



***26. Međunarodni Znanstveni Sastanak
Vakuumska Znanost i Tehnika***

Njivice, 16-17. Svibanj 2019

***26. Mednarodno Znanstveno Srečanje
Vakuumska Znanost in Tehnika***

Njivice, 16-17. Maj 2019

***26th International Scientific Meeting on
Vacuum Science and Technique***

Njivice, 16-17. May 2019

Book of abstracts

Editors

Maja Mičetić, Marko Karlušić

Hrvatsko vakuumsko društvo 2019

Croatian Vacuum Society 2019



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General information

The meeting will be held in Hotel Beli Kamik, Njivice, Island of Krk, Croatia, May 16 - 17, 2019. The main aim of the meeting is to bring together researchers, engineers, technicians and other experts to share their knowledge on the recent developments in the field of Vacuum science and technology and related areas.

International Scientific Meeting on Vacuum Science and Technique is traditionally organized each year by Vacuum Societies of Croatia and Slovenia. It provides the most recent scientific achievements across a broad range of vacuum related scientific disciplines.

Registration desk hours:

Thursday, May 17th,11:30-12:30

Topics:

- **Applied Surface Science**
- **Electronic Materials and Processing**
- **Vacuum Science and Technology**
- **Plasma Science and Technique**
- **Thin Films and Coatings**
- **Surface Science**
- **Biointerfaces**
- **Nanometer Structures**
- **Vacuum Metallurgy**
- **Nuclear Physics**



Program at a glance

Thursday May 16^h, 2019

Registration	11:30 – 12:30
Lunch	12:30 – 13:55
Opening	13:55 – 14:00
Invited lectures	14:00 – 15:20
Sponsor presentation	15:20 – 15:40
Break	15:40 – 16:10
Invited lectures	16:10 – 17:10
Poster session	17:10 – 18:30
Dinner	18:30 – 22:00

Friday May 17th, 2019

Breakfast & room check-out	8:00 – 9:00
Invited lectures	9:00 – 10:00
Sponsor presentation	10:00 – 10:20
Break	10:20 – 10:50
Invited lectures	10:50 – 12:10
Closing ceremony	12:10 – 12:20
Lunch	12:45 – 14:00
Departure	14:00



Programme schedule

Thursday		
11:30 – 12:30	Registration	
12:30 – 13:55	Lunch	
13:55 – 14:00	Opening	
14:00 – 14:20	Invited Lectures chair: Sebastian Dahle	Jordi Sancho Parramon METAL ISLAND FILMS BASED ON ALTERNATIVE PLASMONIC MATERIALS FOR SOLAR ENERGY CONVERSION
14:20 – 14:40		Petar Djinovič APPLICATION OF (UH)VACUUM CHARACTERIZATION TOOLS IN PHOTOCATALYTIC AND THERMOCATALYTIC REACTIONS
14:40 – 15:00		Sefer Avdiaj DETERMINATIONS OF VIRIAL COEFFICINETS USED FOR OPTICAL PRESSURE STANDARD FLOC
15:00 – 15:20		Mile Ivanda POROUS SILICON AND SILICON NANOWIRES FOR SENSING
15:20 – 15:40	Sponsor presentation	Kurt J. Lesker
15:40 – 16:10	Break	
16:10 – 16:30	Invited Lectures chair: Mile Ivanda	Sebastjan Dahle PLASMA BASED DEPOSITION OF SOLID FILMS FROM LIQUID PRECURSORS
16:30 – 16:50		Robert Peter STRUCTURAL PROPERTIES OF ZnO THIN FILMS SYNTHESIZED BY ATOMIC LAYER DEPOSITION
16:50 – 17:10		Lucija Krce STUDY OF BACTERICIDAL EFFECT OF LASER SYNTHETIZED SILVER NANOPARTICLES: A NOVEL MODEL FOR E. COLI GROWTH AND INACTIVATION
17:10 – 18:30	Poster session	
18:30 – 22:00	Dinner and social programe	



Programme schedule

Friday		
8:00 – 9:00	Breakfast	
9:00 – 9:20	Invited Lectures chair: Marjetka Conradi	Rok Zaplotnik FORMATION OF AMMONIA IN LARGE FUSION REACTORS AND IN SMALL LABORATORY PLASMA REACTORS
9:20 – 9:40		Aljaž Drnovšek HIGH TEMPERATURE HARDNESS AND FRACTURE TOUGHNESS OF CrAIN AND CrAISIN HARD COATINGS
9:40 – 10:00		Iva Božičević Mihalić CAPABILITIES OF X-RAY FLUORESCENCE BEAMLIN AT ELETTRA SINCROTRONE TRIESTE
10:00 – 10:20	Sponsor presentation	Scan
10:20 – 10:50	Break	
10:50 – 11:10	Invited Lectures chair: Iva Božičević Mihalić	Fevzi Kafexhiu COMPREHENSIVE CHARACTERIZATION OF THE AlCoCrFeNi _{2.1} HEA SYSTEM
11:10 – 11:30		Mario Rakić LASER RESONATORS FOR NEW LASER PROFILES AND OPTICAL LOGIC GATES
11:30 – 11:50		Marjetka Conradi SHORT- AND LONG-TERM WETTABILITY EVOLUTIOIN AND CORROSION RESISTANCE OF LASER-TEXTURED AND CHEMICALLY MODIFIED STEEL SURFACE
11:50:12:10		Arijana Filipić SAFER WATER WITH PLASMA
12:10 – 12:20	Closing ceremony	
12:20 – 14:00	Lunch	
14:00	Departure	



Abstracts

Invited Lectures

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OPTIKA
M I C R O S C O P E S
I T A L Y



I-1 METAL ISLAND FILMS BASED ON ALTERNATIVE PLASMONIC MATERIALS FOR SOLAR ENERGY CONVERSION

Jordi Sancho-Parramon, Ivana Fabijanić, Stefano A. Mezzasalma, Vesna Blažek Bregović, Matej Bubaš, Boris Okorn and Vesna Janicki

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Alternative materials that can replace gold and silver in plasmonics have been actively investigated over the last decade, including nitrides, oxides, silicides and 2D materials, among others. Copper and aluminum have been usually overlooked as plasmonic materials, as they have trend to oxidize. However, the optical properties of both Cu and Al can provide a comparable plasmonic response to that of Au and Ag for certain type of nanostructures. The plasmonic performance of Cu and Al is investigated using numerical simulations of different nanostructures (spheres, cubes, rods, and coupled particles) and taking into account the presence of oxidization. It is shown that geometry can play a dominant role over material properties and the performance of Cu and Al becomes comparable to that of Ag and Au for systems of non-spherical particles and strong electromagnetic coupling among particles. This observation is experimentally confirmed by the fabrication and characterization of Cu and Al metal island films using electron beam evaporation. Optical characterization of the samples evidences a similar performance to that obtained for Ag and Au and suggests that Cu and Al metal island films can offer an efficient low-cost platform for solar energy harvesting.

I-2 APPLICATION OF (UH)VACUUM CHARACTERIZATION TOOLS IN PHOTOCATALYTIC AND THERMOCATALYTIC REACTIONS

Petar Djinović

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Chemical transformation of industrially relevant molecules is largely dependent on the use of catalysts. These materials decrease the activation energy for transition states, thus accelerating the reaction rate and enabling the reaction to proceed at milder conditions. This is often related to high selectivity for desired products. Tailored design of nanomaterials is recently often employed to enhance their morphology-specific chemical and physical properties, which govern the catalytic activity.

In this lecture I will focus on the use of Transmission Electron Microscopy and (Near Ambient Pressure) XPS, both high vacuum techniques, in characterization of structurally defined nanomaterials for different (photo)catalytic applications.

In the first example, the TiO₂ nanorod/reduced graphene oxide (rGO) catalyst will be discussed in terms of morphological analysis and nature of rGO-TiO₂ bonding and their consequences on photocatalytic total oxidation of bisphenol A aqueous solutions.[1] This represents a model photocatalytic reaction for wastewater purification.

In the second example, catalytic decomposition of N_2O (strong greenhouse gas) to N_2 and O_2 over copper oxide supported on CeO_2 will be discussed.

Catalytic behavior of CuO dispersed over CeO_2 nanocubes, nanorods and polyhedral crystallites was analyzed. It was discovered that CuO/CeO_2 solids should enable high oxygen mobility as well as formation of highly reducible Cu defect sites, in order to ensure high intrinsic activity. It was further revealed that CeO_2 morphology needs to be tailored to expose $\{100\}$ and $\{110\}$ high-energy surface planes, as present in CeO_2 nanorods. Oxygen mobility and regeneration of active Cu phase on these surface planes is easier, which in turn facilitates higher catalytic activity through the recombination of surface oxygen atoms and desorption as molecular oxygen that replenishes active sites for subsequent catalytic cycles.[2]

Syngas (H_2 and CO) is the backbone of modern petrochemical industry and new catalytic pathways for conversion of methane and CO_2 are intensively investigated. In the third example, I will highlight the morphology and chemistry of nickel and cobalt nanoclusters dispersed over CeO_2 and their role during the reforming reaction.[3] The appropriate design of active sites responsible for methane and CO_2 activation (bimetallic clusters below 45 nm, adjacent to oxygen vacancy sites of the $CeZrO_2$ solid solution) enabled kinetic balancing of both reaction halves, producing catalysts that are highly resistant to carbon accumulation in a wide range of CH_4-CO_2 feed compositions. With the employed approach, carbon accumulation can be prevented over metal clusters that are 2-fold larger compared to state-of-the-art. Furthermore, the active state of the catalyst was analyzed with NAP-XPS technique.

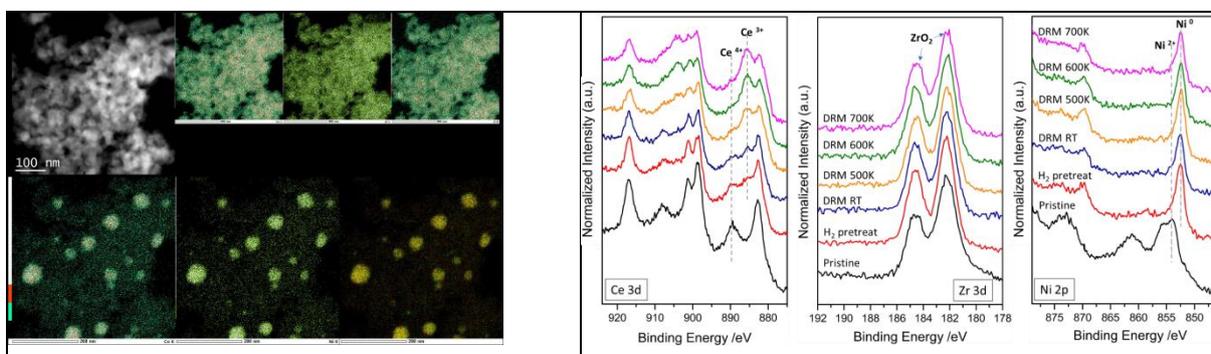


Figure 1. HAADF/STEM micrograph of the 3% NiCo/ $CeZrO_2$ catalyst after 400 h TOS (top left) with corresponding EDXS elemental mapping. Upper row from left to right: Ce L edge, Zr L edge and O K edge. Lower row from left to right: Co K edge, Ni K edge and their overlay. Ideal matching of the Ni and Co maps strongly suggests presence of alloyed NiCo bimetallic clusters. Right handside: NAP-XPS profiles in the Ce 3d, Zr 3d and Ni 2p regions of 4 wt % Ni/ $CeZrO_2$ catalyst before and after H_2 pretreatment and during the methane- CO_2 reforming reaction at different temperatures.

[1] G. Žerjav, M.S. Arshad, P. Djinović, I. Junkar, J. Kovač, J. Zavašnik and A. Pintar: *Nanoscale*, 2017,9, 4578.

[2] M. Zabilskiy, P. Djinović, E. Tchernychova, O.P. Tkachenko, L.M. Kustov and A. Pintar: *ACS Catal.*, 2015, 5, 5357.

[3] P. Djinović and A. Pintar, *Applied Catalysis B*, 2017, 206, 675.



I-3 DETERMINATIONS OF VIRIAL COEFFICIENTS USED FOR OPTICAL PRESSURE STANDARD FLOC

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² *National Institute of Standards and Technology (NIST), Sensor Science Division, 100 Bureau Drive, Gaithersburg 20899, Maryland, USA*

To measure the refractive index of gases with very high resolution we can use an optical resonator like Fabry-Perot interferometer. The National Institute of Standards and Technology is developing a new low-pressure sensor which is based on the measurements of (nitrogen) gas refractivity inside a Fabry-Perot cavity, so this allows a new realization of Pascal. In order to determine the pressure a laser is locked to the optical cavity while the laser frequency is interrogated. The frequency will change according to the pressure (density) of the gas which in our case was nitrogen [1]. For gases with simple electron structure (with mostly two electrons) such as helium the refractivity and density virial coefficients can be calculated through quantum mechanics [2]. For other gases, the value of density virial coefficients must be measured. In this research work, in order to determine the gas density virial coefficients, pressure determinations from the Fixed Length Optical Cavity – FLOC were compared with those of a piston gauge for four different temperatures (30°C to 45°C) in the pressure range from 50 kPa to 150 kPa.

[1] P. F. Egan, J. A. Stone, J. H. Hendricks, J. E. Ricker, G. E. Scace, and G. F. Strouse, *Opt. Lett.* 40, 3945 (2015)

[2] M. Puchalski, K. Piszczatowski, J. Komasa, B. Jeziorski, K. Szalewicz, 2016, "Theoretical determination of the polarizability dispersion and the refractive index of helium," *Amer. Phys. Soc., Phys. Rev. A* 93, 032515 (2016)

I-4 POROUS SILICON AND SILICON NANOWIRES FOR SENSING

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The presented research addresses basic properties and application of silicon/hybrid nanostructures for surface enhanced Raman spectroscopy (SERS) and gas sensing. Porous silicon (PSi) is a semiconducting material fabricated by electrochemical etching in hydrofluoric acid (HF) that has high surface-to-volume ratios and is, therefore, interesting materials for sensing devices and for use in SERS. Here we present the SERS substrates produced by different synthesis methods. Silver nanoparticles on different porous silicon nanostructures have been prepared by the immersion plating in AgNO₃. This procedure enable preparation of SERS substrates based on p-type pSi and for ultra-low concentration detection of R6G with dilution up to 10⁻¹⁵ M.

Another approach is to develop SERS optical fiber probe by growing horizontal silicon nanowires on the planar substrates and optical fiber by vapour-liquid-solid growth by using LPCVD technique. The Ag decorated nanowires shown on Fig. 1 are proved to be sensitive substrates for SERS only if the silicon nanowires thickness, length, volume density as well as metal nanoparticle size and distribution are carefully designed. Finally, as a third example, we will present the sensitivity of oxidized nanoporous silicon in detection of a number of different molecules by resistivity measurements.

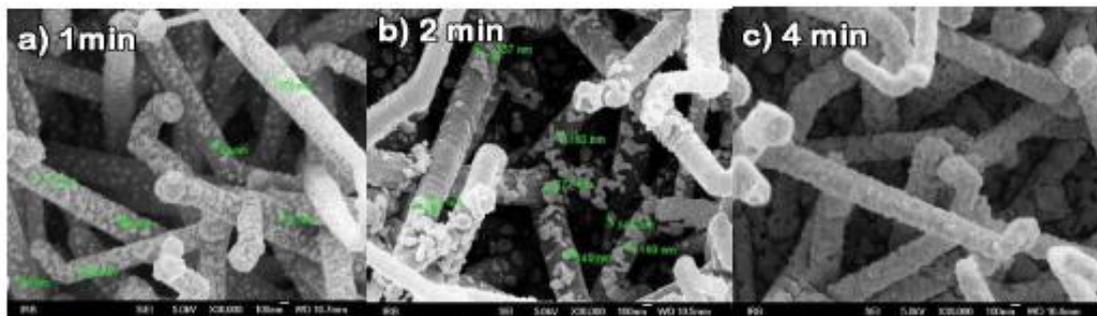


Figure 1. SiNWs on optical fiber obtained by VLS method decorated with AgNPs during various sputtering times: 1, 2, and 4 min.

I-5 Plasma-based deposition of solid films from liquid precursors

Sebastian Dahle

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The deposition of various coatings by means of non-thermal plasma discharges is well known via plasma-enhanced chemical vapour deposition. Based on observations for plasma deposition both, in vacuum and under atmospheric pressure, we developed novel approaches employing dielectric barrier discharges to form solid films. This plasma-enhanced chemical solution deposition offers new perspectives and possibilities for a multitude of applications. In a combination of plasma-chemical and conventional wet-chemical reaction mechanisms, it is well possible to include advantages of conventional coatings and plasma polymers into a single process. For particular applications, issues regarding the precursor supply are avoided entirely, whereas it becomes possible to include sensitive additives and large particles, such as fillers or pigments of diameters up to and well beyond 100 μm , within the precursor phase. While efforts for the implementation of such a technique can be minimal, the process offers the development of property gradients through the deposited films as a systemic benefit. Several example applications will be discussed to further highlight advantages and challenges included with the novel approach.

I-6 Structural properties of ZnO thin films synthesized by atomic layer deposition

Robert Peter^{1,2}, Krešimir Salamon³, Iva Šarić^{1,2}, Ivana Jelovica Badovinac^{1,2}, Aleš Omerzu^{1,2}, Joerg Grenzer⁴, Mladen Petavić¹

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Structural characteristics of ZnO films grown by atomic layer deposition (ALD), on (001) oriented sapphire (c-Al₂O₃) and amorphous SiO₂ substrates, were investigated for a wide range of deposition temperatures (60-240 °C). The samples grown inside the ALD window (140-180 °C) exhibit excellent stoichiometry, high optical transparency in the visible region and the elemental in-depth uniformity. On the other hand, crystallinity of the ZnO is strongly dependent on the substrate, with the sapphire giving the highly (001) and (101)-oriented polycrystalline films, while no preferential texture was observed for the SiO₂ substrate. In addition, the in-plane epitaxial relationship between the deposited films and the substrate for ZnO/c-Al₂O₃ samples is observed. A simple growth model, based on the preferential grain nucleation and temperature depended intragrain/intergrain diffusion, is proposed for the observed texture in the ZnO polycrystalline films.



I-7 Study of bactericidal effect of laser synthesized silver nanoparticles: A novel model for *E. coli* growth and inactivation

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² *University of Split, Faculty of Science, Department of Chemistry, Ruđera Boškovića 33, 21000 Split, Croatia*

³ *University of Split, Faculty of Science, Department of Biology, Ruđera Boškovića 33, 21000 Split, Croatia*

⁴ *Institute of Physics, Bijenička cesta 46, 10000 Zagreb, Croatia*

Study of bactericidal effect of silver nanoparticles (AgNPs), free of chemical byproducts, could be of great importance in understanding the underlying antibacterial mechanism(s). In this work, we present the production and characterization of AgNPs, synthesized by laser ablation of silver in water, and optical density (OD) growth curves of *E. coli* cells treated at sub MIC (minimal inhibitory concentration) and MIC concentrations. Besides the increase of the baseline and reduction of the maximal OD, the main impact of the treatment is the increase of the lag time, which becomes infinitive for the MIC treatment. The obtained bacterial growth curves are described by a novel growth and inactivation model given by the set of three differential equations. Our model fits the OD data very well and explains the apparent lag phase, which we found to be a dynamic state in which the bacterial growth and death rates are close in value.

I-8 FORMATION OF AMMONIA IN LARGE FUSION REACTORS AND IN SMALL LABORATORY PLASMA REACTORS

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In the world's largest fusion experiment ITER the seeding of gaseous impurities will be mandatory in order to protect the tungsten divertor from local heat loads [1]. Nitrogen seems to be the best candidate for radiative cooling. However, nitrogen seeding leads to ammonia production, which could be a potential issue for ITER, while in the D-T phase tritiated ammonia would be produced.

In order to evaluate the addition of the tritiated ammonia to the ITER tritium inventory many ammonia production experiments are being performed in fusion tokamaks, such as JET and ASDEX-U [2]. 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.



We participate in ASDEX-U experiments as spectroscopy analysis experts for the ammonia discharges and RGA experts. We analyzed measurements from nitrogen seeded plasma shots and concluded that ammonia is produced in the plasma shaded areas even in subsequent non-seeded shots [2]. We also studied ammonia production on different materials in a controlled environment in our own dual atom beam laboratory experiment.

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)

1. Neuwirth, D.; Rohde, V.; Schwarz-Selinger, T.; Team, A.U. Formation of ammonia during nitrogen-seeded discharges at ASDEX Upgrade. *Plasma Physics and Controlled Fusion* 2012, 54, doi:10.1088/0741-3335/54/8/085008.
2. Drenik, A.; Laguardia, L.; McDermott, R.; Meisl, G.; Neu, R.; Oberkofler, M.; Pawelec, E.; Pitts, R.A.; Putterich, T.; Reichbauer, T., et al. Evolution of nitrogen concentration and ammonia production in N-2-seeded H-mode discharges at ASDEX Upgrade. *Nuclear Fusion* 2019, 59, doi:10.1088/1741-4326/aafe23.

I-9 High Temperature Hardness and Fracture Toughness of CrAlN and CrAlSiN hard coatings

Aljaž Drnovšek

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Magnetron sputter deposited coatings are frequently used in industrial applications where due to harsh working environment the temperatures can exceed 1000°C. Although PVD coatings have shown to be successful in such applications not much is known how basic mechanical properties of thin coatings change due to high temperature. In recent years the development of new ex-situ and in-situ nanoindentation systems have made the measurement of high temperature mechanical properties possible.

In the current work we tested two widely used PVD coatings for high temperature applications, namely CrAlN and CrAlSiN coating. Although the two coatings consist of similar elements they differ in microstructure due to addition of Si which leads to better room temperature mechanical properties. Both coatings were tested for their hardness and elastic modulus from room temperature to 700 °C in 100 °C steps. Constant hardness drops of 2 GPa/100 °C were measured for CrAlN coatings over the whole temperature range while only minor changes at temperatures above 500°C were measured for CrAlSiN coating.

Fracture toughness measurements were conducted in a similar manner, but measured in-situ by micro-cantilever deflection. The fracture toughness decreases with the increased temperature, interestingly, the observed decreasing trends in hardness and fracture toughness are similar.

These are the first successfully obtained results for coating high temperature fracture toughness and therefore present a first step towards more comprehensive understanding of high temperature mechanical behaviour of hard coatings.



I-10 Capabilities of X-Ray Fluorescence Beamline at Elettra Sincrotrone Trieste

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X-Ray Fluorescence beamline [1] is developed by Elettra Sincrotrone Trieste in partnership with the International Atomic Energy Agency. End-station consists of ultra-high vacuum chamber housing an advanced 7-axis motorized sample manipulator stage. It operates in the energy range from 3.5 to 14 keV. Combining the tunable monochromatic X-Ray beam with the manipulator that enables different excitation and detection geometries, XRF beamline enables wide range of analytical techniques in one single facility [2]. XRF elemental characterization is possible in conventional reflection geometry (45°/45°) resulting in elemental maps with spatial resolution of 200 μm x 100 μm or in angle-dependent geometry via grazing incidence/exit XRF (GI/GE XRF) for surface analysis and depth profiling to total reflection XRF (TXRF) mode for detection of ultra-trace concentration levels in liquid samples or particulate matter. Tunable source with the high resolving power enables X-Ray Absorption Near-Edge Structure (XANES) experiments for chemical speciation studies of specific elements in 45°/45° geometry or in grazing incidence geometry for speciation of trace elements. Simultaneous GI-XRF and X-Ray Reflectometry (XRR) measurements are employed for structural analysis of thin films and layered structures.

Presentation will give an overview of analytical capabilities of XRF end-station, indicating advantages and disadvantages of available techniques which cover wide range of research fields from material science, biomedicine, environmental, biology, cultural heritage to X-Ray fundamental studies.

[1] W. Jark, D. Eichert, L. Luehl, A. Gambitta, *Optimisation of a compact optical system for the beamtransport at the x-ray fluorescence beamline at Elettra for experiments with small spots*, Proc. SPIE, 9207 (2014), 1-12

[2] A. G. Karydas, M. Czyzycki, J.J. Leani, A. Migliori, J. Osan, M. Bogovac, P. Wrobel, N. Vakula, R. Padilla-Alvarez, R.H. Menk, M.G. Gol, M. Antonelli, M. K. Tiwari, C. Caliri, K. Vogel-Mikuš, I. Darby, R. B. Kaiser, *An IAEA multi-technique X-ray spectrometry endstation at Elettra Sincrotrone Trieste: benchmarking results and interdisciplinary applications*, J. Synchrotron Radiat. 25 (2018), 189-203



I-11 COMPREHENSIVE CHARACTERIZATION OF THE AlCoCrFeNi_{2.1} HEA SYSTEM

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A simultaneous achievement of both high strength and high ductility constitutes a tough challenge in high-entropy alloys (HEA), as in material science in general. Problems with poor castability and compositional segregation of HEAs create an additional limitation for industrial applications. The purpose of the present study is to shed some light on the possibilities of adaptation of the AlCoCrFeNi_{2.1} HEA into large-scale industrial production. Primarily, a machine learning approach using Python and Thermo-Calc simulations were applied for the chosen HEA system. Further, vacuum induction melting and ingot casting were used to obtain the AlCoCrFeNi_{2.1} – eutectic alloy with a microstructure composed of alternating soft FCC and hard BCC phases. A comprehensive characterization is being carried out on the as-cast- and heat-treated conditions, including the XRF analysis of the longitudinal section, microstructure characterization (OM, SEM/EDS/EBSD, XRD, TEM), thermal analysis, dilatometry, hardness tests, tensile tests, impact toughness tests, tribological tests, and creep tests.

I-12 LASER RESONATORS FOR NEW LASER PROFILES AND OPTICAL LOGIC GATES

Mario Rakić^{1,2}, Austin W. Steinforth², Andrey Mironov², Gary J. Eden²

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Generation of new laser profiles which are fully defined inside the resonator and require no additional optical shaping presents a challenging task but promise a viable route for use in particle confinement, laser tweezers, magneto-optical and gravito-optical traps, optoelectronics and in communication technology. Here we will show how both linear and cylindrical laser profiles have been realized and emphasizes their application. We used a laser resonator consisting of two mirrors, gain medium CdSe nanoparticle in solution, and an additional optical component placed within the resonator that is ultimately responsible for the shape of the output laser profile. This optical component proved to be the most challenging part of the setup since it had to be almost completely transparent in order to achieve a laser effect. For pump beam we used a 532 nm nanosecond laser and got the output laser emission at 655 nm. We additionally used similar setup to achieve a NOT logical gate which, due to the use of the nanoparticles in solution and not the standard semiconductor elements, present a unique realization of completely optical logic gate. We will show that besides the relatively trivial realization of AND and OR logic gates this setup could also be used to create XOR gates which then allows for realization of a complete optical logical algebra for possible application in information processing.



I-13 SHORT- AND LONG-TERM WETTABILITY EVOLUTION AND CORROSION RESISTANCE OF LASER-TEXTURED AND CHEMICALLY MODIFIED STEEL SURFACE

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We present the results of a 10 month observation of wetting and corrosion properties of nanosecond fiber-laser-textured stainless steel, uncoated and coated with epoxy or FAS-TiO₂/epoxy coating. We performed a comparative study on samples kept under ambient conditions and in conditions with reduced air pressure and humidity. We were able to achieve surface superhydrophobicity either indirectly by aging the laser-textured surface or directly by application of FAS-TiO₂/epoxy coating. The wettability conversion from superhydrophilic to superhydrophobic state of uncoated laser textured surfaces was confirmed with XPS measurements indicating surface contamination with nonpolar elementary carbon. Environmental conditions significantly influenced wettability of uncoated laser-textured steel whereas aging of coated surfaces was not affected. Barrier properties of uncoated and coated laser-textured surfaces are enhanced on short- and long-term basis.

I-14 SAFER WATER WITH PLASMA

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Plasma technologies are being used more and more in various biological fields such as food industry, agriculture and medicine. One of its most widespread purposes in these fields is decontamination, which is mostly focused on the decontamination of surfaces. However, since water scarcity has become one of the biggest global issues, the focus of plasma as a decontamination tool has shifted towards waters as well. Besides urbanisation, industrialisation and climate change, an increasing number of water pollutants caused a rapid decrease of clean freshwater. For decades, viruses were neglected as potential water pollutants but an emerging number of diseases connected with water changed that.

Water transmissible viruses cause millions of infections worldwide, of not just humans but animals and plants as well, causing a great ecological and economic burden. That is why it is extremely



important to inactivate viruses in water. Methods, such as chlorination, used for water decontamination can be environmentally non-friendly, can produce toxic by-products and, in most cases, are not effective against viruses. Plasma on the other hand, presents an easy and efficient solution for water decontamination without producing waste or toxic intermediates.

In our work, we wanted to test if viruses in water can be inactivated by cold atmospheric plasma (CAP). For that we used a CAP system in the single electrode configuration connected to a low-frequency generator (31 kHz) that operated at a peak-to-peak voltage of 6 kV, with total average output power of ~ 3 W.

We chose two morphologically different viruses as a model organism: potato virus Y (PVY) and bacteriophage MS2, both of which can be transmitted by water. PVY is a filamentous virus that can infect different plants but is the most important potato virus pathogen. MS2 is an icosahedral virus, used as a surrogate for enteric viruses transmitted by water. Compared to PVY, its genome is almost three times shorter.

We treated 10 ml of water samples that contained viruses with CAP and verified success and a mode of virus inactivation with different methods. Initial virus concentrations were determined with reverse-transcription droplet digital polymerase chain reaction (RT-ddPCR). Integrity of viral RNA was examined with RT-PCR, viral infectivity was tested with inoculation of test plants, followed by reverse-transcription real-time PCR or with double layer plaque assay. In some cases, the integrity of virus particles was examined with transmission electron microscopy. Optical emission spectroscopy (OES) was used to observe the light emitted by the plasma during the treatments.

We were able to inactivate or sufficiently decrease high concentrations of two different viruses after very short treatment times including only 1 minute. Our results indicate that CAP could be used for viral inactivation in waters and as such it could serve as a powerful tool of which we are in great need of today, to clean our waters and make them safe for everyone.



Abstracts

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I T A L Y



P-1 Deep defects introduced in n-type 4H-SiC by ion-implantation

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We present a study of deep level defects introduced in n-type 4H-SiC epitaxial layers by ion implantation with He, C and O ions.

Silicon carbide is expected to be used as a semiconductor in electronic devices for operation in a harsh ionizing radiation environment because of its high radiation resistance properties. However, it is still crucial to understand the effects of accumulated radiation damage to the electronic properties of these devices. Radiation induced defects can limit the carrier lifetime which diminishes the performance of semiconductor devices. It is important to characterize these deep level defects and control their concentration by material engineering.

Nitrogen-doped ($4.5 \times 10^{14} \text{ cm}^{-3}$) n-type 4H-SiC epitaxial layers were grown by chemical vapor deposition, approximately 25 μm and 50 μm thick. Schottky barrier diodes (SBD) were fabricated by evaporation of nickel through a 1mm x 1mm metal mask on the epitaxial layer. Ohmic contacts were formed by nickel sintering at 950 °C in Ar atmosphere.

The SBDs were implanted in the single-ion regime with 2MeV He, 7.5MeV C or 20MeV O ions at the ANSTO nuclear microprobe facility. The lattice atom displacement density distribution dependence on ion beam energy was predicted using SRIM software. The depth profiles of defects were used to choose energies for each ion species so that the lattice atom displacement density occurs within the depletion region of reversely biased SBDs examined by the DLTS. The SBDs were implanted by ions with various fluences. He ions were implanted with fluences ranging from $5 \times 10^9 \text{ cm}^{-2}$ to $1 \times 10^{10} \text{ cm}^{-2}$, C ions with fluences from $1 \times 10^8 \text{ cm}^{-2}$ to $1 \times 10^9 \text{ cm}^{-2}$ and O ions with fluences of $2 \times 10^9 \text{ cm}^{-2}$ and $1 \times 10^{10} \text{ cm}^{-2}$.

The deep level defects effect on electronic properties of 4H-SiC SBDs were characterized by I-V, C-V, and Deep Level Transient Spectroscopy. The I-V characterization showed that SBDs have a very low leakage current (less than 10pA). Diode series resistance did not increase significantly with the fluence which confirms high radiation hardness of SiC. The C-V measurements showed a net free-carrier compensation, corresponding to damaged regions at depths which agree with SRIM predictions. By using DLTS spectroscopy we have revealed several deep level defects, identified as EH1 and Z1/2 (with a high temperature shoulder identified as EH3). The activation energies and capture cross-sections were determined from the Arrhenius plot.

[1] Pastuović Ž, Siegele R, Capan I, Brodar T (2017) Deep level defects in 4H-SiC introduced by ion implantation : the role of single ion regime



P-2 SELF-ORGANIZED GROWTH OF Ge NANOWIRE MESHES IN Al₂O₃ MATRIX

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We investigate structure of three-dimensional Ge nanowire mesh embedded in an amorphous alumina matrix. A wide range of such nanowire meshes was deposited using magnetron sputtering, by virtue of self-organized growth regime. Structure properties, including the length and width of the nanowires as well as the arrangement of the nanowires in the matrix, were investigated by transmission electron microscopy - TEM and grazing incidence small angle X-ray scattering - GISAXS. A strong dependence of the nanowire structural parameters on deposition conditions, including deposition temperature and working gas pressure, was observed. Different mesh properties and levels of self-ordering were observed.

P-3 Nanostructured titanium-based materials for biomedical applications

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In-stent restenosis and thrombosis are still the two major causes of stent failure. Bare metal stents (BMS) as well as drug-eluting stents (DES), which are also often coated with polymers that have thrombogenic potential, can induce platelet adhesion, activation and thrombus formation. Various technologies have been employed in order to design polymer-free stents, such as microporous stents and drug-loaded inorganic coatings, such would prevent platelet coagulation (thrombosis), uncontrolled proliferation of smooth muscle cells (restenosis) and allow for normal endothelium growth. In present study, nanostructures on the surface of Ti-based materials have been synthesized with electrochemical anodization or hydrothermal method and further treated with oxygen plasma. The interactions with whole blood (platelets), human coronary artery endothelial cells (HCAEC) and human coronary artery smooth muscle cells (HCASMC) have been studied. Surfaces covered with



nanostructures and additionally treated with plasma allow normal proliferation of HCAEC, reduced platelet adhesion and activation and reduced proliferation of HCASMC.

P-4 Laser plasmas produced in various environments studied by optical emission spectroscopy

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Pulsed laser ablation is the process which occurs when laser pulse hits the solid target and ablates material from target surface, due to various energy transfer processes as photodissociation, target heating, electron-phonon coupling and Coulomb repulsion between positive ions remained after electron ejection. The latter results in explosion (ablation) of target material which is ejected in the form of the hot plasma while simultaneously crater is formed at the target surface. If ablation is performed in liquid, liquid pressure ensures that plasma is confined in small space and exists in dynamic state of plasma plume. This process is called Pulsed laser ablation in liquid and presents "green technique" for nanoparticle production.

Optical emission spectroscopy (OES) is technique that can provide us information about dynamics and related processes in laser produced plasmas. From Planck's distribution fit it's possible to determine the temperature of target during laser ablation. From line spectra and Boltzmann fit one can determine temperatures of ablated species in plasma plume.

In this paper, OES is made on laser produced hot plasmas at three different environments above target - vacuum, air and water. Laser ablation is performed with Nd:YAG laser with fundamental wavelength 1064 nm. Laser operates at repetition frequency of 5Hz and pulse duration is 4ns. Analysed ablated targets are Au, Ag, Cu, ZnO and TiO₂. Variations of pulse energies (300 mJ, 200 mJ, 100 mJ) and pulse wavelengths (1064 nm, 532 nm, 266 nm) are used for additional comparison.



P-5 Deep defects created in neutron-irradiated n-type 4H-SiC

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We present a study of electrically active deep defects introduced in n-type epitaxial 4H-SiC wide band gap semiconductor by epithermal and fast neutron irradiation. Characterization of defects in 4H-SiC is crucial for the future improvement of radiation hardness, efficiency and extending the lifetime of 4H-SiC detectors by material engineering.

Schottky barrier diodes (SBD) were produced on nitrogen-doped ($4.5 \times 10^{14} \text{ cm}^{-3}$) n-type 4H-SiC epitaxial layer grown by chemical vapor deposition, approximately 25 μm thick. The Schottky barrier was formed by evaporation of nickel through a metal mask with patterned square apertures of 1 mm \times 1 mm, while Ohmic contacts were formed on the backside of the SiC substrate by nickel sintering at 950 °C in Ar atmosphere.

Irradiation of SBDs by epithermal and thermal neutron irradiation was carried out at the Josef Stefan Institute TRIGA reactor in Ljubljana, Slovenia. The SBDs were irradiated with fluencies spanning several orders of magnitude, in the range from 10^9 n/cm^2 up to 10^{14} n/cm^2 . Only intrinsic defects are introduced in 4H-SiC by the irradiation.

Electrical characterization was carried out by current-voltage, capacitance-voltage, deep level transient spectroscopy (DLTS) and Laplace DLTS measurements. The DLTS measurements were carried out in the temperature range up to 380 K.

Only the well-known $Z_{1/2}$ peak at about 300 K was observed in the DLTS spectra of the non-irradiated sample. The Laplace DLTS measurements were used to resolve the $Z_{1/2}$ deep level into $Z_1 (=0)$ and $Z_2 (=0)$ deep levels assigned to carbon vacancy (V_C) on the hexagonal $V_C(h)$ and the cubic $V_C(k)$ lattice site, respectively. The used Laplace DLTS technique offers an order of magnitude better energy resolution compared to conventional DLTS.

The higher energy resolution enabled us to determine that the introduction rate for $Z_2 (=0)$ is approximately twice the $Z_1 (=0)$ introduction rate, while their concentration ratio $Z_2 (=0):Z_1 (=0)$ is $\approx 4:1$ in the non-irradiated sample. As a result of the introduction of acceptor deep levels, the free carrier concentration decreased linearly as a function of neutron fluence with the removal rate of 6.8 cm^{-1} .

The two additional deep levels EH1 and EH3 with energies around $E_C - 0.4 \text{ eV}$ and $E_C - 0.7 \text{ eV}$ are introduced in the DLTS spectra by the irradiation at the neutron fluences higher than 10^{12} n/cm^2 . The EH1 and EH3 deep levels are tentatively assigned to carbon displacements, most probably to Ci-related defects or simple defect complexes.



[1] Brodar T, Capan I, Radulović V, Snoj L, Pastuović Ž, Coutinho J, Ohshima T (2018) Laplace DLTS study of deep defects created in neutron-irradiated n-type 4H-SiC. Nucl Instruments Methods Phys Res Sect B Beam Interact with Mater Atoms 437:27–31

[2] N.T. Son, X.T. Trinh, L.S. Løvlie, B.G. Svensson, K. Kawahara, J. Suda, T. Kimoto, T. Umeda, J. Isoya, T. Makino, T. Ohshima, E. Janzén, Negative-U system of carbon vacancy in 4H-SiC, Phys. Rev. Lett. 109 (2012).

P-6 PROPERTIES OF Ag-(W, Mo) THIN FILMS

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Some properties of the Ag-(W, Mo) binary thin films which are important for the usability of the alloys were examined. Samples were prepared by magnetron codeposition technique of pure metals in wide range of composition onto various substrates held at room temperature. The structure of films were examined by the XRD and SEM methods. To verify the stability of the samples in a prolonged time scale, SEM analysis was performed on new samples and samples eight years aged. Significant degradation of samples was found. The mechanical properties of Ag-(Mo, W) alloys on sapphire substrates have been examined by nanohardness measurement (load 2mN and 5mN) in a whole range of composition. It was found that nanohardness generally increases with increase of harder component (W, Mo). The film thickness and the stress were measured by the profilometer. The elastic energy fraction of the deformation was correlated with the stress in films.

P-7 MAKING NANOWIRES OF METAL OXIDES AT LOW-PRESSURE

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In the recent direction of vacuum or reduced-pressure research, plasmas generated at low-pressure conditions are being more and more used for synthesis or tailoring of nanomaterials. Low-temperature plasmas have already proven to be a great source for the surface manipulations or supplying building blocks for nanomaterials and their growth. Similarly, such growth is also obtained at heated gas or furnace low-pressure conditions. However, the specific plasma-surface interactions are leading to synergistic effects, where very little is understood regarding basic processes taking place. These reactions simply fuel the growth of nanostructures. To understand these processes at the atomic scale



and mechanisms taking place, we implemented different low-pressure plasma treatments of nanoscale materials. As results of interactions of various plasma species including ions, electrons or neutral atoms, the intrinsic properties of nanomaterials changed. Furthermore, different nanostructures on surfaces were grown. The typical case of such 1D nanomaterial growth are nanowires of metal oxides. These observations are supported by analytical methods in order to unravel what is occurring on the nanomaterial surface or bulk. Moreover, the nanowires were not only grown in different plasmas, where the role of plasma species was studied but also compared to the thermal process of nanowire synthesis. The exhibited growth of nanowires grown at high-temperatures is proven to be much different from nanowires grown in plasmas, whereas not only speed of synthesis but also nanowire properties are changed. All this is explained, by plasma species created within low-pressure plasmas, neutral gas molecule interactions and surface heating effects.

P-8 XPS analysis of 2-mercapto-metylimidazole on copper

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This work presents the XPS analysis of 2-mercapto-metylimidazole that was adsorbed on the copper surface from the chloride solution. A special focus is devoted to the organics-metal manner of connection. High-resolution Cu 2p, N 1s, O 1s, C 1s, and S 2p XPS spectra and Auger Cu LMM spectrum were measured and analysed. Moreover, Tougaard analysis was performed in order to determine the thickness of the thin surface layer [1].

[1] Matjaž Finšgar, Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 190 (2018) 290–297.



P-9 Grain size effect on photocatalytic activity of TiO₂ thin films grown by atomic layer deposition (ALD)

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TiO₂ is a large band gap semiconductor which is chemically and biologically inert and has a good photostability. Thin TiO₂ films are involved in a wide range of applications, from the production of the antireflective coatings, to self-cleaning and photocatalytic layers. In the field of photocatalysis TiO₂ has been intensively studied for the degradation of organic substances in aqueous solutions under the UV illumination. In this work we have studied a correlation between the grain size and the photocatalytic activity of TiO₂ thin films grown by atomic layer deposition (ALD). Since ALD provides excellent uniformity, accurate thickness control, easy control of composition of the films at the low deposition temperature, it is ideal technique for the deposition of thin TiO₂ films on various substrates.

Several TiO₂ samples of the anatase phase with grain sizes in the range from 55 nm to 830 nm and one sample of the rutile phase were grown on Si (100) substrates at different ALD synthesis conditions. In order to exclude the effect of film thickness on the surface morphology and crystallinity, TiO₂ films of approximately the same thickness (100 nm) were used in this study. The surface morphology, structure and thickness were analyzed using scanning electron microscopy (SEM), grazing incidence X-ray diffraction (GIXRD), secondary ion mass spectrometry (SIMS) and X-ray photoelectron spectroscopy (XPS). The photocatalytic activity of as-prepared films was determined by measuring the degradation of methylene blue (MB) under the UV light irradiation at room temperature as a function of the exposure time. Anatase phase TiO₂ films show a high degradation rate of MB under the UV light, with the maximum effect for grains of 200 nm. Therefore, in the present work we have demonstrated the possibility of maximizing photocatalytic activity of TiO₂ thin films by controlling the grain size of polycrystalline films with ALD.

This work was supported by the Croatian Science Foundation under project IP-2016-06-3568. The characterization instruments used in this work were acquired through the European Fund for Regional Development and the Ministry of Science, Education and Sports of the Republic of Croatia under the project Research Infrastructure for Campus based Laboratories at the University of Rijeka (grant number RC.2.2.06-0001).



P-10 Plasma treated CNT gas sensors tested on ethanol

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Ethanol (C₂H₅OH) is a volatile, flammable, colourless organic solvent that people are often exposed to. It can cause addiction and do significant harm to one's body because of which it is necessary to have a means for detection. Carbon nanotube sensors are not a novelty in ethanol sensing; however, we tried to improve their responsivity by using functionalised multi-walled CNTs post-treated with He or Ar atmospheric pressure plasma. Plasma was supposed to change the surface of the sensor by making it more conductive and eventually more stable.

We prepared the Bucky paper from MWCNT functionalized with 8% carboxyl acid (COOH). It was later treated with He and Ar atmospheric pressure plasma in the form of a jet (APPJ) with fixed gas flow rate (1 slm) and at a fixed distance (2.5 cm) for 3 min. The gas sensing measurements were done by observing the change in resistance of the sensor in contact with 2 ml of ethanol in a chamber with 250 ml capacity. That measurement was done with Keithley 2460 oscilloscope. Other than gas sensing measurements, we did a different analysis of the sensor surface properties – SEM, Raman and FTIR. All measurements were conducted right after the plasma treatment (that was done after the Bucky paper preparation) and two weeks after that to investigate how did plasma treated sensors age over time.

All the measurements showed that plasma treatment significantly improves sensing properties and cycle stability. Additionally, Ar plasma gave better results than He plasma. The ageing effect is present but not comparable to the properties of the untreated sensor.

P-11 Swift heavy ion irradiation of graphene: exploring charge state effects

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Graphene is the finest example of the new class of materials known as 2D materials. Many exceptional physical and chemical properties found in this material attributed him a title of “wonder material”. For example, graphene exhibits extraordinary strength, chemical inertness and acts as impermeable membrane for everything except protons [1]. Irradiating graphene with ion beams can lead to production of vacancies and larger holes in it, thus making porous graphene suitable for filtering applications, sensing and catalysis. Our previous work on ion irradiation of graphene demonstrated successfully these points: range of ion beams and energies available at the RBI accelerator facility has been found suitable for production of porous graphene, both for the graphene on the SiO₂ substrate



[2] and in the freestanding graphene [3]. Production of graphene membrane in this manner, and evaluation of its performance, has been reported in ref. [4].

In this contribution, we present experimental results obtained by Raman spectroscopy together with first results of molecular dynamics simulations of damage introduced in graphene via swift heavy ion irradiation. We explored effects of the electronic excitation due to ion passage within the substrate SiO₂ underneath a single layer graphene. Additionally, we have experimentally shown that increase of the damage production in graphene is related to the ion charge state, and not only to electronic stopping power. By using molecular dynamics simulations we will investigate this novel feature where the ion charge state will be used as a parameter influencing electronic stopping power of the swift heavy ion.

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[2] O. Ochedowski et al., *Nanostructuring Graphene by Dense Electronic Excitation*, Nanotechnology 26 (2015) 465302.

[3] H. Vázquez et al., *Creating nanoporous graphene with swift heavy ions*, Carbon 114 (2017) 511

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P-12 Non-ambient XRD analysis of jumping crystals of oxitropium bromide and its derivative

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Thermosalient materials, or more colloquially called jumping crystals, are materials that during heating/cooling undergo an energetic phase transition which is so sudden and abrupt that the crystals are ballistically projected to heights of several hundred times larger than their own dimensions. Apart from providing visually extremely attractive phenomenon, these materials have a tremendous technological potential as the future self-actuation devices (nanoswitches, thermal sensors, artificial muscles, etc.)¹.

In situ X-ray analysis, often referred to as analysis at non-ambient conditions allows a study and investigation of any macroscopic property of a material that is directly related to its structural property (e.g. crystallographic symmetry, crystallite size, vacancies, size and shape of nanoparticles or pores). Temperature, pressure, gas atmosphere and mechanical stress trigger phase transformation, chemical reactions, crystal proprieties and so on.



Here we present a systematic experimental study of the thermosalient effect in oxitropium bromide and methscopolamine bromide with non-ambient x-ray diffraction measurements (in the range from 100K to 500K at low vacuum conditions), which enabled us to elucidate the thermosalient phenomenon in this system². Oxitropium bromide and methscopolamine bromide have very similar molecular structures, the only difference being that one ethyl group is replaced by methyl group in the case of methscopolamine bromide. Both compounds have medical uses, oxitropium bromide is used as a bronchodilator, whereas methscopolamine 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. Both systems are characterized by uniaxial negative thermal expansion, but there is an abrupt change of the unit cell parameters during the phase transition for the oxitropium bromide whereas the parameters are changing perfectly linearly in the whole temperature range of existence for methscopolamine bromide.

As in most of the thermosalient systems, immense negative thermal expansion seems to be the most likely candidate for the driving force behind this phenomenon in oxitropium bromide, leading the system to a sharp thermosalient phase transition which causes the crystals to jump. On the other hand, no structural phase transition seems to take place in methscopolamine bromide. Therefore, the question remains – what is causing the jumping in methscopolamine bromide – a system so close to oxitropium bromide but exhibiting totally different kind of thermosalient effect.

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P-13 CORROSION OF FeCrAl ALLOYS IN NITROGEN GAS AT HIGH TEMPERATURE

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The corrosion mechanism of commercial FeCrAl alloy (Kanthal AF) during annealing in nitrogen gas at 900 °C and 1200 °C was studied. Isothermal and thermo-cyclic tests with varying total exposure times, heating rates, and annealing temperatures were performed. Oxidation test in air and nitrogen atmosphere were carried out by thermogravimetric analysis. The microstructure was characterized by



scanning electron microscopy (SEM-EDX), Auger electron spectroscopy (AES), and focused ion beam (FIB-EDX) analysis. Our results show that the progression of corrosion takes place through the formation of localized subsurface nitridation regions, composed of AlN phase particles, which reduces the aluminum activity and causes embrittlement and spallation. The processes of Al-nitride formation and Al-oxide scale growth depend on annealing temperature and heating rate. We found that nitridation of the FeCrAl alloy is a faster process than oxidation during annealing in a nitrogen atmosphere with low oxygen partial pressure and represents the main cause of alloy degradation.

Acknowledgment

This work was supported by the Slovenian Research Agency through the project Selective plasma oxidation of alloys, ID L2-8181.

P-14 EXTERNAL MODIFICATION OF Ti ALLOY INDUCED BY O₂ PLASMA TREATMENT

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Plasma treatments are effective surface modification techniques, especially treatments of materials with low-pressure oxygen plasma. The treatment of the surfaces of materials with non-thermal plasma can lead to surface activation and functionalization. Plasma surface modification play an essential role in biomedicine. It can improve biocompatibility, biofunctionality, and strongly influence biological response in vivo. As biomaterials, titanium (Ti) alloys have attracted more attention in biomedicine. Treated Ti alloys with oxygen plasma are introduced to whole blood. Knowing the vascular implants (stents) could cause damage to the arterial walls by platelet activation and causing thrombus responses, therefore bioactive Ti alloys are treated with oxygen plasma to improve protein adsorption and cell adhesion. This treatment shows significantly reduced adhesion and activation of platelets in comparison with untreated Ti alloy.

P-15 Analysis of magnetron sputtering and triode sputtering plasmas using optical emission spectroscopy

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Magnetron sputtering and triode sputtering are plasma-based physical vapour deposition techniques for preparation of high-quality thin films. Magnetron sputtering is used for deposition of thin films on



an industrial scale in many technological areas, e.g. tribology, photovoltaics, microelectronics, optics, and others. Triode sputtering, on the other hand, is a less-known sputtering technique which is mainly employed for the preparation of thin films in laboratories. In magnetron sputtering, dense plasma is generated next to the target due to the strong magnetic field extending above the cathode. In triode sputtering, there is no magnetic field next to the target, instead auxiliary plasma source is used to sputter target material. Cathode voltage in magnetron sputtering is typically around -300 V, while in triode sputtering it is much higher, typically -1800 V. Due to very different cathode voltages and other discharge characteristics, the properties of the two types of plasmas differ considerably. The aim of this work was to analyze magnetron and triode sputtering plasma by optical emission spectroscopy.

We operated magnetron and triode discharges at different argon pressures and cathode voltages. The optical emission spectra were measured for several discharge parameters and target materials. Measurements showed very different characteristic spectra. In both sputtering regimes, the lower-wavelength lines (i.e. close to UV) increased in intensity with increasing discharge current and at the constant pressure. These lines, which belong to metal atoms and ions, increase in comparison to the lines for argon atoms and ions. This means that at higher discharge currents, the concentration of metal atoms and ions is also higher. On the other hand, the gas pressure did not significantly affect the optical spectra. The intensity of metal lines slightly increased in comparison to the argon lines when the current was constant and the pressure was increased from 0.2 Pa to 0.8 Pa. At approximately the same discharge power, the spectra for triode plasma significantly differed from the magnetron plasma. The triode plasma exhibited much more intense lines for titanium ions as compared to titanium atoms. Contrary, in magnetron plasma, the optical lines for titanium atoms were much stronger than for the titanium ions. This suggests much stronger titanium ionization in the triode sputtering technique than in the magnetron sputtering technique.

P-16 Properties of Ge quantum dot lattices in amorphous Al_2O_3 , Si_3N_4 and SiC matrices

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Production and basic properties of self-assembled Ge quantum dot (QD) lattices embedded in Al_2O_3 , Si_3N_4 , SiC matrices were studied. The materials are produced by magnetron sputtering deposition, using different substrate temperatures. Deposition regimes leading to self-assembled growth type and formation three-dimensionally ordered Ge QD lattices [1] in different matrices were investigated and determined.

Special interest was devoted to oxidation of Ge quantum dots in different matrices, and the best conditions for production of non-oxidized Ge quantum dots were found. Influence of shell addition to Ge QD core was explored, and its effect on oxidation of Ge. It was found that Si shell prevent



Ge oxidation even in oxide matrix. However, the shell oxidizes by itself. Optical properties of Ge quantum dot lattices in different matrices show strong dependence on Ge oxidation [2].

[1] M. Buljan et al., "Ge quantum dot lattices in Al₂O₃ multilayers," J. Nanoparticle Res., vol. 15, no. 3, 2013.

[2] N. Nekić et al., Production of non-oxidized Ge quantum dot lattices in amorphous Al₂O₃, Si₃N₄ and SiC matrices, Nanotechnology, <https://doi.org/10.1088/1361-6528/ab1d3c> (2019)

P-17 Upscaling of a laboratory plasma reactor to an industrial size

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A large plasma reactor was developed for industrial treatment of agriculture seeds to improve germination and to eliminate contamination with pathogens. The main discharge tube was made of a Pyrex glass with a length of 2 m and a diameter of 20 cm. Two excitation copper coils with five turns were mounted in the middle of the tube. The transfer of power was optimized by using two parallel overlapping and offset excitation coils that were serially connected to an RF generator via a matching network. Such a configuration enabled formation of more homogenous plasma in the discharge tube, and reduction of side effects caused by capacitive coupling in the plasma system. RF generator operated with an adjustable power up to 8kW. The system was pumped with a Roots pump backed with a two-stage rotary pump. The nominal pumping speed was 400 m³/h. This plasma reactor enables simultaneous treatment of several kilograms of seeds.

P-18 Effect of helium atmospheric pressure plasma jet on laser induced breakdown

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Here we show how preionisation, in terms of helium atmospheric pressure plasma jet, affects the laser induced breakdown. Since the plasma jet evolves in time and space, the electron density present in the laser focus is tuned by changing the time delay between the high voltage rising edge and the laser pulse arrival. It is shown that the free electrons present in the plasma jet decrease the breakdown



threshold. In general, more free electrons available for the inverse Bremsstrahlung leads to higher energy absorption rate from the laser pulse. The effect of laser pulse energy and high voltage amplitude on the ignition of the laser induced plasma is investigated. Measurements of absorbed energy along with optical emission spectra are done for different focal positions of the laser pulse. From that, the donut shaped electron density spatial profile is confirmed.

P-19 Advancements in laser optic catalytic sensor for characterization of non-equilibrium gaseous plasma

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Nowadays several methods for neutral atom density measurements in low pressure non equilibrium gaseous plasmas exist. Among those, only few are able to measure neutral atom density in real time and these are: Laser Stimulated Fluorescence (LSF), Cavity Ring Down Spectroscopy (CRDS), NO titration and Laser Optic Catalytic Sensors (LOCS). Each of them has its own pros and cons. As opposed to measuring electrically charged particles, which can be easily accumulated on a sensor's surface by adjusting voltage polarity, measuring neutral atoms in low pressure plasmas by non-optical techniques is quite difficult. By employing a catalytic sensor tip on an optical fiber, IR laser together with IR light detector and optical components, and custom designed electronics, we have developed an innovative measuring technique (LOCS) that meets real time system requirements when measuring neutral atom density. To date, we have tested the system in oxygen, air and hydrogen low pressure gaseous plasma afterglow. To be able to measure the density of neutral atoms inside plasma, we have developed a special housing for the sensor itself, however, during process testing several issues, which need to be solved, emerged. Besides measuring hydrogen and oxygen neutral atoms in a plasma afterglow, we report preliminary measurements of hydrogen atoms inside plasma.

P-20 Sterilization of corn seeds with H₂ and SO₂ plasma

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Use of plasma technologies in agriculture is new discipline, rapidly growing in last few years. Plasma is used for treatment of various seeds, vegetables and fruits in order to improve seed germination and



decontamination of bacteria, viruses, fungi and toxins from the surface. This way, plasma treatment also prolongs the durability of foods.

In our study, corn seeds which were contaminated with three different fungi *Fusarium*, *Penicillium* and *Mucor* were used. Corn seeds were exposed to SO₂ and hydrogen plasma. Hydrogen plasma was created by inductively coupled electrodeless radio-frequency discharge in E and H-mode. The generator operated at the standard frequency of 13.56 MHz. The forward power was set to 150 W in E-mode and to 280 W in H-mode. In each experiment about 13 g of infected corn seeds were treated. The pressure was fixed to 15 Pa and the flow rate was 52 sccm. When corn seeds were treated with SO₂

P-21 β -TaON THIN FILMS: PRODUCTION BY REACTIVE MAGNETRON SPUTTERING AND THE QUESTION OF NON- STOICHIOMETRY

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In this work we report on the structure, morphology, composition, chemical bonding and optical properties of tantalum oxynitride (Ta-O-N) thin films prepared by reactive direct current magnetron sputtering. The films have been investigated by grazing incidence X-ray diffraction (GIXRD), X-ray reflectivity (XRR), grazing incidence small angle X-ray scattering (GISAXS), time-of-flight elastic recoil detection analysis (TOF-ERDA), X-ray photoelectron spectroscopy (XPS) and spectroscopic ellipsometry (SE). The composition and the partial pressure of the reactive atmosphere (N₂+O₂) have been varied in order to find conditions suitable for the β -TaON phase production. As prepared thin films are amorphous and annealing at $T_a > 750$ °C is necessary to promote crystallization. We discuss the role of N₂ gas on the kinetics of sputtered particles and its influence on the oxidation rate and porosity of the growing film. The anion composition in Ta-O-N films strongly depends on the reactive gas condition during the deposition. We found that the O/N ratio in β -TaON films increases with more N₂ or O₂ gas in the chamber, whereas the corresponding absorption onset and valence band maximum show a blue shift of up to 0.5 eV. These results were related to the non-stoichiometry in β -TaO_xN_y crystallites with $x > y$.

P-22 Periodically nanorippled graphene as a route towards straintronics

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Nano-rippled graphene, a structurally modified strained graphene, presents a novel material with a large range of possible applications including sensors, electrodes, coating, optoelectronics, spintronics and straintronics. In this work we will show two ways of producing strained graphene with well-defined uniaxial and biaxial periodic modulations, and their resulting changes in electron behavior, a bottom-up and a top-down approach. In the bottom-up approach we have synthesized macroscopic single layer graphene on a stepped Ir(332) substrate, investigated its morphology and electronic structure using scanning tunneling microscopy (STM) and spectroscopy (STS), and transferred it to a dielectric support. Upon transfer, a millimeter sized graphene flake with a uniform periodic nanoripple structure is obtained, which exhibits a macroscopically measurable uniaxial strain. The periodic one dimensional arrangement of graphene ripples was confirmed by atomic force microscopy (AFM) and polarized Raman measurements. In the top-down approach we assemble a biaxially strained graphene by transferring it on 20 nm SiO₂ nanospheres deposited on Si wafer in a monolayer with hexagonal close packing. Such graphene exhibits altered electronic band properties and strain related effects which we have measured using STM and STS. Our findings demonstrate a viable route to alter the electronic structure of graphene by means of strain and substrate interaction and are promising for large-area electronics and optoelectronics applications.

P-23 Porozne nano-membrane: razmatranje svojstava razgranatih mreža pora u keramičkoj Al₂O₃ podlozi

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U radu ćemo pokazati rezultate istraživanja hidrauličkog otpora pri protoku fluida kroz trodimenzionalne mreže šupljina u keramičkoj alumina matrici. Spomenute membrane pripremaju postupkom magnetronskog rasprašenja koji uz posebne uvjete depozicije omogućava samouređujući rast trodimenzionalno uređenih mrežastih struktura pogodnih za izradu membrana.



U radu je istražen utjecaj geometrije trodimenzionalnih mreža na hidraulički otpor pri protoku fluida. Rezultati pokazuju mogućnost znatnog smanjenja hidrauličkog otpora u odnosu na ravne cilindrične šupljine, odabirom geometrije nanomreža.

P-24 Optimizacija strukturnih parametara mreža nanošupljina za skladištenje vodika

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Nanoporozni materijali su između ostalog korisni za skladištenje raznih plinova, zbog svoje velike unutarnje površine na koju se molekule plina mogu vezati. Danas je posebno zanimljivo skladištenje vodika u takvim materijalima jer takav način skladištenja ima brojne prednosti nad standardnim skladištenjem pod visokim tlakom.

U ovom radu istraživana su optimalna strukturna svojstva pravilnih mreža nanošupljina u amorfnim matricama za skladištenje plinova. Takvi materijali dobivaju se postupkom depozicije magnetronskim rasprašenjem, koji u posebnim uvjetima depozicije vodi do nastanka pravilno uređenih mreža nanošupljina. Pronađeni su strukturni parametri za koje takvi materijali imaju maksimalnu unutarnju površinu. Dobiveni rezultati su uspoređeni sa realnom strukturom izrađenih materijala, te su određeni potencijalno najbolji materijali za skladištenje vodika.

P-25 Effect of nitrogen presence during the deposition on structural and optical properties of Ge quantum dot lattices in alumina matrix

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Effect of nitrogen presence during magnetron sputtering deposition of Ge quantum dot lattices embedded in alumina matrix was studied. Three series of samples were deposited by varying substrate



temperature, nitrogen flow rate and germanium content. The aim was to examine the effects of these parameters on structural and optical properties of the material and the self-assembled growth regime. Special attention was devoted to oxidation of Ge quantum dots, and its effect on the optical properties of the material. It was shown that the presence of N affects the size of Ge QDs, their oxidation and optical properties. Deposition temperature and Ge content also affect Ge QD sizes and consequently band gap, and optical properties. Thus, the combination of the mentioned parameters presents a powerful tool for control of the materials properties that are important for the optoelectronic applications.

P-26 NEW VACUUM GAUGE CALIBRATION SYSTEM BY PRIMARY STATIC EXPANSION METHOD AT IMT

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Institute of Metals and Technology (IMT) in Ljubljana is realizing and maintaining Slovenian national standards for vacuum and pressure. IMT has accreditation for calibrations of vacuum and pressure gauges in the range from 10^{-6} Pa to 500 MPa. Slovenian national standards at IMT are currently composed of a set of vacuum and pressure transfer standards which are traceable to foreign national metrology institutes (NMIs) with primary realization of the pressure scale. In vacuum range our transfer standards are spinning rotor gauge (SRG) and capacitance diaphragm gauges (CDGs). For both types of instruments the long term stability between two periodic calibrations is one of the main contributions to the uncertainty of accredited calibration and measurement capability (CMC). To reduce our CMC in the vacuum range we decided to construct a new primary static gas expansion calibration system which will become the Slovenian national standard for vacuum range from 10^{-2} Pa to 10 kPa.

We will present main construction details of the new IMT static expansion calibration system and preliminary evaluation of the relevant metrological properties for calculation of the generated calibration pressure and its uncertainty.

The purchase of the new static expansion system was partly financed by the European Union from the European Regional Development Fund (ERDF).

P-27 Application of SF₆ plasma for surface fluorination of polymers

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SF₆ plasma was investigated for use in surface fluorination and hydrophobization of polymers. SF₆ plasma was created in a Pyrex tube by radiofrequency plasma operated at a fixed nominal power of 200 W. The pressure of SF₆ gas was varied in a range between 10 and 200 Pa. Polymer polyethylene terephthalate was exposed to SF₆ plasma for 40 s.

The surface of plasma-treated polymer was analysed by X-ray photoelectron spectroscopy (XPS) and water contact angle (WCA) measurements, whereas the SF₆ plasma was characterized by optical emission spectroscopy. After plasma treatment, 46 at. % of fluorine was found on the polymer surface, whereas the oxygen concentration decreased from the initial 26 to 10 at. %. It was found that the extent of the polymer surface fluorination depended on the pressure. Up to a threshold pressure of approximately 130 Pa, the XPS concentration of fluorine on the polymer surface (~ 46 at. %) and the surface hydrophobicity (WCA 106°) were similar, which was explained by the full dissociation of the SF₆ gas, leading to high concentrations of fluorine radicals in the plasma and thus causing the saturation of the polymer surface with fluorine functional groups. Above the threshold pressure, the amount of fluorine on the polymer surface significantly decreased to only 16 at. %, whereas the oxygen concentration increased (from 10 at. % to almost 30 at. %), leading to the formation of more hydrophilic surface (WCA 35°).

P-28 Neutral reactive gaseous species in plasma reactors for tailoring surface properties of solid materials

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Neutral reactive species play an important role in tailoring surface properties of solid materials upon exposure to gaseous plasma. Unlike charged particles that acquire kinetic energy in the strong electric field in sheath between plasma and surface the neutral particles remain thermal at the gas kinetic temperature which is usually close to the room temperature in low-pressure plasma sustained by electrodeless discharges of low power density. The interaction of neutral species with solid materials is thus pure chemical so they interact specifically with different materials depending on the surface composition. The density of neutral reactive species therefore depends on the type of material facing plasma and strong gradients are observed next to materials of high probability for surface recombination. Several examples will be shown and explained.

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Figures on the cover:

Wettability development on a laser-textured stainless steel surface (Marjetka Conradi)

Electric field enhancement (logarithm) scale for an Ag dimer in water, with radius 25 nm and separated 2 nm for light polarized parallel to the axis dimer. (Jordi Sancho Parramon)

Growth data of *E. coli* treated with different concentrations of laser synthesized silver nanoparticles, i.e. different colloid volume shares *v.* The OD in absorbance units is given on the left axis and the corresponding bacterial concentrations in CFU/ml is given on the right axes. (Lucija Krce)

A Fixed Length Optical Cavity - NIST (Sefer Avdiaj)





1. ANALITSKA OPREMA

www.jeol.com www.oxford-instruments.com/products/microanalysis

- presevni elektronski mikroskopi (**TEM, FEGTEM**)
- vrstični elektronski mikroskopi (**SEM, FEGSEM**)
- Auger spektrometri in XPS spektrometri (**ESCA**)
- masni spektrometri (**MS MALDI TOF, GC TOF, QMS**)
- **NMR** spektrometri, ESR spektrometri
- **EDS** –energijsko disperzivni spektrometri
- **WDS** - valovno dolžinski spektrometri
- **EBSD** – analiza z elektronsko difrakcijo
- **EBL** – elektronska litografija

2. PRIPRAVA VZORCEV

www.jeol.com

www.gatan.com

www.rmcproducts.com

www.quorumtech.com

www.2spi.com

- vakuumski napaževalniki
- instrumenti za brušenje in poliranje, jedkanje
- vakuumska impregnacija
- **FIB** - instrumenti za ionsko jedkanje
- **IS** – ionsko tanjšanje za TEM
- Mehanska priprava za TEM vzorce
- diamantne žage (na disk, na diamantno nit)
- vakuumski napaževalniki
- mikrotomi in ultramikrotomi
- **CP** – ionsko jedkanje površin brez defektov na površini in mejah zrn za FEGSEM

3. KRIOTEHNIKA

www.cryopal.com

- kontejnerji za prenos, pretakanje, skladiščenje tekočih plinov (tekoči dušik, helij, ...)
- kontejnerji za shranjevanje bioloških vzorcev v tekočem dušiku
- kontejnerji za suh transport zmrznjenih vzorcev na temperaturi tekočega dušika
- instrumenti za programirano zamrzovanje do -196°
- zaščitna oprema za kriotehniko



4. VAKUUMSKA TEHNIKA

www.pfeiffer-vacuum.com

www.vatvalves.com

www.hsr.li

- vse vrste vakuumskih črpalk (rotacijske, suhe, roots, membranske, difuzijske, turbo-molekularne, krio, ...)
- vakuumski merilniki
- kvadрупolni masni spektrometri (QMS)
- vakuumski sistemi po naročilu
- He detektorji netesnosti
- vakuumski ventili, komore, komponente, olja, masti, ...

5. LABORATORIJSKI INSTRUMENTI

www.labogene.com

- zamrzovalne omare - horizontalne, vertikalne, različnih velikosti do -90 °C
- liofilizatorji do -110 °C temperature kondenzorja
- termostatisane vodne kopeli
- brezprašne komore
- vakuumski koncentradorji
- samostoječe zamrzovalne centrifuge
- namizne centrifuge

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Mikrotomoci, histokinetti, farbači, parafinski uklapači itd.

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