22. MEDNARODNO ZNANSTVENO SREČANJE VAKUUMSKA ZNANOST IN TEHNIKA
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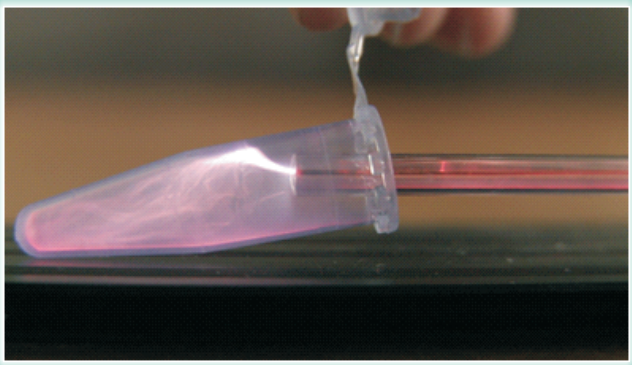
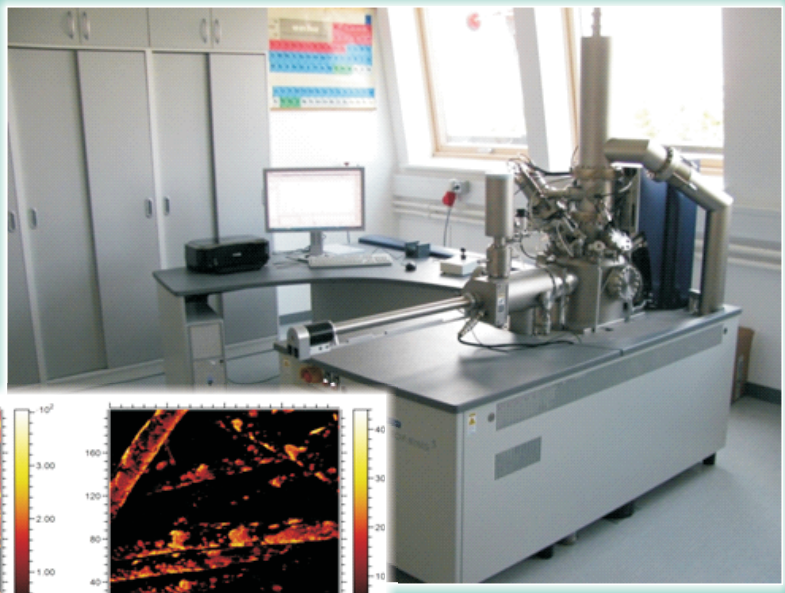
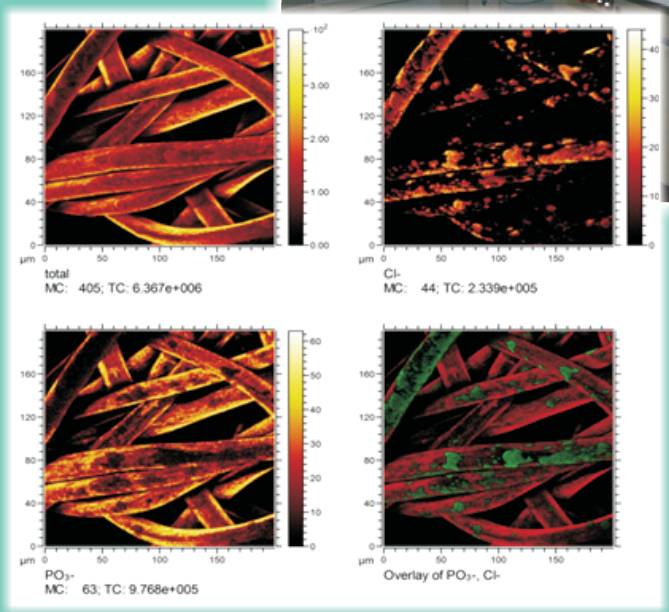
PROGRAM IN KNJIGA POVZETKOV
PROGRAMME AND BOOK OF ABSTRACTS

Osilnica,
21.-22. Maj 2015 / 21-22 may 2015







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Janez Kovač, Gregor Jakša

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HIGH RESOLUTION X-RAY EMISSION SPECTROSCOPY WITH ION BEAMS

S. Fazinić, I. Božičević Mihalić, D. Cosic, T. Tadić, M. Jakšić
Ruđer Bošković Institute, Bijenička cesta 54, 10000 Zagreb, Croatia

Energy dispersive X-ray Emission Spectroscopy (ED-XES) is widely used for elemental composition determination in unknown samples. It can be used for simultaneous detection of almost all elements of the periodic table. It requires minimal or no sample preparation and creates little or no damage to samples under analysis and as such is often declared as nondestructive and in some situations noninvasive method. Wide range of designs exist for related instrumentation, from relatively simple and small hend-held instruments for in-situ applications, stand-alone laboratory scale spectrometers, to devices integrated with more complex instruments, like electron microscopes, ion beam or synchrotron accelerator facilities.

Very often photo-ionization is employed as extitation mechanism (XRF – X-ray fluorescence) with the use of radioisotopes, x-ray tubes or synchrotrons as sources of primary radiation. It is also common to use electron (EPMA – Electron probe Micro-Analysis) or proton beams (PIXE – Particle Induced X-Ray Emission) to ionize inner atomic shells. Si(Li) or silicon drift detectors (SDD) are usually used in combination with analog or digital data acquisition systems to collect X-ray spectra which should be processed to extract information about the elements present in examined material.

Although ED-XES spectra are in principle chemically invariant, small influence of chemical effects could be observed even with Si(Li) or SDD detectors. A decade ago it was pointed out that for optimal fitting of X-ray spectra obtained by PIXE and XRF the database of fundamental parameters should recognise chemical effects on X-ray intensities and peak shapes. Nevertheles, it is still a common practice in routine ED-XES analysis to neglect such small variations of Kβ and Kα intensities and positions as a function of solid state and chemical effects. None of the commonly used algorithms for analysis of ED-XES spectra (either PIXE or XRF) takes in account possible chemical effects on X- ray intensity ratios or energies.

However, many experimental studies have been done with the aim to exploit observed variations in X-ray spectral shapes for chemical speciation. When measured with high resolution X- ray detection systems, fine structure in Kα and Kβ X-rays is observed, and the influence of chemical effects on individual Kα and Kβ line positions and intensities has been studied with numerous reports available in the literature.

During the last decade we studied chemical dependence of relative energies and intensities of the *K* X-ray band lines for various compounds of fourth-row (*3d*) transition metals, which show enhanced sensitivity to chemiucal effects. Spectra have been obtained by proton excitation using wavelength dispersive (WD) system. Recently we have also developed small WD spectrometer specifically designed for the use with our ion microprobe with the goal to explore the possibility for performing chemical speciation on microscopic samples utilizing focused ion beams available at our ion microprobe. The system has been optimized for K X-ray energies of light elements from Al to Cl. In addition, the spectrometer could be usefull in situations where ED PIXE spectra show high peak overlaps where many M, L and K X-ray lines overlap in ED spectra. During the design stage an X-ray tracing program XTRACE was used to check the usability of a simple flat crystal as dispersive element and to optimize the spectrometer geometry. Dedicated vacuum chamber, housing the diffraction crystal, sample holder and CCD X-ray detector, was constructed and positioned behind the main ion microprobe vacuum chamber.

Here our work on high resolution XES with ion beam excitation (PIXE) will be presented, including studies on high resolution K-shell X-rays of *3d* metal compounds and about the development and applications of high resolution PIXE spectrometer specifically designed for use with our ion microprobe.

MAGNETRON SPUTTERED GROWTH DEFECTS IN A TRIBOLOGICAL CONTACT

Aljaž Drnovšek, Peter Panjan, Matjaž Panjan, Miha Čekada
Jozef Stefan Institute, Jamova 39, 1000 Ljubljana

The growth defects are present in all PVD coatings. They have the influence on their tribological properties as well as on corrosion and oxidation resistances. Although they form the first junction points in the contact area between two sliding bodies, there are not many studies that mention the growth defects and their influence on the tribological contact. Their roll is the most intensive in the beginning of the sliding, in the run-in period, but they influence can stretch to a steady state friction. How does this transition from the run-in to the steady state friction occurs, and more important how can the growth defects affect the tribological performance, is still not well understood.

To investigate the behaviour on microscale in the contact area we used tool steel coated with TiAlN hard coating prepared by unbalanced magnetron sputtering. A set of ball-on-disc tribological experiments with a low number of cycles was conducted. As a counted body either the alumina (Al_2O_3) or the softer 100Cr6 ball was used. The scope is that we managed to track individual nodular growth defects before and after the tribological test. This enabled us a more detailed "cycle to cycle" view on the role of growth defects in the tribological contact.

SMALL ANGLE X-RAY SCATTERING AT THE AUSTRIAN SAXS BEAMLINE AT ELETTRA

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The scattering of X-rays at small angles (SAXS, close to the primary beam) was found to provide structural information via inhomogeneities of the electron density with characteristic dimensions between one and a few hundred nm. SAXS was for the first time applied for the study of metallic alloys (Guinier) 20 years ago. Today SAXS is a powerful technique for structural analysis of various types of materials: metal alloys, polymers in solution and in bulk, biologic materials (tendons, muscles, macromolecules in solution), emulsions, porous materials, nanoparticles, aerosol, etc. SAXS can provide information about the shape, size and distribution of nanoparticles and larger molecules, and also the internal structure of disordered and partially ordered systems.

In most typical experiments, the SAXS technique is performed in transmission geometry. In situations where the transmission mode is not a feasible option, such as when the sample of interest is a thin film on an opaque substrate or when only the surface microstructure is of interest, Grazing Incidence SAXS (GISAXS) is used, i.e. grazing incidence reflection geometry to obtain information from surfaces and near surface parts of material. The SAXS / GISAXS technique has many advantages: it provides a non-destructive structural probe, it does not require a special sample preparation, can be applied for in situ characterization, yields an excellent sampling statistics (average over a macroscopic range of several mm), provides information on the nanometer scale about the particle geometry, size distribution, spatial correlation etc.

As SAXS measurements are done close to the primary beam (″small angels″), it requires a very brilliant source of X-rays, like synchrotron radiation. At the synchrotron Elettra near Trieste (Italy) is the Austrian SAXS- beamline which is an outstation of the Institute of Inorganic Chemistry (Graz University of Technology) from Graz, Austria.

In this talk will be presented the experimental possibilities of the Austrian SAXS-beamline showing examples from pharmacy and material science.

VACUUM CONTINUOUS CASTING OF SHAPE MEMORY ALLOYS

Gorazd Lojen
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The technical importance of most engineering materials is based on their mechanical, electrical or magnetic properties, which should, for most materials, be as independent as possible from environmental influences. Beside these conventional materials, there is another group known as functional materials. Functional materials are not as interesting for their properties under certain conditions, as they are for how they respond to certain stimuli from the environment. Among others, shape memory alloys (SMA) belong to this group.

SMA exhibit unconventional correlation of strain, stress and temperature, which is based on crystallographically reversible thermoelastic martensitic transformation. It can be either temperature-triggered or stress-triggered. In martensitic state, SMAs are capable of large apparently plastic deformation (pseudoplastic deformation). The temperature-triggered reverse transformation ($M \rightarrow A$) in pseudoplastically deformed material is accompanied by unusually large strain (shape memory effect – SME). If external forces counteract, the stress can strongly increase, whereby a constructional element is able to perform mechanical work. The stress-triggered martensitic transformation is also accompanied by unusually large strain, but in this case the strain is apparently elastic and without significant changes of stress level (superelasticity - SE).

The transformation temperatures (start- and finish-temperature of martensitic transformation) are very sensitive to chemical composition, while mechanical properties, achievable sizes of SME and SE and the functional fatigue are strongly influenced by impurities, the most detrimental being oxygen, nitrogen and carbon. At the same time, the workability of most SMA is quite poor. Manufacturing of semi-finished and finished products of smaller cross-sections from slabs and ingots is difficult, time- and work-consuming and therefore costly. In this regard, essential simplification of production could be possible by utilisation of continuous casting processes, which enable production of sheets, foils, ribbons, wires, rods, tubes etc. directly from the melt.

Vacuum-continuous casting combines the advantages of vacuum-melting with advantages of continuous casting. Through melting under vacuum, contact with active gases (oxygen, carbon dioxide, nitrogen, hydrogen...) and melting loses are prevented and degassing of the melt is assured. In this way, higher purity can be achieved and the composition-control is much easier, while continuous casting enables faster and cheaper production of small cross-section semi-finished products.

A NOVEL SENSOR FOR REAL-TIME NEUTRAL ATOM DENSITY MEASUREMENT (application on treatment of viscose samples)

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Nowadays plasma material treatment is present both in academia/research and industry. One aspect of plasma material treatment is activation of materials that are used as an absorbent layer in wound patches (chronic wounds of diabetes patients). The absorbent layer is usually made of nonwoven viscose fibers. Absorbent fiber properties are not optimal due to the moderate hydrophobicity of viscose. To achieve better absorption, the surface of the absorbent layer has to be activated. In our case we used weakly ionized low pressure (ICP) oxygen plasma. O-atoms that chemically react with the viscose surface cause the surface to saturate with polar functional groups (better wettability). Due to quite high O atom flow, the surface functionalizes quickly (few seconds) after which the etching of the viscose starts. Only putting the viscose sample into an ICP reactor is not enough, because the surface is being etched while the interior is not functionalized. This is due to the presence of strong oxygen atom gradients transversely to the surface of the sample. In order to achieve functionalization of the interior, a directed flow of atoms through the porous sample was used [1] in a configuration with RF ICP plasma reactor. To control the process measurement of O atom density behind the viscose sample (in the flow direction) O atom diagnostic method is needed. A standard catalytic probe (SCP) is a fairly simple and accurate diagnostic method that exploits O atom recombination to heat a catalytic tip that is used as a thermocouple [2]. However, a single measurement with SCP takes few 10 s and is therefore inappropriate for process control. Also, while working at low tip temperatures (couple 100 °C) the SCP is practically useless since the reaction products of interaction between viscose and atomic oxygen contaminate probes' tip.

To be able to measure oxygen atom density in real time an innovative Laser Optic Catalytic Sensor (LOCS) was used [3]. The sensor employs heterogeneous surface recombination of neutral oxygen atoms on the catalytic tip made from nickel. The ball shaped tip is mounted on an optical fiber which is connected to a suitable electronics containing a diode laser (808 nm) for heating the catalyst tip, an IR light detector and a control device. The tip is heated to a high temperature which is sustained at a constant level. The laser power is adjusted automatically to sustain tip temperature according to the intensity of heterogeneous recombination of neutral oxygen atoms on the catalyst surface. The power needed for sustaining the selected catalyst temperature therefore depends on the intensity of heterogeneous surface recombination and thus on the density of neutral oxygen atoms in the vicinity of the probe tip. The sensor was set behind the viscose sample in the flowing afterglow and used to measure O atom densities in real time at different RF generator power.

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APPLICATION OF LOW TEMPERATURE ATMOSPHERIC PRESSURE PLASMA IN DENTAL MEDICINE

Vedran Šantak[1,2], Rok Zaplotnik[3], Alenka Vesel[3], Iva Šrut Rakić[1], Zrinka Tarle[4], Slobodan Milošević[1]
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The oral cavity is a microbial habitat with over 700 species that live in harmony with the human body [1]. Hard dental tissues include dentin, cementum and enamel which makes main building material of teeth. This is a group of tissues that was subjected to various degrees of mineralization during development, which is typically in the form of hydroxyapatite. Because of their environment teeth interact continuously with physiological fluids, containing biologically important ionic species [2]. The behavior of this species can affect the solid, the environment and biological system. Recently, it was shown that low temperature atmospheric pressure plasma jet (APPJ) could be applicable to therapies in field of dental medicine [3]. The opportunity to examine and understand interactions between APPJ and hard dental tissue has not been fully exploited particularly at the fundamental level where surface science techniques can provide detailed information [4]. To asses interaction between APPJ and hard dental tissue, XPS, AFM, OES and contact angle measurements were conducted. Sample preparation for surface techniques will be discussed in sufficient detail. After the treatment with APPJ chemical, morphological and wettability changes were revealed on the surface of the dentin and enamel. This results bring APPJ closer to clinical application in dental medicine [5, 6]

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MODIFICATION OF BIOMATERIALS FOR SELECTIVE ADHESION OF CELLS

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Adsorption of serum proteins and endothelia cell growth and proliferation on a plasma modified polymer surfaces is a new field of interdisciplinary science and as such not sufficiently explored. Polymer materials were treated in plasma with exactly determined and optimized parameters. With systematic treatment under certain conditions and analysis of the surfaces after plasma treatment, we set the optimal conditions to obtain the best biocompatibility. Faster and better endothelialization was achieved on the surface of the polymers, treated in oxygen plasma. With latter, we significantly improved the biocompatibility of artificial polymer material, which as such, could have an important role in vascular reconstructive surgery, where it is necessary to replace the damaged part of the blood vessel with an artificial vascular graft.

Our results, regarding cell adhesion on plasma treated polymer surfaces clearly show that plasma treatment has an important effect on cell adhesion, proliferation and morphology as well as on oxidative homeostasis. Plasma treatment leads to changes in chemical surface composition of PET polymer reflecting different adsorption affinity of proteins for polymer surface. Modification of polymers with oxygen or CF4 plasma increases binding cell culture media components like proteins and amino acids. However, characteristics of oxygen treated surface are the most favorable for protein adhesion forming a thick layer on the polymer surface. Contrary properties of fluoride plasma treated surfaces probably lead to different conformational binding of proteins forming a thinner layer. High protein content on the surface has a beneficial effect on endothelial cell adhesion and proliferation consequently leading to significantly faster endothelialization of polymers modified with oxygen plasma. Furthermore, as endothelialisation of vascular grafts would make them non-thrombogenic, it is necessary to reveal the optimal surface modification that would selectively enable the endothelial cell growth. Oxidative homeostasis of cells on polymer surfaces is another important factor, when considering cell viability. It is proven that plasma treatment induces intracellular ROS production; as well plasma surface modification of polymers has important effect on cell oxidative homeostasis.

Our results indicate that a combination of optimal plasma parameters and chemical processes can lead to significant improvement in biocompatibility of material and to desired biological response. Thus, oxygen plasma-treated prosthetic implants could have an important role in vascular reconstructive surgery. Nevertheless, all of the studies were performed *in vitro*, so prior *in vivo* applications, experimental studies *in vivo* are necessary to be performed and tested.

EFFECT OF DISTRIBUTION AND SIZE OF CARBIDE PARTICLES ON CREEP DEFORMATION ACTIVATION ENERGY

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Creep deformation is a slow and continuous deformation of steel, which usually takes place at elevated temperatures where changes in the microstructure are governed with the diffusion of elements in the substitution solid solution in ferrite. For the creep deformation, movement of dislocations is necessary and consists of two processes, gliding and climbing. All components of the microstructure which represent obstacles to dislocation movements increase creep resistance of the steel. Intensity of the dislocations movement depends on the activation energy for this process. Activation energy for creep can be calculated, however, the effect of changes in microstructure and changes in precipitates distribution is often not taken into account.

Therefore, the focus of our work was to determine the effect of the distribution of precipitates in steel microstructure on the creep activation energy. The effect of precipitates distribution was examined on X20CrMoV12-1 (X20) and X10CrMoVNb9-1 (P91) steel, subjected to different annealing times (2h and 400h) in order to obtain two different distributions of precipitates: uniform distribution of precipitates in ferrite matrix and the presence of precipitate stringers at the ferrite grain boundaries. Creep rate and creep activation energy were calculated for different creep test conditions, using tensile stress of 170 MPa and four different temperatures (550, 580, 610 and 640°C).

Using Arrhenius equation, activation energy was calculated for experimental and calculated creep rates. Average value of calculated theoretical activation energy is 249 KJ/mol, which is in the range of quoted values for α iron self-diffusion activation energy. Experimental and calculated creep rates agree for creep tests carried out at low temperatures. However, at higher test temperatures experimental creep rates are significantly higher and obtained values for activation energy are from 400 KJ/mol to 550 KJ/mol. The explanations of these observed deviations will be presented and discussed in the presentation.

TAILORING OF LAYERED 2D MATERIALS: EPITAXIAL GRAPHENE AND BEYOND

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Attractive properties of graphene and follow-up 2D materials can be exploited in various applications where the electronic structure of these materials are easily subjected to tailor-made solutions. First of all, the ability to synthesize large-area and high quality 2D materials, preferably in an inexpensive way, and transfer them to any support, is crucial for the successful development of future applications. Moreover, the atomic-scale control of synthesis process and of further material manipulation is of academic and applicative importance.

In this talk, I will review some of our recent efforts regarding 2D materials. In the ultimate 2D material, graphene, a chemical adsorption either "on top" or "underneath" it, is a suitable tool for the charge carrier modifications. In epitaxial graphene systems deposition of atoms and molecules often leads to intercalation where species are pushed between graphene and its support. Besides the common effect of the charge transfer, the intercalation can affect the binding interaction and more subtle properties of graphene, e.g. magnetism. In fact, properties of layered materials, including copper- and iron-based superconductors, dichalcogenides, topological insulators, graphite and epitaxial graphene, can be manipulated by intercalation. We explain the microscopic mechanism and dynamics of graphene intercalation. Another direction of graphene electronic structure tailoring is related to a precise stress control which can be realized by graphene growth on flat or specifically on a stepped surfaces and we focus to such systems in order to exploit uniaxial strain engineering. Finally, the ability to grow 2D dichalcogenide materials via atmospheric pressure CVD synthesis is demonstrated.

CHARACTERIZATION AND APPLICATION OF SiO_2 SURFACES MODIFIED WITH AMINOSILANES

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Modification of inorganic surfaces with organosilane self-assembled molecules (SAM) is very interesting research field due to a wide range of applicability. By modification we change both, chemical and physical surface properties and consequently functionality of whole material. The most commonly used SAM molecules for modification of silicon surfaces are aminosilanes. The amount of coatings and the morphology of aminosilane layers depend on many parameters. For the preparation of homogenous and reproducible coatings it is essential to understand surface chemistry, molecular interactions and modification parameters.

Single crystal silicon wafers were modified with different silanes (aminoalkylsilanes: APTMS, APDMS, APEMS, APTES; aminoarylsilane APhS; aminoalkylsilane derivatives: UPS, EDA, DMS and alkylsilane ODS). Chemical composition and morphology of the modified surfaces were determined using surface sensitive characterization techniques XPS, AFM, SEM, ToF-SIMS and MTR-IR. Results show that the reactivity of aminosilanes with the Si-oxide surface and the polymerization of aminosilanes depend on the number of possible bonding sites of aminosilane molecule. Further we studied the influence of solvent on the modification process. We discovered that the amount of coating and consequently the morphology of modified surface can be easily controlled with the use of the appropriate solvent. The structure of silane layers is also strongly related to the structure of reactive organic part in the silane molecule. Obtained results show that it is necessary to control precisely all the modification parameters to obtain smooth and uniform aminosilane coverage, otherwise uncontrolled silanization leads to the formation of thick and rough aminosilane layers, which may significantly influence on the application of modified surfaces.

The aim of the study was also to demonstrate the applicability of aminosilane modified silicon surfaces. From the obtained results we determined optimal silanization conditions and modified silicon capacitive microsensors with a purpose of using them for vapor trace detection of explosive gases. We demonstrated that the sensors modified with various silanes respond differently to the presence of TNT vapor.

WHAT CAN WE LEARN FROM A COFFEE MACHINE?
(about the quantum structures)

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Transport phenomena in modern quantum structures are very complex and the conductivity path for charged particle is not easy to predict. This is the point where we can get help from a coffee machine.

We shall give a short introduction into percolation theory. We shall start the discussion with a discrete percolation and analyze the network options which would be suitable to describe transport phenomena in solar cells based on quantum elements. Further we shall expand our description to continuous percolation and describe nucleation based on few models.

If time permits we shall describe application on random resistance network and analyze its behavior in classical linear regime.

QUANTUM DOT LATTICES OF Ge/Si CORE/SHELL QUANTUM DOTS IN ALUMINA GLASS MATRIX FOR APPLICATION IN SOLAR CELLS

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Materials consisting of semiconductor core-shell nanostructures, like nanowires or quantum dots attract special attention due to their highly adjustable electronic structure and optical properties. Especially interesting for the application of such materials in solar cells is the fact that Ge/Si core/shell quantum dots have a type II band alignment, leading to the separation of charge carriers [1,2].

We have recently developed a method for the growth of self-assembled core/shell Ge/Si quantum dots in an amorphous alumina matrix [3]. The method is very simple and consists of magnetron sputtering deposition process of Al_2O_3/Ge/Si multilayer. The formed dots are spontaneously ordered in a three-dimensional body centered tetragonal quantum dot lattice.

The light absorption properties of these complex materials are significantly different compared to the films that form quantum dot lattices of the pure Ge, Si or a solid solution of GeSi prepared by the same method. The core/shell-based materials show a strong narrow absorption peak, while others show only broad, low-intensity bands. The properties of the absorption observed for the core/shell quantum dots is in accordance with the theoretical predictions from Ref. [1], and it is characteristic for type II confinement.

Tuning the radius of the Ge core and thickness of the Si shell we are able to tune the position of the absorption peak in a broad range of energies. To demonstrate that effect several sets of Ge/Si/Al_2O_3 - films differing by deposition conditions are compared.

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SILICON NANOSTRUCTURING FOR ADVANCED APPLICATIONS

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Inorganic semiconductor materials have for a long time been a standard member of electronic devices with silicon being the material of choice for most applications. Introducing structuring in silicon has proven to be advantageous for its many properties and applications. Introduction of silver nanocrystals in the porous silicon is promising for Surface Enhanced Raman Sppectroscopy (SERS). Here we present the results on preparation of stable and uniform SERS solid substrates using macroporous silicon (pSi) with deposited silver and on the devices based on heterojunctions of porous silicon formed by anodisation in HF-based electrolytes and organic thin films formed by vacuum evaporation. Macroporous silicon is produced by anodisation of p-type silicon in hydrofluoric acid. The as prepared pSi is then used as a template for Ag depositions. The noble metals were deposited in three different ways: by immersion in silver nitrate solution, by drop-casting silver colloidal solution and by pulsed laser ablation (PLA). Substrates obtained by different deposition processes are evaluated for SERS efficiency using methylene blue and rhodamine 6G at 514.5, 633 and 785 nm. Substrate concentrations detected in most of the cases were in the nanomolar range suggesting that it may be possible to detect lower analyte concentrations when using porous instead of crystal silicon template. Heterojunction interfaces between organic and structured silicon substrate in devices show promise in using advantages of both materials simultaneously. For inorganic part of the heterojuction we choose porous silicon due to its compatibility to established CMOS technology and its enormous surface area being presented to thin organic films. For organic part of the junction we investigated the applicability of various organic semiconductor thin films for optoelectronic applications. Our porous silicon-based devices show optical response and moderate sensitivity in the infrared spectral region.

UPPER KOLPA RIVER – THE CRADLE OF SLOVENIAN-CROATIAN VACUUM EXPERTS

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Many of important Slovenian and Croatian vacuum researchers were at home in the same Upper Kolpa Region where 22nd Croatian-Slovenian meeting on vacuum techniques took place in 2015. An interesting regional fact is explained by the extraordinary technical heritage of the population of upper Kolpa region, beginning with the local medieval mills. In 1651 forges processing of Carniola iron ore was set up by Count Peter Zrinjski in Čabar. The products were exported by the harbor of Bakar. The 19th century immigrants from Slovenian side Aleksander Vilhar (*1814; † 1868) and Wilhelm Vilhar (*1840), produced one of the first Croatian saw steam powered using vacuum techniques in Milanov vrh between Čabar and Prezid in the year 1847/48. Aleksander's grandson Dušan Vilhar (*1867; †1913) produced a steam sawmill on the basis of more modern vacuum techniques in Gerovo. The physicist Franc Kvaternik (Osilnica *1919; †1981) examined it there. His home was just few miles on the west across the Kolpa River. Franc Kvaternik was the first writer of the post-war Slovenian physics textbook. Among other vacuum related experts originating from the upper Kolpa valley are dr. Janez Kovač (Srobotnik), dr. Matjaž Panjan and dr. Peter Panjan (Sodevci). The present writer from nearby Fara was commissioned to describe their achievements.

GISAXS CHARACTERIZATION OF THE ION-IRRADIATION INDUCED SURFACE TRACKS

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Ion- irradiation of materials is a powerful tool for the design their properties via changes induced within the ion tracks. Characterization of these changes is usually difficult, especially if the tracks are completely or partially below the surface as well as in amorphous systems where the contrast between the track and the surrounding material is low. Therefore, a simple and efficient method for the characterization of ion beam induced changes and all similar effects in materials is of great importance.

Here we present models and a program for the analysis of different types of ion-irradiation induced tracks at the material surface or just below it by GISAXS (grazing incidence small angle x-ray scattering). The structure of the formed tracks is often non-continuous, i.e. the tracks consist of nano-sized structures aligned along the ion beam trajectory. The proposed method allows determination of all important ion track properties including their structure, shape, size, separation as well as all properties of the formed nano-objects for the non-continuous tracks. The efficiency of the method is demonstrated using ion tracks produced in Ge+ITO mixture film irradiated by 15 MeV Si ions. The developed models are incorporated into a new freely available program *GISAXStudio* for processing and analysis of materials modified by ion beams using GISAXS.

TOF-SIMS, XPS AND AFM CHARACTERIZATION OF AMINOSILANE MODIFIED Al-OXIDE SURFACES

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Aminopropyltrimethoxysilane (APTMS) was applied for the modification of Al-oxide surfaces. Surfaces modified with organosilanes are now widely used in many industries due to their large number of application: adhesion promoters, in chromatography, as sensors or biosensors, in medicine, corrosion protection, etc. The influence of the different solvents on the morphology of the modified Al-oxide surfaces was studied since the possible heterogeneity may significantly influence the application of such surfaces. Before the modifications all the Al-oxide surfaces were cleaned with HF and then oxidized in a controlled manner using an oxygen plasma. We deposited the self-assembled silane layers from a diluted solution of APTMS in three solvents with different polarities (toluene, acetonitrile and ethanol) under various reaction conditions. Surfaces were characterized using Time-of-flight secondary ion mass spectrometry (ToF-SIMS), X-ray photoelectron spectroscopy (XPS) and Atomic force microscopy (AFM). The chemical structure was determined with ToF-SIMS; surface composition and the chemical bonding were determined by XPS; and surface morphology and roughness was investigated using AFM. Our results show successful bonding of APTMS molecules to the Al-oxide surface and that amount of coatings strongly depends on the type of solvent. Using ToF-SIMS technique we have proved the covalent bonding of the APTMS molecules with Al-oxide surface (signals at 71 = $SiOAl^+$, at 103 = SiO_2Al^-…) even in polar solvents which may cause solvolysis of deposited layers.

PRETRAŽNA MIKROSKOPIJA TRANSMITIRANIH IONA POMOĆU PLINSKOG DETEKTORA

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Prolaskom brzih iona kroz tanke uzorke dolazi do gubitka njihove energije koja je proporcionalna debljini uzorka na mjestu prolaska iona. Korištenjem pretražnog (scanning) fokusiranog ionskog snopa moguće je raditi oslikavanje (imaging) promjena debljina uzorka kroz koji ioni prolaze. Što je energija iona manja odnosno što je veća težina iona, mogućnost detekcije većih promjena u debljini uzoraka raste. Kako bi omogućili oslikavanje promjena u debljini na nanometarskim razinama, potrebno je koristiti teške ione, čija je detekcija konvencionalnim poluvodičkim detektorima jako otežana zbog brzog oštećenja samog detektora. Nedavno su razvijeni jednostavni plinski proporcionalni detektori sa SiN prozorima koji su neosjetljivi na ozračavanje teškim ionima [1,2]. Također je njihova energijska razlučivost u MeV-skom području energija bolja od silicijskih detektora. U radu je opisan dizajn i konstrukcija minijaturnog plinskog detektora, ispitivanje njegovih karakteristika za detekciju teških iona, te demonstracija korištenja detektora za STIM metodu na ionskoj mikroprobi akceleratorskog sustava na Institutu Ruđer Bošković.

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IRRADIATION OF Al/Ti MULTILAYER BY PICOSECONDS LASER BEAM AND FORMATION OF NEW PHASES

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Al/Ti multilayer system of 15 alternate Ti and Al thin films was deposited on a Si substrate to a total thickness of 900 nm. Laser treatment was performed in air by defocused Nd:YAG laser pulses (150 ps) with energies of 7 and 10 mJ. Laser beam was scanned over the 5x5 mm surface area. Analyses were performed by Auger electron spectroscopy (AES), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM) and transmission electron microscopy (TEM). Nano-hardness measurements were performed by Vicker's method with applied load of 5 mN.

Obtained results show that irradiation with picoseconds laser pulses leads to the formation of Ti_2O_3 in the top Ti layer. This process is more pronounced for higher energy and/or higher number of applied laser pulses. On the surface of all samples very thin amorphous TiO_2 layer was formed. Laser irradiation induces surface melting and transformation of relatively flat surface into mosaic shaped one for samples treated with higher energy and higher number of applied laser pulses.

STRUCTURE AND NANOHARDNESS OF TaN_x THIN FILMS PREPARED BY REACTIVE MAGNETRON SPUTTERING

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Tantalum nitride films exhibit some remarkable properties - high hardness, thermal stability, chemical inertness, good conductivity, catalytic activity - which make them attractive for various applications. Here we present the results of structural and mechanical characterization of tantalum nitride films widely differing in nitrogen content. Series of TaN_x thin films (thickness ~ 100 nm) were produced by reactive magnetron sputtering of pure Ta target in the mixed $Ar+N_2$ working gas atmosphere. Different stoichiometric composition of the films was achieved by varying the nitrogen content in the working gas mix at constant total pressure. Deposited films were subsequently annealed for 1h at different temperatures from 450°C to 950°C in 100 degrees steps. The structure and phase composition of the films were determined by the XRD measurements, while nanohardness measurements gave insight into the mechanical properties of the as-grown and annealed thin films. The obtained nanohardness data were compared to the preliminary TaN_x phase diagram in order to establish a relation between mechanical and strucural properties of the investigated films. It is found that nanohardness strongly depends upon phase composition, which can be efficiently controlled in a reactive dc sputtering process and subsequent annealing.

AUTOMATION AND CONTROL FOR ELECTRON BEAM DEPOSITION OF ULTRATHIN MULTILAYER SYSTEMS

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Current trends toward plasmonic multilayer systems design and production, which are composed of ultrathin metal-dielectric composite films, raised the requirements for technical realisation of automation and precise control of deposition procedures.
Here we present the setup of electron beam deposition system which enables layer thicknesses down to 2 nm, with subnanometer control.

Precision determination of proportional-integral-derivative (PID) controller parameters for feedback control of deposition rate, as well as oxygen flow parameters for the deposition of titanium dioxide and silicon dioxide layers, was done in an iterative procedure until optimal set of parameters was found.

This experimental setup has been tested by depositing ultrathin layers of metals (silver, gold, chromium and aluminium) and dielectrics (silicon dioxide and titanium dioxide) in different combinations and in multilayers with up to 25 films.

CHARACTERIZATION OF BIXBYITE-Ta_2N_3 THIN FILMS PRODUCED BY SPUTTERING IN PURE N_2 ATMOSPHERE

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Tantalum nitride thin films were prepared by reactive magnetron sputtering in pure nitrogen atmosphere. The effects of annealing temperature on the film structure, morphology, composition, density and electrical resistivity were investigated. The crystal structure of as deposited film was found to be cubic Ta_2N_3 with a defect fluorite-type structure. This phase is stable up to 850 °C, where it transforms into $Ta_{0.75}N$ phase (NaCl type structure) with one Ta vacancy per unit cell. We found continuous grow of crystallites with annealing temperature. The density of the films, being slightly lower than the density of theoretical Ta_2N_3, is found to be thermally stable. Analysis of the composition showed that the nitrogen located at the grain boundaries out diffuses from the film, reducing the N/Ta atomic ratio from 2.05 to 1.38 for as deposited and annealed film at 950 °C, respectively. Electrical resistivity decreases with annealing temperature, and it depends on the nitrogen concentration between crystallites through the percolation theory. For the bixbyite-Ta_2N_3 we found lowest resistivity of ~5 mΩcm for film annealed at 750 °C, while the $Ta_{0.75}N$ films showed resistivities in the range of 1.7-3 mΩcm.

ZnO NANOPARTICLES PRODUCED BY PULSED LASER ABLATION IN WATER MEDIA FOR UV PROTECTIVE COATINGS

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Low dimensional ZnO structures have attracted a considerable interest in the recent years due to the unique properties and their potential for application in areas such as optoelectronics and photovoltaic.

Degradation of the UV-protective coatings (mostly polymer-based) due to the prolonged light exposure is a well know problem in the coatings industry. A so called "nano-coatings", made of ZnO nanoparticles are among the best candidates to replace standard UV-protective coatings.

Here, we present a preliminary study on the optical properties and morhphology of ZnO nanoparticles produced by pulsed laser ablation in water media. ZnO nanoparticles were prepared by pulsed laser ablation of a pure ZnO target (99.999%, Kurt J. Lesker) and ZnO with addition of 3% of Al in deionized water. The Zn target was irradiated by a Nd:YAG laser with λ = 1064 nm, 100 mJ of output energy and operating at 5 Hz.

The optical absorbance in the UV–visible region of colloidal ZnO nanoparticles was recorded using a UV-VIS spectrophotometer Perkin Elmer (Lambda 25). The spectra clearly exhibit a ZnO surface plasmon peak at 335 nm.

Further, the morphology of ZnO nanoparticles was investigated by a field emission scanning electron microscope (SEM, Jeol 7600F). Spherical-shaped particles with diameter around 120 nm were formed using a ZnO with 3% of Al target for the preparation of ZnO nanoparticles. When a pure ZnO target was used, interestingly, a 3D ZnO network was formed.

STUDY OF INFLUENCE OF LASER ABLATION SYNTHESIZED SILVER NANOPARTICLES ON E. COLI BACTERIA

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Laser ablation in liquids is a very convenient technique for production of nanoparticles free of chemical byproducts. Silver nanoparticles made in this way could show antimicrobial activity. For the purpose of this work we produced colloidal silver nanoparticles in water using a 1064 nm ns Nd:YAG laser at 100 mJ output energy and 5 Hz repetition rate. Size-distribution of nanoparticles was obtained from atomic force microscopy (AFM) images. We found the average diameter of silver nanoparticles to be around 10 nm with a relatively narrow size distribution. Obtained colloidal solution is stable on a scale of few months, which implies that nanoparticles are well dispersed and/or charged. We use this colloidal solution to investigate antimicrobial effects of silver nanoparticles on different strains of E. coli bacteria where the changes on bacterial membrane are monitored by AFM. Here we present our preliminary results.

STABILITY OF TiO_2 NANOTUBES AFTER ANNEALING

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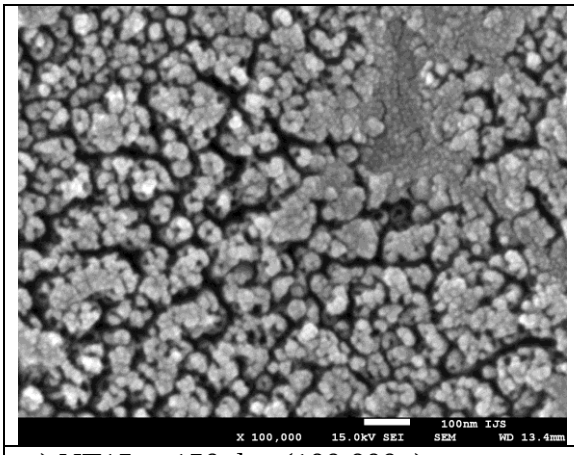
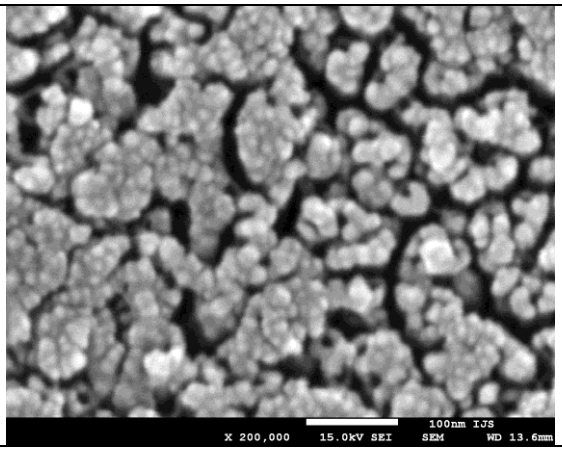
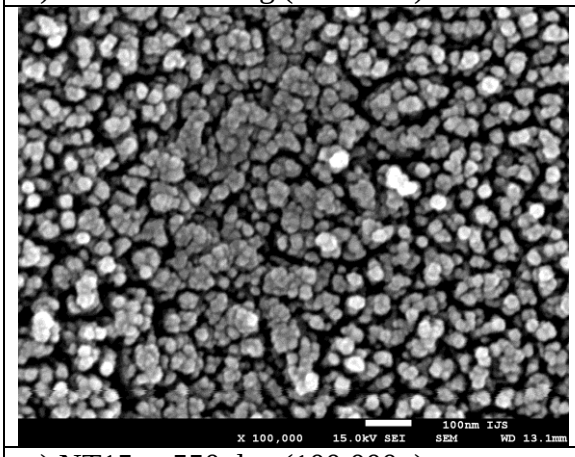
Titanium dioxide (TiO_2) nanotubes have found many uses in medical applications and photovoltaics. This study is dealing with the stability of TiO_2 nanotubes after annealing. Nanotubes with radius of 15 nm (NT15) were treated at three different annealing temperatures 150, 350 and 550°C. Later on, samples were gold coated and analysed by scanning electron microscope (SEM) at two different magnifications (100.000x and 200.000x). Thickness of gold coating was 5 nm and SEM accelerating voltage was 8 keV. It was observed that temperature affects the morphology of nanotubes in an undesired manner. Even at lower temperature of 150°C we notice that nanotubes are disrupted. In some places we can find patches where surface morphology of nanotubes is completely lost. At higher magnification we observe on NT15 at 150°C that nanotubes are closed and in some parts totally destroyed. At annealing of NT15 at 350°C similar effects are noticed, while at annealing at 550°C we observe even more sever morphological changes. In this case we practically cannot see any nanotubular structure on the surface, it seems as all nanotubes are covered or destroyed.



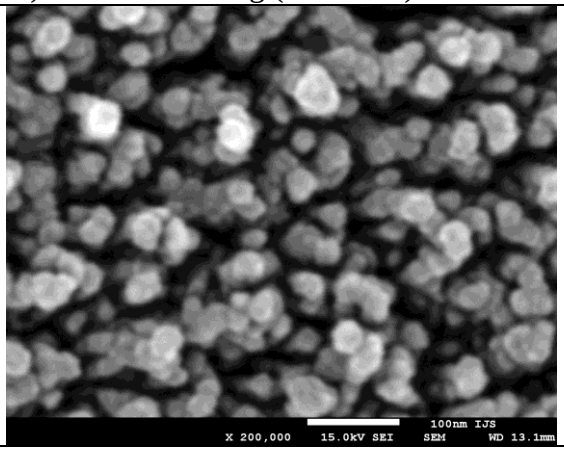
a.) NT15 at 150 deg (100.000x)



b.) NT15 at 150 deg (200.000x)



c.) NT15 at 550 deg (100.000x)



d.) NT15 at 550 deg (200.000x)

SYNTHESIS AND CHARACTERIZATION OF Pt NANOCATALYSTS AT METAL OXIDE BASED SUPPORTS FOR FUEL CELLS APPLICATION

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Proton exchange membrane fuel cells (PEMFCs) are promising candidates for an environmental friendly and efficient energy conversion, with many prospective practical applications in portable, stationary and transport devices. Platinum based catalysts exhibit the best catalytic activity and stability for reactions taking place in fuel cell: oxygen reduction and hydrogen oxidation. Pt nanoparticles homogeneously distributed over carbon support (commercially named Vulcan and Ketjen black) are state of the art catalyst materials for both anode and cathode reactions. However, high cost and scarcity of platinum are the main problems to be solved before commercialization of these clean energy providers. Many efforts have been made to achieve sufficient low Pt loading (mg(Pt)/cm) to gain satisfactory power density: 0.9 – 1.2 g(Pt)/kW, for stationary, un-interrupted power supply, while less than 0.4 g(Pt)/kW for large scale commercialization in automotive applications is required. Limited durability caused by degradation of the carbon support, usually used in commercially available Pt catalysts, increases total fuel cell costs and reduces its life time. One of the key requirements for this class of fuel cells application is that the fuel cell must be tolerant of frequent start-stop cycling. During startup and shutdown procedure high potential difference is created, causing carbon corrosion and oxygen evolution on the air cathode. This mechanism has been called "reverse-current decay mechanism". Namely, carbon corrosion to CO_2 could cause permanent carbon loss, loss of the catalytic activity, even the whole catalyst degradation. Platinum further accelerates the carbon corrosion rate, leading to agglomeration of the catalyst particles and severe degradation of fuel cell cathode.

Here we present the following research activities: (*i*) development and characterization of new interactive metal oxide based supports with improved durability for fuel cells application (titanium oxide and tin oxide based supports); (*ii*) development and characterization of new Pt nanocatalysts on above mentioned supports and (*iii*) electrochemical characterization-testing the catalytic activities of these catalysts for oxygen reduction as well as for hydrogen oxidation and methanol oxidation reaction. The following methods were used for physical characterization of the synthesized materials, X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and X-ray photoelectron spectroscopy (XPS). For the characterization of the catalytic activities of the synthesized catalysts electrochemical techniques cyclic voltammetry and linear sweep voltammetry at rotating disc electrode were applied.

MICROSTRUCTURAL AND MECHANICAL PROPERTIES OF Ag-Mo THIN FILMS

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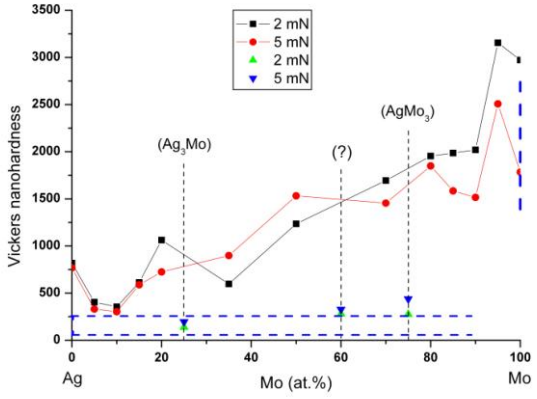
Thin film alloys of equilibrium immiscible Ag-Mo system have been prepared in a full range of composition by codeposition of pure silver and pure molybdenum sputtered by two independently controlled magnetron sources. Deposition rate onto various substrates at room temperature was about 0,2-0,3 nm/s, and the final film thickness was about 0,5 μm. The prepared films were annealed at 300K for 8 yrs. The structure of prepared films was examined by the XRD, GISAXS and SEM methods. The Ag-Mo system Glass Forming Range predicted by atomistic theory is $Ag_{90}Mo_{10}$-$Ag_{12}Mo_{88}$. However, no completely amorphous films have been obtained in the predicted GF composition range. Also, some complex multilayer structures were observed by the SEM, indicating elemental separation either during deposition or annealing. On the Mo-rich crystalline side the grass-like surface of the film is observed.

The mechanical properties of Ag-Mo alloys on sapphire substrates have been examined by measurement of nanohardness (load 2mN or 5mN) in a whole range of composition. It was found that nanohardness generally increases with addition of harder component (Mo), with some notable exceptions - at alloy compositions where hypotethical Ag_3Mo and $AgMo_3$ might be expected. The results will be discussed in terms of film structure.



ULTRAFAST ELECTRON DYNAMICS IN QUASY-1D Pb NANOWIRE ARRAYS ON STEPPED Si SURFACE

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As a prototypical quasi-1D system Pb nanowires adsorbed on a Si(557) substrate were studied experimentally to explore the impact of structural and electronic band anisotropy to the hot electron lifetimes in directions parallel and perpendicular to the nanowires. These particular aspects were investigated using angle-resolved two photon photoemission (2PPE) and femtosecond time-and angle-resolved 2PPE. In the experiments, two unoccupied electronic bands were identified which were shown to be located on the lead wires. The analysis reveals a momentum-dependent population dynamics.

CLOSELY PACKED Ge QUANTUM DOTS IN ITO MATRIX: INFLUENCE OF Ge CRYSTALLIZATION ON OPTICAL AND ELECTRICAL PROPERTIES

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Simple methods for fabrication of semiconductor quantum dots in transparent oxide matrices play an important role in various nanotechnology applications. Here we present a method for the low-temperature production of closely packed crystalline Ge QDs embedded in ITO matrix. The films are produced by magnetron sputter deposition followed by thermal annealing. The crystallization of Ge is found to occur already at 300^{o}C, what is significantly lower than the crystallization temperature of Ge produced by the same method in silica or alumina matrices. The dependencies of the conductivity of the films and their optical properties on the materials structure, Ge content in the ITO matrix and the annealing conditions are demonstrated. The crystallization of Ge causes a huge increase the films conductivity and it also affects the films optical properties.

THE INFLUENCE OF AN OBLIQUE MAGNETIC FIELDS ON MAGNETIZATION PROCESSES OF MAGNETIC RIBBONS

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The measurements on amorphous ferromagnetic ribbons have shown that direct (J_D) and alternating (J_A) core-current can affect the *M-H* loop and cause effects like decrease the coercive field *H*c (hence decrease hysteresis loss *E*), shift the center of the *M-H* loop (*C*) along the *H*-axis, and change the permeability, maximum (*M*m) and remanent (*M*r) magnetization of the sample. These effects are associated with transverse field *H*p generated by J_D (static *H*p). The magnitude of *H*p increases linearly with the distance from the center of the ribbon and reaches the maximum at its surface, hence the term "surface field". The influence of surface fields Hp on the parameters of M-H loops of amorphous and nanocrystalline $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ ribbon and amorphous VITROVAC 6025Z ribbon has been investigated [1, 2]. In amorphous $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ ribbon Hc decreases with Hp with unchanged Mm, whereas in nanocrystalline $Fe_{73.5}Cu_1Nb_3Si_{15.5}B_7$ and VITROVAC 6025Z ribbons Hc increases with Hp and Mm decreases with Hp. This unusual increase of Hc with Hp is ascribed to the influence of Hp on the surface domain structure (SDS) and strong interaction between SDS and inner (main) domain structure (MDS) in these materials. A model is developed which takes into account this influence and explains the experimental results [3].

[1] S. Sabolek, E. Babić, D. Posedel and M. Šušak, Phys-Stat.Sol (a) 202 (2005) 1161
[2] Petar Popčević, Emil Babić and Stjepan Sabolek, IEEE Transactions on Magnetics, 44 (2008) 2095
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LARGE SCALE TRANSFER AND CHARACTERIZATION OF A PERIODICALLY NANO-RIPPLED GRAPHENE

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Nano-rippled graphene, a structurally modified graphene, presents a novel material with a large range of possible applications including sensors, electrodes, coating, optoelectronics, spintronics and straintronics. In this work we have synthesized macroscopic single layer graphene with well-defined uniaxial periodic modulation on a stepped Ir(332) substrate and transferred it to a dielectric support. An important feature of this system is that the graphene lattice is rotated in several different, well-defined orientations with respect to the direction of the periodic modulation and that the periodicity of the modulation can be modified by changing the graphene synthesis parameters. The applied fast transfer process does not damage the Ir crystal which can be repeatedly used for graphene synthesis. Upon transfer, a millimeter sized graphene flake with a uniform periodic nano-ripple structure is obtained, which exhibits a macroscopically measurable uniaxial strain. The periodic one dimensional arrangement of graphene ripples was confirmed by AFM and polarized Raman measurements. This rippled graphene system, with the possibility to control its morphology, could be tailored to fit a range of applications.

DEPOSITION OF PLASMA POLYMERIZED ANTIMICROBIAL POLYMERS

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Antimicrobial effect is the surface property connected also to the kind of functional groups present on the surface of material [1]. Surfaces enriched by amino functional groups are appropriate for medical applications as the active amino groups bound to biological material like bacteria. Plasma polymerization provides uniform functional surfaces and vast variety of parameters is available to alter the composition and properties of precipitated polymers. Deposited polymers are solvent free, highly cross-linked and adhere well to the polymeric, glass or metal surfaces. Plasma polymers were deposited onto polyethylene terephthalate mesh inside a metal plasma reactor (modified GEC cell) utilizing inductive coupled radio-frequency plasma at 13.5 MHz. Ammonia and hexane in different proportions from 25.5 % to 90.2 % of ammonia were used as plasma gases to obtain polymer films with nitrogen containing functional groups. Bands in vibrational spectra of synthesized polymers indicate presence of N-H bonds. XPS analysis of plasma polymer surface indicates increased nitrogen content at higher percentage of ammonia in a fed gas, with maximum 28.5 % of nitrogen at 86.0 % of ammonia in a fed gas. Contact angles drop from 80.8 degrees at 31.6 % of ammonia in a fed gas down to 4 degrees for 86.0 and 90.2 % of ammonia in a fed gas.

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SURFACE CHEMISTRY OF 3D PRINTED GUIDING TEMPLATES FOR USE IN ORTHOPAEDIC SURGERY

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Guiding templets used in orthopaedic surgery have gained significant importance in the last decade. By development of 3D printing technologies, the field of personalized medical implants, tools and guiding templates has rapidly grown. 3D printing of personalized guiding templates for orthopedic surgery enables surgeons to detect the exact location for drilling or cutting which increases accuracy and minimizes X-ray usage during surgery. Moreover, such procedures are less invasive and reduce postoperative complications. The materials, used for guiding templates, that come into contact with human body should withstand standard sterilization procedures. New materials, used for 3 D printing are widely studied, especially their chemical stability, as the surface properties should remain unchanged.

For the purpose of our study, the 3D printed guiding templates for use in orthopedic surgery (Figure 1) made by UV photopolymerization of poly (utrethana-acrylate)s were studied. The effects of different sterilization procedures; autoclave, ethylene oxide and plasma on their chemical properties were studied by X-ray Photoelectron Spectroscopy (XPS). In order to use this material for medical application their chemical composition after sterilization should remain unchanged. Differences in chemical composition between different sterilization techniques were observed. Our study showed that only plasma sterilization did not alter chemical composition of the surface.

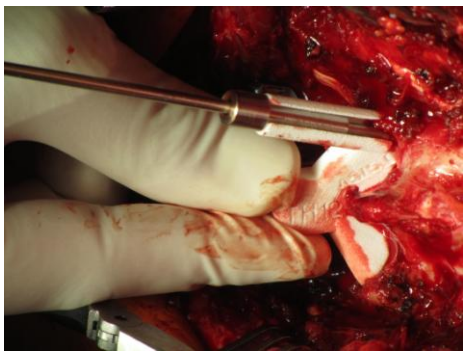

Figure 1. Thoracic pedicle screw hole preparation, assisted by a guiding template, made with the use of 3D printing technology

OPTICAL EMISSION SPECTRA OF GASEOUS PLASMA UPON DEGREASING OF CERAMIC OBJECTS

Miran Mozetič[1], Alenka Vesel[1], Aleksander Drenik[1], Nikša Krstulović[2], Rok Zaplotnik[1,2]
Slobodan Milošević[2]
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[2]*Institute of Physics, Bijenicka 46, 10000 Zagreb, Croatia*

Experiments on discharge cleaning of porous ceramic samples in oxygen plasma were performed. The samples were soaked with oil and exposed to inductively coupled oxygen plasma at the pressure of 75 Pa. Optical emission spectroscopy was applied to reveal the temporal evolution of reactive gaseous radicals suitable for oxidation of organic molecules as well as reaction products. The intensity of the major atomic oxygen lines at 777 and 845 nm predominated at the beginning of the cleaning process and they were an order of magnitude larger than any other spectral feature. The other features observed were H atomic lines from Balmer series and OH bands. The cleaning intensity was indicated by appearance of the CO-radical bands that became predominant when the oxidation of the oil was most intensive. Once the oil was totally removed the intensity of the CO bands dropped below the background. The results show applicability of the technique for monitoring evolution of the products cleanliness in a batch industrial process.

SURFACE PROPERTIES OF PET POLYMER TREATED IN SO_2 AND H_2S PLASMA

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SO_2 and H_2S plasmas were used to modify PET (polyethylene terephthalate) surface and to bind sulphur-containing functional groups to the surface. Plasma was created by RF generator operating at 150 W (E-mode) and the gas pressure was 30 Pa. Samples were placed in the middle of the coil and were treated for different periods. After plasma treatment they were analysed using X-ray photoelectron spectroscopy, atomic force microscopy and water contact angle measurements. SO_3 and SO_4 groups were found for SO_2 plasma-treated surface, while treatment in H_2S plasma caused formation of -SH groups, sulphur deposition and formation of polysulphides. Pronounced changes in the surface morphology were observed for both plasma-treated samples. Water contact angle decreased from 75° to approximately 30° after SO_2 plasma treatment and to 60° after H_2S plasma treatment.

STUDY OF ATMOSPHERIC PRESSURE PLASMA JET FROM OPEN AIR TO VACUUM

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Atmospheric pressure plasma jets (APPJ) attract interest due to many technological applications in biomedicine, materials treatments, food industry etc. Comparing to low-pressure plasmas they offer new possibilities due to simpler setup and ability to operate in atmospheric environment. This work is focused on studying APPJs at controlled under-pressure conditions, contrary to the open air conditions. The APPJ electrode was a copper wire with a diameter of 0.1 mm inserted in a 50 mm long and 1.5 mm outer diameter borosilicate glass capillary tube [2]. Power source was the same as in ref. [3], AC at 25 kHz at about 2.5 kV.

This capillary plasma source was inserted into a glass tube of 11.5 cm diameter and 150 cm length. In order to maintain under-pressure conditions, the tube was connected to a rotary vacuum pump and vacuum gauge while the gas (for example helium, purity 99.996%) input flow was controlled with mass flow controller in the range of several L/min. The experimental setup allows various diagnostics to be applied: imaging, spatially resolved optical emission spectroscopy, determination of voltage-current curves and E-M probe measurements, versus total pressure and mass flow in the tube.

We describe different regimes of the APPJ behaviour depending on the total pressure and mass flow, in terms of the jet lengths, and discuss jet operating mode transitions form atmospheric pressure conditions towards vacuum conditions.

This work has been supported by Croatian Science Foundation under project #2753.

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ATMOSPHERIC PRESSURE PLASMA NEEDLE JET PARAMETERS INFLUENCED BY SAMPLE SURFACE

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[1]*Institute of Physics, Bijenička 46, 10000 Zagreb, Croatia*
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Plasma treatment of samples can be used for different surface modifications. When samples of various sizes, made out of various materials are treated with plasma they change plasma properties. This influence is especially pronounced when atmospheric pressure plasma jets are used for surface modifications. Here we report on reciprocal influence between the sample surface and atmospheric plasma jet. This correlation is important since it changes plasma parameters and plasma itself, depending on the sample-material surface, presence of liquid or treatment distance. However, in experiments and treatments of surfaces with atmospheric plasma jets, this relationship is usually disregarded. The designed multiple harmonics source was applied to a single electrode plasma jet and a comparison between the most commonly used single harmonic (sine voltage waveform) and the original multiple harmonics AC-driven atmospheric pressure plasma jet was performed. Some advantages compared to single harmonics driven jets were discovered. In order to investigate reciprocal influence, we implemented electromagnetic and optical emission spectroscopy characterization of atmospheric multiple harmonic AC-driven plasma needle jet (25 kHz, 2.5 kV, 3 mA). Characterization was performed during treatment of various samples. We have shown that sample material and its distance from the tip of the electrode have a pronounced influence on atmospheric pressure plasma jet electromagnetic and optical characteristics, such as jet length, shape, colour, voltage, current, power, electromagnetic field and concentrations of plasma species. It was shown that for a given flow there is a critical distance (≈ 15 mm) between the tip of the wire and the sample surface for which jet emission intensity, especially ionic, is at maximum. Here we presented the evidence that during treatment of samples with atmospheric plasma jets, one must take into account the reciprocal influence of sample surface and plasma jet, while the sample material, its size and distance from the beginning of the jet changes both discharge and plasma parameters.

PRIMARY STATIC EXPANSION CALIBRATION SYSTEM AT IMT: VOLUME RATIO MEASUREMENTS

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Pressure Metrology Laboratory at the Institute of Metals and Technology has extended its measurement capabilities with a new vacuum calibration system. System is composed of four vacuum chambers having different volumes: (i)V_B -balast volume for gas preparation, (ii) V_0 – initial small volume, (iii) V_1 – big expansion volume, and (iv) V_2 – intermediate expansion volume. Initial gas pressure before expansion can be in the range from 100 Pa to 100 kPa and is measured by a pressure balance which is directly traceable to primary standards in PTB-Germany, or with a 100 kPa FS and a 1 kPa FS capacitance diaphragm gauges, which are internally calibrated by the pressure balance or static expansion system. The system enables primary generation of calibration pressures by static expansion method in a range from $2x10^{-3}$ Pa to 6.5 kPa.

To calculate calibration pressure after gas expansion, ratio of the volume of the chamber containing the gas before expansion and the volume of the chamber into which the gas is expanded has to be known with best possible accuracy. Volume ratio between different chambers and their combinations were determined using two methods: (i) NPL method of accumulation of gas by successive expansions and direct method using linear vacuum gauge (spinning rotor gauge). In the presentation details of the system will be given and results of preliminary measurements of volume ratios will be presented.

Acknowledgements: Primary static expansion calibration system has been partly financed by European Regional Development Fund (ERDF) through Metrology Institute of Republic of Slovenia (MIRS), Contract No. C2132-13-000032. Volume ratio measurements were also financially supported by MIRS through Contract No. C3212-10-000073.

COMPARISON OF TOF-SIMS SPECTROMETRY USING MeV IONS AND keV CLUSTERS

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Mass spectrometry of secondary molecular ions, resulting from ion irradiation of materials, is becoming an increasingly important tool for surface characterization of organic materials, biological systems, semiconductors, metals, ceramics, glass, paper etc. Advanced molecular imaging can be performed by secondary ion mass spectroscopy (SIMS), using primary ions or ion clusters in keV or MeV energy range. Usually time-of-flight (TOF) setup for detection of high-mass molecules is coupled with SIMS technique (TOF-SIMS) offering a possibility for a precise molecular imaging and mapping.

Primary ions of energy in the keV and MeV range have specific interaction mechanisms with materials surface under investigation and this should be explored to maximize a quality of obtained molecular images. Since SIMS-based molecular imaging is a new technique and only few such instruments are available in the world, many related phenomena are still not understood.

In this work we compare the results on mass spectrometry of some biological samples like amino acids (leucine and glycine) using keV and MeV by using new ToF SIMS spectrometers in Zagreb and Ljubljana. In particular we studied dependency on type of primary ions, energy dependence for secondary ion yield and damage cross-section for different biological samples. Experiments were performed at two advanced ToF-SIMS instruments installed in Croatia and Slovenia having very specific and complementary characteristics which can be explored even for better quality of applications. Ruđer Bošković Institute in Zagreb has newly installed ToF-SIMS spectrometer working with monomer ions in high energy range (5-10 MeV, Si, O) whereas at Jožef Stefan Institute in Ljubljana ToF -SIMS instrument is operating with ion clusters (Bi^{3+}, Bi^{5+}) at low energy range (20-30 keV).

Avtorsko kazalo – Index

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