface for pigment bonding.

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INFLUENCE OF PREPARATION PARAMETERS ON MORPHOLOGY AND STRUCTURE OF TiO_2 DEPOSITED AT ZnO NANORODS FOR PHOTOVOLTAIC APPLICATION

A. Gajović[1], I. Panžić[1], K. Juraić[1], N. Krstulović[2], D. Belić[1], M. Plodinec[1,3], D. Gracin[1], A. Šantić[1], M. G. Willinger[3]

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Nanostructured titanium dioxide is widely used for photovoltaic applications, primarily as electron transporting layer in perovskite and dye sensitized solar cells, basically due to suitable band gap for acceptance of electrons from active layer of solar cells. However, by combination of high reactivity of TiO_2 together with the large binding energy of ZnO, the process of electron transfer between the corresponding conduction and valence bands can be facilitated in the composite system. Different techniques have been reported for the growth of ZnO/TiO_2 nanocomposites in various forms.

With the aim to prepare high performance TiO2@ZnO core–shell nanostructure for photovoltaic application, in this work TiO_2 was deposited on ZnO nanorods by pulsed laser deposition (PLD) and magnetron sputtering (MS). We studied influence of the preparation parameters to crystal structure and morphology of nanocomposite and correlate them with the optical and electrical properties.

ZnO nanorods (ZNR) arrays were prepared by sol-gel procedure followed by annealing of nanorods to obtain crystallinity. TiO_2 thin films were prepared on ZNR using PLD in Ar or vakuum and by reactive MS with the aim to select the preference deposition procedure. The structural phase of TiO_2 were primary studied by confocal micro-Raman spectroscopy (mRS), while morphology and crystal structure on nanoscale were studied by high resolution transmission and scanning electron microscopy (HRTEM and HRSEM). The optical properties are evaluated by UV-vis spectroscopy, while for electrical characterization impedance spectroscopy was applied.

Raman spectroscopy results indicated that TiO_2 thin films prepared by MS and PLD were amorphous after deposition. The annealing temperature for crystallization were optimised to obtain anatase structure, since mRS study showed the formation of rutile already at temperature between 450 and 500 °C. It was shown that morphology of the TiO_2 layers obtained by PLD considerably depend on preparation atmosphere and the number of pulses. In the case of preparation by MS the attention should be focused on reactive magnetron atmosphere as well as on the pre-treatment of the ZNR substrate. The obtained phase composition and morphology of the TiO_2 will be discussed in the frame of the preparation parameters, while optical and electrical properties of prepared TiO_2@ZnO core–shell nanostructure will be discussed in the view of structure and morphology of the obtained TiO_2 layers.

Acknowledgements: This work has been supported by Croatian Science Foundation under the project IP-2014-09-9419 and by Croatian-Germany DAAD bilateral project.

FORMATION OF OXIDES ON CoCrMo SURFACES AT ROOM TEMPERATURE: AN XPS STUDY

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The cobalt-chromium (CoCr) based alloys belong to one of the most important class of metallic biomaterials, primarily due to the exceptional strength and corrosion protection, slow wear rate and good biocompatibility. We have employed x-ray photoemission spectroscopy (XPS) in order to examine the RT oxidation behaviour of CoCrMo alloy, either exposed to oxygen atmosphere or bombarded with low energy oxygen ions. The results are compared with the oxidation of pure Co, Cr and Mo metals. In contrast to the formation of only few monolayers of oxides during the RT oxidation of pure metals in oxygen atmosphere, the oxidation of all three metals from CoCrMo follows the logarithmic growth kinetics that involves the electric field driven migration of metal cations or oxygen anions. On the other hand, oxygen ion bombardment of CoCrMo provides a more efficient oxidation path and creates thicker oxide films composed of oxides of all three metals, such as CoO, Cr2O3 and MoO3, with an additional spinel component, CoMoO4. Ion-induced oxidation follows the parabolic growth rate, characteristic for the diffusion of cations or anions through the charged defect sites, such as singly or doubly charged vacancies or interstitials.

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THERMOSALIENT MYSTERY

Teodoro Klaser[1], Željko Skoko[1], Ivor Lončarić[2], Panče Naumov[3] and Michele Zema[4]
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Molecular crystals, capable of fast and reversible change of shape in the form of jumping, twisting, curling, bursting and bending are quickly emerging as perspective actuators on the nano/microscale. These thermosalient materials (or more colloquially known as jumping crystals) can by the collective motion of their atoms use external energy provided as heat and transfer it into mechanical motion – work. In contrast to their polymer counterparts, in single crystals this process happens instantaneously. With the main aim of getting rapid and reversible motion of such materials, triggered by external heat, is at the frontier of the material science research. Here we present our results on three thermosalient systems: oxitropium bromide, methylscopolamine bromide and N'-2-propylidene-4-hydroxybenzohydrazide.

Oxitropium bromide and methylscopolamine bromide have very similar molecular structures, the only difference being that one ethyl group is replaced by methyl group in the case of methylscopolamine bromide. Both compounds have medical uses, oxitropium bromide is used as a bronchodilator, whereas methylscopolamine bromide used to prevent nausea and vomiting caused by motion sickness. They also both exhibit thermosalient effect – unexpected and abrupt jumping of the crystals during heating and cooling. This is where the difference stops. In the case of oxitropium bromide thermosalient effect is caused, as in most other thermosalient compounds, by the topotactic phase transition during which unit cell changes drastically thus causing the crystals to jump to heights several times larger than their dimensions.

On the other hand, surprisingly, methylscopolamine bromide does not seem to show any phase transition at all, but yet, its crystals are also joyfully jumping around during the heating or cooling of sample.

In the third system, N'-2-propylidene-4-hydroxybenzohydrazide, there is not one, but two thermosalient phase transitins. Our theoretical calculations prove that in this case thermosalient effect is caused by yet another unusual property – negative compressibility, which causes softening of low-energy phonons.

These three systems which all exhibit thermosalient behavior, but which is caused by different mechanisms, demonstrate the complexity and mystery of this phenomenon.

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PLASMA STRUCTURES IN MAGNETRON SPUTTERING DISCHARGES

Matjaž Panjan

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Magnetron sputtering is widely used plasma technology for the deposition of thin films and coatings. Despite its longstanding use in the laboratory research and industrial production, the physics of magnetron discharges is not fully understood. Research in recent years has shown that magnetron plasmas are not azimuthally homogenous, instead, they exhibit distinct plasma structures that are called spokes. Spokes are observed for a wide range of discharge conditions (i.e., pressures, currents), magnetron geometries and are an essential feature of all operational regimes, including DCMS, HiPIMS and RFMS [1-3]. These structures commonly have an arrowhead-like shape with the arrow pointing in the **E**×**B** direction and travel with azimuthal velocities of several km/s. Studies of the spoke phenomenon have changed our understanding of several physical processes in the magnetron discharges. In the talk, I will present our efforts to understand the physics of spokes and magnetron plasmas in general. I will discuss the plasma potential distribution [4] and the influence it has on the transport of charged particles [5], sputtering process and overall sustainability of the discharge. I will demonstrate that electric fields associated with spokes cause re-energization of electrons and thus sustain the spoke and the discharge. Spokes also influence the ion energy distribution and therefore indirectly affect the thin film growth.

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IN SITU SYNTHESIS OF LATERAL HETEROSTRUCTURES OF HEXAGONAL BORON NITRIDE AND GRAPHENE ON Ir(111)

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Investigation of graphene, hexagonal boron nitride (hBN), transition metal dichalcogenides and other modern 2D materials is of great interest due to many potential technological applications these materials have. Furthermore, by combining 2D materials into vertical or lateral heterostructures, new opportunities arise for engineering of new systems on the micro- and nano-scale with specific properties. Motivated by this, in this talk a study on lateral heterostructures of hBN and graphene on iridium surface will be presented.

Synthesis of the heterostructures was monitored in real time with in situ low-energy electron microscope (LEEM). The procedure used for the synthesis was sequential chemical vapour deposition (CVD), where two precursors (borazine and ethylene) were dosed onto Ir(111) surface at 900 °C one after the other. This resulted in the growth of 2D lateral heterostructures consisting of well-defined hBN islands surrounded by graphene. However, the interface between hBN and graphene is not sharp and contains a transition region where alloying of the two materials takes place. This is substantiated by monitoring the concentration of the adsorbates on the iridium surface during heterostructure synthesis. Selected-area electron diffraction (μ-LEED) was used to examine the crystal structure of the heterostructures, including the alloy region which is essentially a 2D hexagonal mixture of B, C and N atoms. Spatial extension of the alloy region can be tuned by modifying the synthesis temperature and by applying oxygen treatment during the synthesis, which provides routes for fabrication of a sharp hBN-graphene boundary if desired. Annealing of hBN-graphene heterostructures at temperatures above 1000 °C results in disintegration of hBN, leaving hollow graphene microstructures on the surface of iridium that can be further structurally modified by re-growth or oxygen etching treatment. This effectively provides foundations for a new method for in situ production of graphene microstructures and fabrication of new graphene-based systems.

MODIFICATION OF Ti/Zr MULTILAYER BY FEMTOSECOND LASER BEAM

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Femtosecond laser texturing holds promise for the surface modification of biomaterials, due to a wide application to all materials; the possibility of getting a wide variety of structures with micro- and nano-scaled features; and a fast, repeatable and contactless process. Laser processing is unique method, which allows production of bioactive surface with formation of the desired oxide, creation of nano/micro textures and change wettability of the surface.

Due to excellent mechanical properties and moderate biocompatibility, Ti/Zr multilayer thin films, as novel nanolayered composites were deposited by ion sputtering on Si substrate. Subsequently, the Ti/Zr thin films were irradiated by femtosecond laser pulses in air to induce the following modifications: (i) mixing of components within the thin film structures, (ii) formation of ultrathin oxide layer at the irradiated surfaces, and (iii) structuring of the surface arrays in form of ripple structure. The main focus of this experimental study was examined a novel Ti/Zr bimetallic nanolayered composite in order to create a biomimetic surface with changed wettability for cell adherence. For this purpose, fibroblast cells were seeded over the laser-modified surfaces of Ti/Zr multilayer thin films. Using SEM and laser scanning confocal microscopy images of the modified areas with cell culture revealed cell adhesion and growth depending on surface composition and morphological forms. These results indicated a good adhesion and proliferation of cells, with some tendency of the cell orientation along of ripples.

MOLECULAR DYNAMICS SIMULATIONS AND VISUALIZATION OF PLASTIC DEFORMATION IN METALLIC MATERIALS

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In molecular dynamics (MD) simulations metallic materials are described using the embedded atom model, where atomic nuclei as represented by hard spheres that obey Newton's equations of motion, embedded in a potential dependant on the distance between neighbouring nuclei. In this way, MD simulations enable us to track the movements of individual atoms in a sample in response to deformation. Time and length scales accessible by MD simulations allow for an atomic resolution view of the nucleation of dislocations and their flow and interactions during plastic deformation. Visualizations based on the simulations can be used to help students and researchers understand, conceptualize and discuss mechanisms of plastic deformation in metallic materials.

PLASMA MODIFICATION OF POLYPROPYLENE TUBES

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Modification of Polypropylene (PP) tubes by cold Atmospheric Pressure Plasma Jet (APPJ) is suggested in this work. 1,5mL microcentrifuge tubes from three different manufacturers were closely examined and their characteristics before and after plasma treatment compared. The influence of gaseous plasma treatment on surface morphology was studied by Atomic Force Microscopy (AFM), changes in surface wettability by measuring the Water Contact Angle (WCA), while surface chemical changes were analysed by X-ray Photoelectron Spectroscopy (XPS) survey spectra and carbon high resolution spectra. Differential Scanning Calorimetry (DSC) was used to analyse the amount of crystalline and amorphous phase in every sample. After close examination of polymer characteristics, tubes were used in biological experiments where the improvement of plasma treatment was determined. Flow cytometry of extracellular vesicles isolated in plasma treated tubes was performed and protein adsorption was measured. Results of our study show that gaseous plasma treatment is an intriguing technique to uniformly alter surface properties of PP tubes and improve EVs yields up to 24 %.

HALL EFFECT OF THE APPROXIMANTS TO DECAGONAL QUASICRYSTALS

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The Hall effect has been studied in single crystals of monoclinic Y-Al-Ni-Co[1,2], orthorhombic o-$Al_{13}Co_4$[3], monoclinic m-$Al_{13}Fe_4$ and m-$Al_{13}(Fe, Ni)_4$[4] and Taylor phase T-$Al_{73}(Mn,Fe)_{27}$[5]. The Hall coefficient R_H of each alloy has been measured for all combinations of the electrical current and magnetic field directions and in the temperature interval from 90 to 370 K.

In the first four samples, which belong to the $Al_{13}TM_4$ (TM = transition metal) class of approximants to the decagonal quasicrystals (d-QCs), the Hall coefficient exhibits well defined anisotropy: R_H is positive hole-like or zero for the magnetic field parallel to the plane that corresponds to the quasiperiodic plane in d-QCs, and is negative electron-like or zero for the magnetic field perpendicular to this plane. The only exception is R_H in $Al_{13}Fe_4$ for the field parallel to the stacking direction, which changes the sign from positive to negative value with the increase of temperature. The results for the anisotropy of R_H are correlated to the anisotropy of R_H in d-Al-Ni-Co and d-Al-Cu-Co [6] quasicrystals and a brief overview of the theoretical results is presented [7].

In T-$Al_{73}(Mn,Fe)_{27}$ the Hall effect contains both normal and anomalous (magnetic) Hall effect. Therefore, the results for the magnetic susceptibility were used for the separation of these two contributions. It was found that neither the normal nor the anomalous Hall coefficient depend significantly on the direction of the magnetic field and current with the respect to the crystalline axes.

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THE EFFECT OF PRE-TREATMENTS ON PROPERTIES OF ALUMINIUM ALLOYS AND THEIR CORROSION PROTECTION BASED ON HYBRID SOL-GEL COATINGS

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Aluminium alloys AA2024 and AA7075 are most commonly used in transportation industry. Due to the presence of intermetallic particles they are susceptible to localized corrosion. before performing any kind of protection, it is necessary to properly pre-treat the surface which will be treated. In the present work the effects of mechanical and chemical pre-treatments of AA7075 and AA2024 aluminum alloys were studied in terms of changes of morphology, composition and wettability of the surface, and of the related corrosion properties in sodium chloride solution. The mechanical treatments tested include grinding under water and diamond polishing under non aqueous liquid. The chemical treatments included etching with sodium hydroxide and with a commercial cleaner and desmutting in nitric acid and leads to a smaller current density in the polarization curves. The study of the effect of pre-treatments was carried out by Scanning Electron Microscopy equipped with Energy Dispersive X-ray Spectroscopy, potentiodynamic polarization measurements and X-ray photoelectron spectroscopy.

After pre-treatment treatment of the surface is carried out to protect it against corrosion. In the present work hybrid sol-gel coatings, based on silane precursors 3-glycidyloxypropyl(trimethoxysilane) (GPTMS) and tetraethoxysilane (TEOS) were used as corrosion protection of AA7075. To enrich the barrier properties of coating, SiO_2 nanoparticles were added to the final solution. Inhibition and self-healing effect were achieved by the incorporation of corrosion inhibitor cerium nitrate ($Ce(NO_3)_3$) into the coating. Due to oxygen reduction on cathodic sites OH^- ions causing the local increase in pH thus enabling the precipitation of cerium oxide/hydroxide. Ce^{3+} ions can further oxidize into Ce^{4+} ions and insoluble Ce(IV) oxide/hydroxide may be formed to protect cathodic sites. A bi-layer system of two sols was applied on the AA7075, where the first layer was doped with $Ce(NO_3)_3$ and the second was undoped. Self-healing effect of coatings was confirmed using different techniques such as immersion test, electrochemical impedance spectroscopy (EIS), X-ray photoelectron spectroscopy (XPS)and scanning electron microscopy (SEM) with chemical analysis (EDS).

According to the EIS and immersion test, self-healing effect was effective after 4 days of immersion in 0.1 M NaCl. The presence of Ce^{3+} and Ce^{4+} ions was confirmed with XPS analysis (Fig.1).

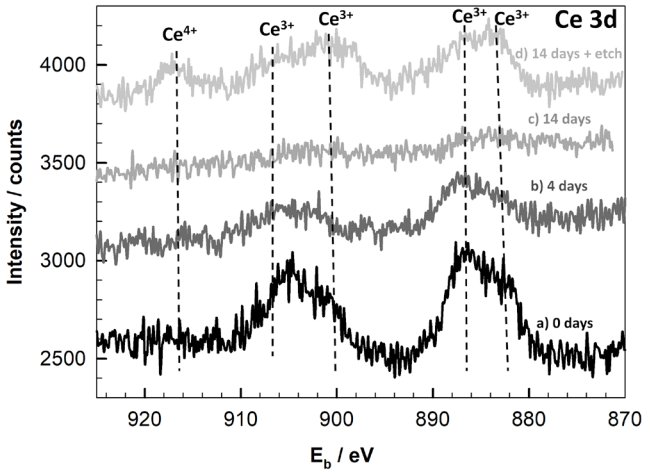

Figure 1: XPS Ce 3d spectra recorded within a cross-shaped cribe on GTS-Ce+GTS coating on AA7075 after different immersion time.

MEASURING THE EFFECT OF SURFACE CONDITIONS ON GAS FLOW CONDUCTANCE

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In the molecular gas flow regime, interactions between gas molecules and a tube's inner surface affect gas flow conductance of the tube. Under certain conditions, conductance of a tube can be measured with the pressure decay method. Here, a vacuum chamber of volume V is pumped through the tube, which allows for conductance C to be determined from the slope of the logarithm of the pressure decay curve $C=V\times\mathrm{d}(\ln(p))/\mathrm{d}t$, if upstream pressure p is measured with sufficient precision. In our case, estimated uncertainty of the measured conductance is 0.3%, and reproducibility is below 0.2%. Conductance was measured for an electropolished stainless steel tube with inner diameter 7.76 mm and length 776 mm, using various pure gases. Conductance was then measured after several surface treatment steps, including heating with exposure to either O_2 and H_2, and vacuum baking the tube surface. The tube was then etched in aqua regia in order to produce macroscopic roughness of the inner surface, and the previous process was repeated. For He gas the maximum difference of tube conductance of different treatments was as high as 19 The observed difference in values of conductance in molecular regime was attributed to changes of effective tangential momentum accommodation coefficient. Results were compared with published research on tangential momentum accommodation coefficient of spinning rotor vacuum gauge.

DETERMINATION OF COLLOIDAL TiO_2 NANOPARTICLES CONCENTRATION PRODUCED BY LASER ABLATION OF Ti IN WATER

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Pulsed laser ablation in liquid is widely-used technique for production of nanoparticles which has some advantages compared to chemical techniques, as low-cost, low time-consumption, ecological acceptability, high purity of nanoparticles, low ratio of unwanted reaction products and possibility of adjusting large number of parameters to obtain nanoparticles with wanted features. In this paper we present innovative proof-of-concept method for determination of TiO_2 NP concentration in colloidal suspension produced by laser ablation of Ti target in water. The proposed method combines crater volume microscopic measurements and size-distribution of NP, which is obtained from SEM [1-4]. Laser ablation is performed with 1064 nm Nd:YAG laser at pulse rate 5Hz and different number of pulses (1000p, 3000p, 5000p). It was found that TiO_2 NP distribution has maximum value at about 100 nm in diameter. TiO_2 nanoparticles are dominantly amorphous as is confirmed by XRD measurements and also band-gap analysis by Tauc plot obtained from UV-VIS spectroscopy. For comparison, concentration is also calculated from UV-VIS data and Mie scattering theory, involving same size-distribution data from SEM. The validity of this proposed method is verified by comparing theoretical and experimentally obtained parameters.

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INTERACTION OF PLATELETS WITH PLASMA TREATED TiO_2 NANOTUBES

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Vascular stents implantation could induce injuries of arterial walls that initialize platelet activation and blood clot/thrombus formation. In order to avoid inflammatory response, common bare metal stents were often coated with drug-infused polymer films. Although favourable effects of the released anti-inflammatory or anti proliferative drugs have been observed, the polymer coating has been associated with several adverse clinical effects, such as late stent thrombosis and restenosis (1). Various technologies have been employed in order to design polymer-free drug eluting stents, such as microporous stents and drug-loaded inorganic coatings (1), such would prevent thrombus formation and promote the proliferation of human coronary artery endothelial cells (HCAEC). For that purpose, self-arranged layers of TiO_2 nanotubes (NTs) - freshly prepared NTs (1 week old), old NTs (2 months aged) and NTs after treatment with oxygen plasma - have been synthesized in present research and their interactions with whole blood and HCAEC have been studied. TiO_2 NTs were synthesized by electrochemical anodization of Ti foil as shown previously (2). In order to clean the materials of electrolyte residuals and activate the surface, samples were treated with oxygen plasma as shown in Ref. (3). Scanning electron microscopy (SEM) and X-ray photoemission spectroscopy (XPS) were used to determine morphology and surface chemistry of the TiO_2 NTs. Water contact angle (WCA) measurements were performed in order to evaluate their wettability. Samples were incubated for 1 h with whole blood obtained from healthy volunteers via vein puncture. Oxygen plasma treated surfaces showed significantly reduced adhesion and activation of platelets in comparison with fresh, aged TiO_2 NTs and Ti foil. Plasma treatment is also beneficial for HCAEC proliferation. This results indicate that plasma treated TiO_2 NTs are promising polymer free inorganic coatings for successful stent implantation, which could reduce thrombogenicity.

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INFLUENCE OF THE ELECTRON SCANNING MICROSCOPY IN A LOW VACUUM ON THE MORPHOLOGY OF THE MATERIALS

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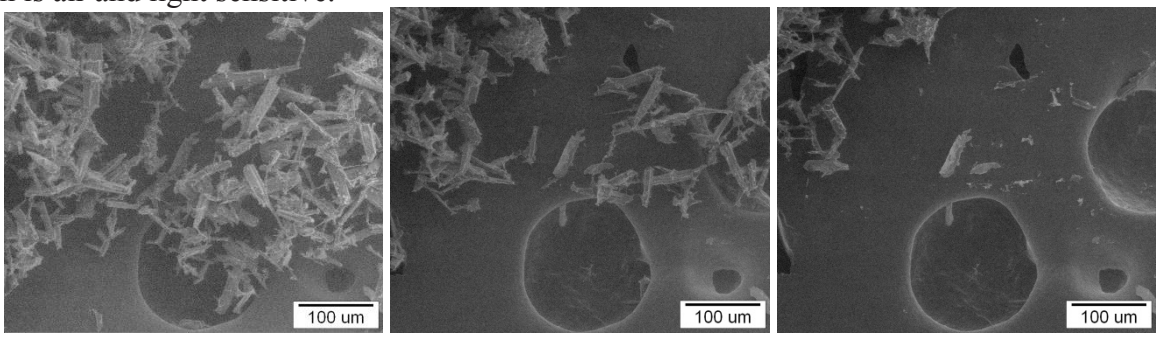
The environmental scanning electron microscopy (ESEM) has enabled a new way of exploring non-conductive materials, even if they are wet or sensitive for high vacuum. It also allows observing in-situ processes within the microscope chamber. This method does not require any additional preparation of the samples with prior surface modification using fixation or coating with a conductive layer.
The most significant challenge by using of ESEM, however, is to retain the original and unchanged shape of the analysed material during vacuuming and imaging. The second challenge is how to control the in-situ process. Many parameters affect the reliable microscopy and they can be divided into instrumental parameters and parameters related to the properties of the investigated materials.
The most critical instrumental parameters are the pressure in the chamber and the stage temperature. The pressure variations are affected by the amount of added water vapour in the chamber. Frequently, pressures between 50 Pa to 150 Pa are applied for dry samples at room temperature. The operator's experience and skill are essential by tuning the parameters according to shape variations of the samples during imaging.
Fig. 1 shows the shape change and sublimation during observation of the vanillin crystalline powder, which is air and light sensitive.



a) b) c)

Figure 1: The crystalline powder of vanillin, 1a) initial state, 1b) image after 37 seconds, 1c) image after 56 seconds. Microscopy conditions: pressure 80 Pa, acceleration voltage 5 kV and electron current 90 pA.

The changes in the shape of the material during the imaging have a significant influence on properties such as chemical composition, density, thermal and electrical conductivity, temperature stability, porosity and humidity of the analysed material. In Figure 2 is an example of polymer balls. The full balls did not change their shape, while the porous balls changed their shapes significantly.

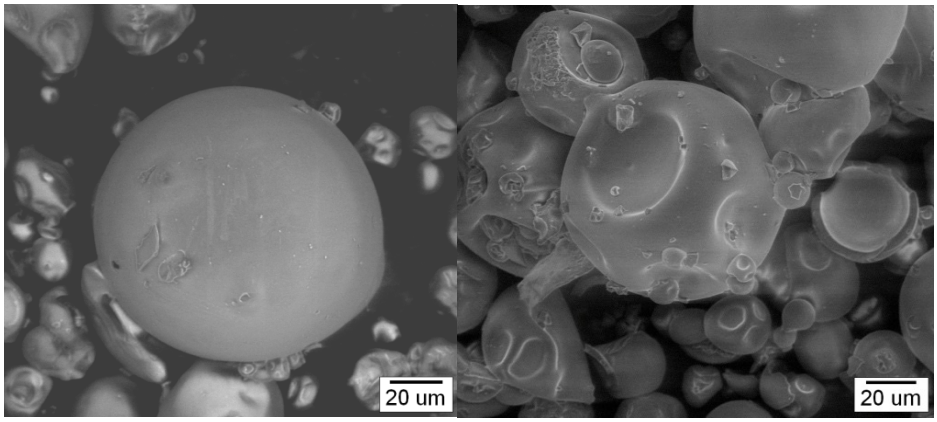


Figure 2: The polymer particles, 2a) a polymer ball with unchanged shape, 2b) porous polymer balls changed their shape. Microscopy conditions: pressure 80 Pa, acceleration voltage 5 kV and electron current 340 pA.

During low-vacuum microscopy, some dynamic processes can take place, such as drying, electron-beam heating, melting, evaporation and sublimation of the substances, which result in changes of the original shapes of samples. We have found that the environmental scanning electron microscopy is unsuitable for all types of samples. In many cases, however, we can provide a reliable analysis using appropriate instrumental parameters, pre-cooling of the samples in liquid nitrogen, or use a cooling unit (Peltier stage).

GROWTH OF THIN LAYERS OF Ge NANO-WIRES IN ALUMINA ON POROUS SUBSTRATES

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Thin self-supporting materials containing semiconductor quantum dots or nano-wires are very interesting for various detector and membrane applications [1]. Herein, we present fabrication method of thin layers of alumina containing three-dimensionally ordered Ge nano-wires [2]. We show that the film is possible to grow on the substrate which contains pores/holes with the diameter of 20-40 nm. Thus, the self-supported film is produced above the holes. The minimal film thickness needed to achieve the full coverage of the substrate holes is found to be nearly equal to the lateral size of the holes.

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GARLIC CLOVE TREATMENT WITH LABORATORY-SCALE, LOW-PRESSURE OXYGEN PLASMA

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To improve crop germination, growth, and yield, chemical or biotechnological procedures are often used, but are either environmentally harmful, or involve complex and lengthy processes. As crop production is increasingly limited by climate change, further technical improvements are desired. One environmentally benign method of physically improving these properties is low-pressure oxygen plasma. In the field of plasma agriculture, so far, germination, growth and other properties of seeds and seedlings have been positively influenced by plasma treatment.
On a laboratory scale, we have used inductively coupled, low pressure radio frequency (RF) oxygen plasma to treat garlic cloves and observe its effect on their surface properties and germination. As expected, the wettability of the clove skin surface has been increased by plasma treatment, as determined by water contact angle measurements. X-ray photoelectron spectroscopy and scanning electron microscopy have linked this effect to garlic clove surface chemical composition and surface morphology changes. The described modifications have a cumulative effect on the clove germination (sprout emergence), dependent on the plasma treatment parameters chosen. With optimal selection of treatment conditions, sprout emergence can be improved, with possible implications for yield improvement.

SURFACE CHEMISTRY OF THERMALLY REDUCED GRAPHENE OXIDE

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The surface chemistry of carbon materials plays the key role in their application. However, identification of oxygen functional groups on carbon surfaces is challenging and requires careful analysis. In the present study we have used complementary surface techniques such as Fourier-transform infrared spectroscopy (FTIR), temperature-programmed desorption (TPD) and X-ray photoelectron spectroscopy (XPS) for the analysis of surface changes occurred during gradual thermal reduction of graphene oxide (GO). The results of FTIR, XPS and TPD methods have shown that the surface functional groups on as-prepared graphene oxide are: epoxy and alkoxy, carbonyl groups prone to transformation to a substituted ketones and aldehydes, carboxyl, carboxylic anhydride (less and more stable form), lactone, phenol and carbonyl/quinone groups. It was established that thermal reduction of GO is three-stage process, where each stage is comprised of specific surface chemistry that is governed by different thermal stability of oxygen-containing groups.

MODELLING OF SIMULTANEOUSLY OBTAINED SMALL AND WIDE ANGLE SYNCHROTRON-RADIATION SCATTERING DEPTH PROFILES OF ORDERED TITANIA NANOTUBE THIN FILMS

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Thin-films of vertically aligned titanium dioxide nanotubes are key constituents of charge transfer layers in 3rd generation photovoltaics. The beneficial charge transport primarily occurs due to the favourable microstructural features, i.e. the large effective surface-to-volume ratio. Here measuring methods are selected which are sensitive to microstructural features: Simultaneous Grazing Incidence Small and Wide Angle X-Ray Scattering (GISWAXS) and Scanning Electron Microscopy (SEM). Further, a model is developed for the simulation and explanation of the GISWAXS experimental data. Titanium nanotube arrays were investigated, which were previously successfully produced by electrochemical anodization of titanium thin film evaporated onto a zinc-oxide coated glass substrate. The developed model was shown to be appropriate to describe the obtained samples, which consist of arrays of single nanotubes with a diameter of 40-80 nm, and a titanium dioxide layer with a porosity of 30 – 50%. Voltage applied during the anodization can be used to tailor the average nanotube diameter. Within this work, we presented simultaneous GISWAXS as a favourable tool for the fast and successful study of the average nanotubes' diameters in nanotubes arrays.

ATMOSPHERIC PRESSURE PLASMA JET ASSISTED IMPREGNATION OF GOLD NANOPARTICLES INTO PVC POLYMER

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Atmospheric pressure plasma jet due to its high activity [1,2] is used as a tool to design polymer/ nanoparticles composite materials for various applications. The aim of this research is to get a cheap and green method for nanoparticles impregnation into polymer surfaces. The proposed route consists of nanoparticle synthesis by laser ablation in water and nanoparticles impregnation into polymers assisted by atmospheric pressure plasma jet. The impregnation is achieved by increased roughness of treated samples containing nanoparticles which are embedded into such rough structures. The concentration of applied nanoparticles is of order of $4 \cdot 10^8$ ml^{-1} and it is calculated from evaluated craters [4,5] left after ablation by a method described in [6]. This proof-of-concept method is based on pre- or post-treatment of PVC polymer drop coated with Au nanoparticles by helium atmospheric pressure plasma jet.

Keywords: atmospheric pressure plasma jet; polymer nanocomposites; nanoparticles impregnation; polymer treatments; poly(vinyl chloride), PVC

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QUANTITATIVE DEPTH PROFILING OF ULTRA-THIN FILMS AND MULTILAYERS

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Nanosized thin films and multilayered structures found very important technological applications in many fields, such as metallurgical hard coatings, microelectronics, corrosion protection, optical layers, decoration coatings, adhesion interlayers, tribological coating etc. New requirements in thin film applications pushed the thickness scale of such layers to the nanometer and even to the sub-nanometer range. Due to intrinsic nature of thin film structures being thermodynamically unstable and also due to requirements for thermal treatment of such layered structures, the inter-diffusion processes often take place. Knowledge of the inter-diffusion phenomena helps to predict and to control thermal stability, chemical reactions, phase transitions and mass transport occurred in materials. To follow the diffusion processes in such nanolayered structures, the depth distribution of elements in very shallow region (up to 20 nm) must be known with very high depth resolution (1–3 nm). High-resolution shallow depth profiling based on ion bombardment of solid surface and thin films is an appropriate analytical technique to provide such information on elemental depth distribution. It combines a controlled removal of material by ion sputtering and subsequent analysis of newly formed surface by one of surface analytical techniques, such as secondary ion mass spectroscopy – SIMS, X-ray photoelectron spectroscopy – XPS, Auger electron spectroscopy – AES and glow discharge Optical Emission Spectroscopy – GDOES. In the past years, the investigators of this joint project contributed significantly to the methodology of depth profiling and to the technical developments for improving the depth resolution of this analytical approach.

In this work we compared different depth profiling techniques like GDOES, ToF-SIMS and AES depth profiling. The multilayers of Ag/Ni, Ni/Cr, Ni/C and W/Nb deposited on Si were analysed at different sputtering conditions (Ar^+, O_2^+, Cs^+ sputtering). We found that depth profiling with methods analysing the sputtered particles (GDOES and SIMS) are very fast due to high density of such particles available and they are also very sensitive for small changes in composition of thin films. Using SIMS method matrix effects should be taken into account to obtain chemical composition of analysed thin films. The depth profiling with an electron spectroscopy method like AES is rather slow due to smaller amount of emitted particles – electrons but the direct chemical composition of multilayers can be obtained from measurements.

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STERILIZATION OF WOODEN ARTIFACTS BY APPJ

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In this work a cold atmospheric pressure plasma jet (APPJ) for sterilization of wooden artifacts is used. APPJ is rich of cold reactive species responsible for treatment of surfaces without thermal damage [1,2].
This research includes the application of a plasma jet for surface treatment of various wooden artifacts in terms of sterilization of microorganisms at the surfaces. This method will complement standard techniques of sterilization/disinfection based on using hazardous chemicals, vacuum or gamma rays or using of chemical solvents.
This method is tested on wooden artifacts covered with golden layer which was infected with *Coniophora puteana* and using Ar, He and Ar/O2 gasses for APPJ. The plasma processing is monitored with IR camera and optical emission spectroscopy as a versatile tool for plasma processes monitoring [3-5].
It was found that Ar APPJ appeared as most promising candidate for sterilization of wooden articats as fungi did not survived the treatment making it comparable to standard techniques. The advantage of this method is the fact that surface of the artifact did not survived any damage what was not the case for standard treatment.
Acknowledgements: This work is mainly a part of *PlasmaArt* project supported by Adris Foundation and Croatian Science Foundation under project IP-11-2013-2753.
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STUDY OF SURFACE TREATMENT OF HUMAN HARD DENTAL TISSUES WITH ATMOSPHERIC PRESSURE PLASMA JET

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Surface of human hard dental tissues (enamel and dentine) was treated using a helium single-electrode atmospheric pressure plasma jet (APPJ) with the aim of enhancing surface properties of treated material. Treatment by plasma was monitored by means of optical emission spectroscopy (OES). Modification of the enamel and dentine surface was observed using contact angle measurements and X-ray photoelectron spectroscopy. Even a short treatment time (about 1 s) lead to a significant reduction in the water contact angle: 45% for the dentine and 32% for the enamel.

OES spectra show typical content of helium atmospheric plasma jets. Apart from various transitions between highly excited He atoms we also observed transitions from N_2 and O_2 molecules as well as OH, H, O and NO radicals. These results were explained by diffusion of the surrounding gas into the He jet. The OH and O radicals are reactive enough to cause oxidation of many compounds what is necessary for the bleaching effect.

The surface of the untreated teeth samples consisted mostly of the elements C, O, N, Ca and P. Their relative concentrations changed during 9-min He APPJ treatment. Significant decrease of C and N relative concentrations suggested the removal of adsorbed compounds containing carbon and nitrogen and disclosure of underlying inorganic hydroxyapatite structure. The Ca/P ratio has increased from 1.26 to 1.72, which is very close to an ideal ratio for the highest volume of remineralisation of the human dental enamel. Oxidation of the transparent organic matrix in the enamel, one of the major processes in tooth bleaching, was confirmed with the increase of O concentration, from about 31 to 46 at.%.

Helium APPJ treatment therefore proved to be a potential tool for chemical surface modification of hard human dental tissues.

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CHARACTERIZATION OF NON-EQUILIBRIUM GASEOUS PLASMA BY OPTICAL EMISSION SPECTROSCOPY

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A review of optical emission spectroscopy used for characterization of non-equilibrium plasma created by various gaseous discharges is presented. The collaboration between researchers was established over a decade ago and enabled fruitful scientific and technological results. We took advantage of the complementary character of collaborating groups, the Croatian with expertise in optics and the Slovenian in plasma technologies. Optical emission spectroscopy was applied for characterization of gaseous plasma suitable for tailoring surface properties of polymeric foils [1], cleaning porous ceramics [2], degradation of bacteria [3,5], modification of ink-jet paper [4], functionalization of different polymers [6-9, 12], modification of aluminum foils [10,11], modification of PVC materials [13], studying reactive species in plasma jets [14, 15, 17, 18], modification of various materials using high power-density plasma [16, 19] and modification of human teeth [20-22]. Innovative approaches to characterization of gaseous species in fusion reactors have been suggested and a common project has been proposed.

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ATMOSPHERIC PRESSURE PLASMA TREATMENT OF WOOL YARNS

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The application of low temperature plasmas in the field of textile industry started in the second half of the 20th century. Since then, plasma technology has emerged as an environmentally friendly alternative to conventional wet processing of textiles that includes high consumption of water, chemicals and energy. Low-pressure plasma is well-known and developed technology with outstanding features such as high concentration of reactive species, superior chemical selectivity and good stability and uniformity in surface modification of textiles. Atmospheric pressure plasma (APP) has emerged as a cost-competitive technology with a possibility of integration in a conventional production line for continuous processing of textile materials. APP treatment is usually applied as a pretreatment process for cleaning, etching and activation of textile surface. It is the first step involved in textile wet processing that removes natural impurities and those added during the process of manufacture thus making the fibre accessible to water, dyes and finishing chemicals. APPs suitable for textile treatments are (i) corona discharge, (ii) dielectric barrier discharge (DBD), (iii) atmospheric pressure glow discharge (APGD) and (iv) atmospheric pressure plasma jet (APPJ) [1]. In this research, for the treatment of siro wool yarns, an atmospheric pressure argon plasma was used, described in previous work [2] and developed at The Institute of Physics Zagreb. The yarn was continuously passing through 130 mm long glass tube of outer diameter 4 mm and inner diameter 1 mm in which argon plasma was generated. Applied plasma parameters were: gas flow rate 2.2 l/min, frequency about 25 kHz, current amplitude around 5 mA, amplitude voltage of 5 kV, power of 2.5 W and yarn speed movement 1.28 cm/s [3]. Analysis of morphological changes using scanning electron microscope (SEM) revealed cleaning of the surface and indications of etching of wool scales. After APP treatment breaking force increased by 26.6 % and breaking elongation by 34.7 % compared to untreated samples. This could be due to etching of the surfaces which caused higher friction between fibres and therefore higher force is required to overcome frictional force and for breakage to occur. Modification of breaking elongation can be attributed to softening of the wool scales making the fibre more elastic. Hairiness and unevenness of the yarn were examined in order to assess the impact of APP treatment on physical properties of wool yarn. A slight decrease of yarn unevenness was observed, whereas the hairiness increased. Subsequent winding of the yarn directly after plasma treatment could have additionally influenced hairiness. It can be concluded that atmospheric pressure argon plasma treatment can have significant impact on mechanical and physical properties of wool yarn. Further researcher needs to be conducted in order to examine influence of plasma on yarn hairiness.

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COUPLING OF LASER PRODUCED PLASMA AND ATMOSPHERIC PRESSURE PLASMA JET

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In the laser-induced breakdown in gases, electrons accelerate in the laser field and ionise the gas by colliding with the gas species. What mostly determines if the breakdown will occur is the ionisation energy of the gas target, and the irradiance of the laser beam. However, if there is some concentration of excited atoms in the gas, the breakdown will occur more easily, and the generated electrons liberated from the atoms will have higher kinetic energies. In this work, we have used atmospheric pressure plasma jet as a target for laser-induced breakdown. Pulsed DC source was used for the plasma generation, with variable pulse width and was synchronised in time with the Nd:YAG laser pulse. This type of electrical discharge is a pulsed phenomenon and accordingly, concentration of different species in the plasma change in time. Depending on the delay between the electrical discharge initiation and the laser pulse arrival, we have measured the temporal evolution of the light emission from the laser-produced plasma, using a monochromator and a fast photomultiplier tube.

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DOSE-DEPENDENT CYTOTOXICITY OF NANODIAMONDS ON PLASMA-TREATED *Saccharomyces cerevisiae* CELLS

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The tremendous increase in the use of functionalised nanomaterials for industrial processes and consumer products, inevitably promotes their interaction with living organisms and environment. Previous studies have investigated cell−nanoparticle interactions under otherwise favourable conditions, yet in real life, organisms are subject to environmental stresses, which may affect their response to nanoparticles. This work investigates the effect of atmospheric-pressure plasma, a model stress inducing environment rich in highly-reactive ROS and RNS species, UV light, and mild heat, on the interactions between inert nanodiamond particles (NDs) and a eukaryotic model organism, *Saccharomyces cerevisiae*. Plasma treatment affected the cell membrane permeability and enhanced the size of the pores, resulting in the increased nanoparticle uptake. Accumulation of nanoparticles in larger deposits inside the cells and around the cell wall affected cell structure, cell wall morphology and membrane permeability. Plasma-treated cells exposed to 100 μg/ml NDs for 24 h showed significant inhibition of metabolic activity and 55% reduction in cell viability, whereas at lower concentrations (0, 5 and 50 μg/ml) of NDs, no significant effect on cell viability or cell growth was observed. These results suggest that presence of intra- or extra-cellular stresses is an important determinant of cell fate upon exposure to nanoparticles.

ANALYSIS OF INCLUSIONS IN STEELS

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Control over the type, size and morphological properties of inclusions in steels is a very important aspect of modern steel making. For this, accurate and detailed analysis of inclusions are needed. The standard approach employs an optical microscope and analysis of images according to standards. However, this approach lacks detailed information about composition, and inclusion sizes and morphological properties are not well characterized. The second approach to inclusion analysis can be an utilising the scanning electron microscope. With an added EDS detector, inclusion composition can also be determined. However, this process is longer so it is usually performed in an automated way. To get more details about individual inclusions, additional methods such as electron backscattered diffraction, combined with EDS analyisis, can be used.
The contribution will show an example of inclusion analysis in steels in all crucial steps to obtain reliable results.

PENETRATION OF OXYGEN PLASMA RADICALS THROUGH THE TEXTILE STRUCTURE

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A special plasma system was constructed for the late afterglow treatment of textiles. A piece of textile (viscose with a density 50 g/m^2) was mounted in a special sample holder and placed perpendicularly to the drift of plasma radicals. RF oxygen plasma at various powers (10 W – 30 W) and pressures (100 Pa – 200 Pa) was used as a source of oxygen plasma radicals. The pressure gradient established along the tube enabled penetration of plasma radicals throughout the layer of the textile. A nickel catalytic probe was used to measure the loss of oxygen atoms when passing through the textile layer. The O-atom density was measured before and after passing the textile layer. The O-atom density dropped for about 10 % after passing the textile.

AMMONIA PRODUCTION IN ASDEX-U DIVERTOR AND IN A LABORATORY DUAL ATOM BEAM EXPERIMENT

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In magnetically confined fusion reactors divertor heat loads needs to be reduced with promoting radiation in the plasma edge. This is possible with a certain concentration of low-Z impurities. Contrary to fusion devices with carbon-based plasma facing components (PFCs) in fusion devices with all-metal PFCs, such as the upcoming ITER, low-Z impurities from plasma-surface interactions are not enough, therefore seeded gases needs to be introduced into the plasma. So far nitrogen seems to be the best choice and in current plans, nitrogen will be seeded in ITER plasma shots [1].
However, nitrogen seeding leads to ammonia production, which could be a potential issue for ITER [2]. In the D-T phase of ITER nitrogen seeding would lead to tritiated ammonia production, which would be retained in the vessel and thus contribute to the in-vessel tritium inventory. Moreover, the tritated ammonia would reduce ITERs duty cycle, due to ITS adsorption in the active surface of the ITER cryo pumps.
Therefore, in order to estimate the in-vessel ammonia production in ITER, many ammonia production experiments are performed in current all-metal PFC fusion tokamaks, such as JET with ITER-like wall and the full tungsten ASDEX-Upgrade (AUG). Complementary to those large scale experiments also a smaller laboratory experiments are performed in plasma labs such as ours in the Department of surface engineering at Jožef Stefan Institute.
In order to study ammonia production, we designed a dual beam experiment with two atom sources (microwave MW plasma). The experimental setup consists of two MW surfatron plasma sources, quartz plasma chambers, glass reaction chamber, MKS baratron, mass flow controllers (MFCs), vacuum pump, catalytic probe, optical emission spectrometer and residual gas analyzer (RGA). The MW plasma sources and plasma quartz chambers serve as two independent atom sources. In one, only hydrogen atoms are produced and in the other only nitrogen atoms. Therefore, the number densities of the nitrogen and hydrogen atoms can be easily controlled with discharge parameters such as flow and power. The atoms beams are crossed in the glass reaction chamber which limits the N-H reactions to what can be a very well defined area. Furthermore, in this set up there is no plasma-phase destruction of surface-produced ammonia. We studied a production of ammonia on different materials in a controlled environment and an impact of Ar and He on ammonia production.
We also participate in an on-going nitrogen seeded experiments in AUG tokamak under MST1 EUROfusion work package as spectroscopy analysis experts for the ammonia discharges and RGA experts. We mounted a new RGA in the mid plane of the AUG and connected it to the computer in the ASDEX control room. With the analysis of the RGA and OES measured data of the nitrogen seeded shots, we concluded that a significant amount of ammonia is produced in the plasma shaded areas through surface reactions of neutral species.
Our studies are supported with tasks under EUROfusion work packages: Medium Sized Tokamaks (WP MST1), Plasma Facing Components (WP PFC) and Liquid Metal Divertor Design (WP DTT1-LMD)

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