DEPARTMENT OF SURFACE ENGINEERING AND OPTOELECTRONICS F-4

The research program is associated with vacuum science, technology and applications. The main activities are focused on plasma science, the modification of advanced biomedical materials and products for improved biocompatibility, the characterization of inorganic, polymer and composite materials with different thin films on the surface, the modification and characterization of fusion-relevant materials, the thermodynamics of trapped gases and methods for sustaining an ultra-high-vacuum environment, vacuum optoelectronics, and basic research in the field of surface and thin-film characterization by electron and ion spectroscopy techniques.

Head:
Prof. Miran Mozetič

The advanced plasma reactors available in our labs as well as at our partners in Slovenia and abroad were used for tailoring the surface properties of solid and liquid materials. The surfaces and thin films were characterized by various complementary techniques, which allow us to understand the complex mechanisms involved in plasma–surface interactions. The analytical equipment for the surface and thin-film analyses was upgraded with two important innovations. First, there was an Auger electron spectrometer, model JAMP 7830F, produced by Jeol company. This instrument replaced a 40 years old Auger electron spectrometer, model SAM 545A, which remained operable until the new instrument was commissioned. The recently installed Auger JAMP 7830F instrument is a combination of a field-emission electron microscope with a high lateral resolution and an Auger electron spectrometer. The instrument operates in ultra-high vacuum and allows us to analyse accurately the composition of surfaces, thin films and nanostructures within a region of a few nm. Sputtering with an argon-ion gun allows for the removal of the surface layer and thus depth profiling up to a depth of around 1 micrometre.

The analytical equipment for surface and thin-film analyses was upgraded with two important additions, an Auger electron spectrometer and an ion gun for the ToF-SIMS spectrometer.

Another important improvement in our analytical equipment was the upgrade of the ToF SIMS ION-TOF.5 instrument, an advanced mass spectrometer for the precise surface characterization or organic and inorganic materials. This instrument was upgraded with a new sputter ion gun, model DCS, produced by the ION TOF company, which allows for high-precision analyses of thin films of thicknesses between a few nanometres and a few micrometres. A new ion gun performs the bombardment of the sample surface with either Ar^+, O_2^- or Cs^+ ion beams at low kinetic energies (from 0.5 to 2 keV) and high sputtering rates up to 1 nm/s. Such performances are the consequence of highly focused beams. In this way a controlled removal of the surface layers can be obtained. The simultaneous mass analysis of emitted secondary ions from a sample surface allows us to measure the depth distribution of the elements in thin films and multilayer structures. Such depth profiles can be measured with a very high depth resolution (a few nm) and high sensitivity for the detection of elements, even in the ppm region. Both new instruments in our laboratory will also make possible advanced studies of diffusion phenomena in thin solid films, atomic transport through internal interfaces of multilayer structures and analyses of the depth distribution of elements, dopants and impurities. Thanks to these two recent upgrades of our analytical equipment we will continue and extend our decades-long tradition in high-quality surface and thin-film analyses.

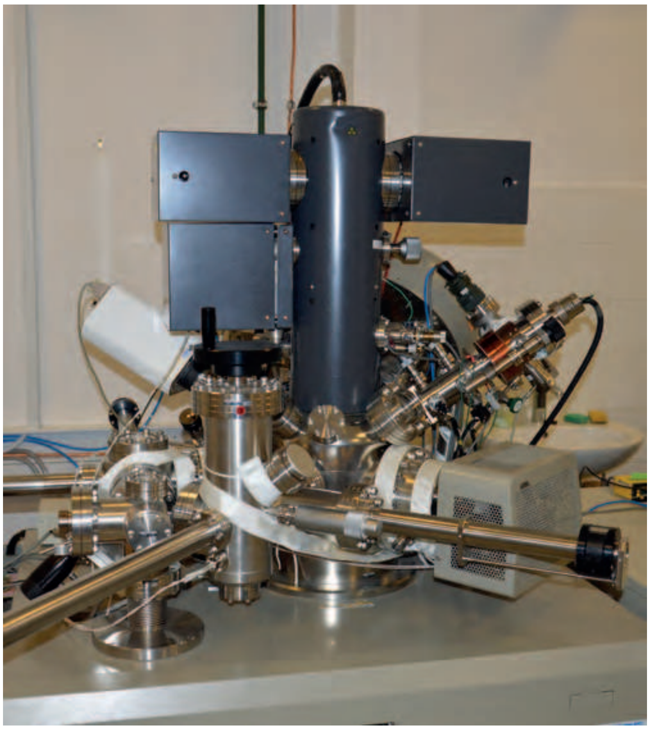

Figure 1: Auger electron spectrometer installed in 2017 enables the precise analyses of surfaces, thin films and nanostructures with depth and lateral resolutions of a few nm and about 20 nm, respectively.

Photo-catalysts offer several promising applications, such as hydrogen evolution via water splitting, CO_2 reduction and the removal of organic pollutants from water. Due to its high photocatalytic activity and chemical stability, low cost, water insolubility and nontoxicity, TiO_2 has been widely applied, studied and identified as the best photocatalyst for the decomposition of pollutants present in an aqueous medium. The drawbacks of TiO_2 are a wide band-gap energy and fast electron-hole recombination. Many different attempts have been tested to overcome

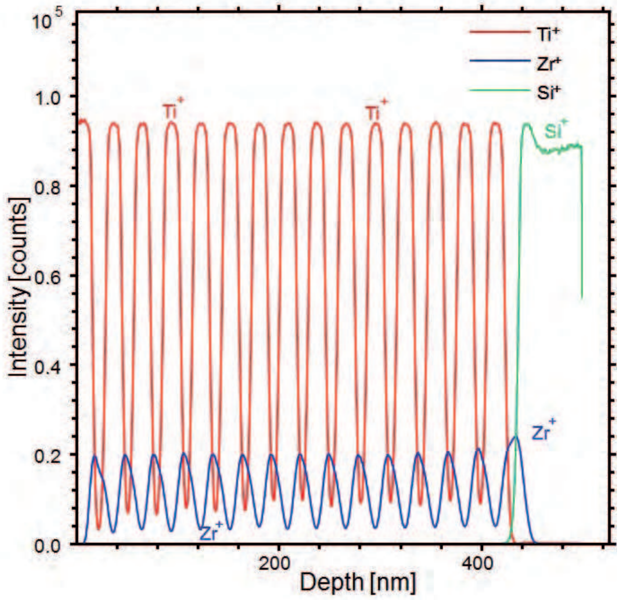

Figure 2: High-quality SIMS analysis can be recognized by sharp interfaces between layers in a multilayer structure of 30 layers of Ti(15 nm)/Zr(15 nm) on Si substrate.

the drawbacks of TiO_2, such as doping TiO_2 with other metal or non-metal elements or coupling with different semiconductors. Modifying TiO_2 with carbon materials, like carbon nanotubes and graphene, has attracted a lot of attention due to an exceptionally high electron mobility in carbon. In collaboration with National Institute of Chemistry from Ljubljana we performed a study on photo-catalysis from composite materials of TiO_2 nanorods and reduced graphene oxide (rGO) with nominal loadings of rGO from 4 to 20 wt. %. The composites were prepared by hydrothermal synthesis. We investigated in detail how the increasing amount of rGO influences the structural and electrical properties, and how this manifests itself in the photocatalytic activity. Our group performed precise XPS analyses of the chemical composition, chemical bonding and electronic structure of the composite materials. In this way we found that the charge separation in TiO_2+rGO composites is related to a perfect matching of TiO_2 and rGO valence band maxima. As a consequence, the electron-hole recombination in composites was slower, resulting in more electrons and holes being able to participate in the photocatalytic reaction. Degradation rates of pollutants in water in photocatalytic experiments correlated well with the XPS and other results.

The graphene decorated with catalytic nanoparticles represents an attractive material for electro-catalysis, providing it is oriented vertically on a substrate so that the effective surface area becomes enormous. With our colleagues from the National Institute of Chemistry we investigated the possibility of using carbon nanowalls (CNWs) for increasing the stability of electrodeposited nanocatalysts, which is a major task for future industrial applications. The most common degradation mechanism is the loss of active surface area due to nanoparticle growth via coalescence/agglomeration. We proposed a particle confinement strategy via carbon nanowall deposition to overcome the degradation of the catalyst's nanoparticles. With a CNW-modified electrode a much better stability was obtained compared to the non-modified electrode. IL-SEM images before and after 15,000 ageing cycles confirmed the superior stability of the CNW-protected Ag nanocatalyst exhibiting no agglomeration or coalescence. The graphene structure acts as a barrier for particle migration and cluster formation. This strategy can be considered as a general way to prevent coalescence/agglomeration of metallic nanoparticles for any electrochemical reaction and other applications beside electro-catalysis and thus increase the long-term stability of nanoparticles.

Since one of the greatest challenges in the commercialization of graphene and its derivatives is the production of high-quality material in bulk quantities at low price and in a reproducible manner, we were also closely collaborating with the University of Lisbon on this topic. The main focus area was the synthesis of pure graphene and carbon nanowalls. Our results demonstrated that a microwave plasma-enabled synthesis exhibits a great potential for a scalable route that would enable the continuous, large-scale fabrication of free-standing graphene and nitrogen-doped graphene sheets. The method's crucial advantage relies on harnessing unique plasma mechanisms to control the material and energy fluxes of the main building units on the atomic scale. By tailoring the high-energy density plasma environment, a controllable selective synthesis of high-quality graphene sheets at 2 mg/min yield with prescribed structural qualities was achieved. The method demonstrated the great promise for the large-scale fabrication of graphene and its derivatives, and is a cost-effective alternative to the presently used chemical methods. A fruitful collaboration on this topic resulted in a successful EU Horizon 2020 FET-Open project "Pegasus".

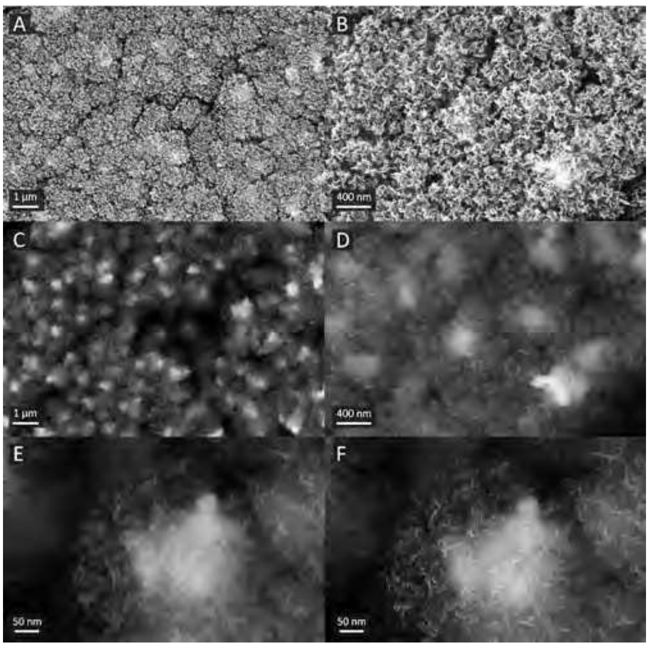

Figure 3: SEM images of Ag nanoparticles on a CNW electrode obtained with different detectors before degradation cycles (A-E). (F) - after 15,000 cycles.

Titanium is commonly used for body implants, and can be coated with a nanostructured oxide film to improve its properties. A novel approach for the modification of titanium alloys used for vascular implants (stents) was studied. It is well known that the biological response of a stent is still far from optimal, mainly due to restenosis. Currently, the restenosis presents the main drawback on all metal stents, as it occurs in more than 33% of the cases. Stents can be divided on bare metal stents

(BMS) and drug-eluting stents (DES). With DES the problems of allergenic reactions as well as the risks of restenosis are lowered, as DES release anti cell-proliferative, immunosuppressive or anti-thrombogenic drugs, which inhibit the proliferation of smooth muscle cells and reduce thrombus formation. However, it was shown that DES also inhibits normal endothelium growth, which potentially leads to thrombosis. Thus the aim of our work was to alter the surface properties of stents in order to reduce platelet adhesion and at the same time improve the endothelial cell proliferation without the need to use drug-eluting coatings. The stents were coated with titanium oxide nanotubes. By altering the conditions of electrochemical anodization, nanotubes of different diameter and length were fabricated. The as-deposited coating was further treated with an oxygen plasma in order to obtain super-hydrophilic surface finish, which significantly influenced the proliferation of endothelial cells (HCAECs). Furthermore, a significant decrease in the activation and adhesion of platelets was observed on these surfaces, which reduces the possibilities for undesired thrombotic reactions. Due to the applicability of our results, the innovative approach was protected by filing a patent application. A contract for collaboration with the Swiss company Rontis, one of the leading producers of vascular stents, was signed.

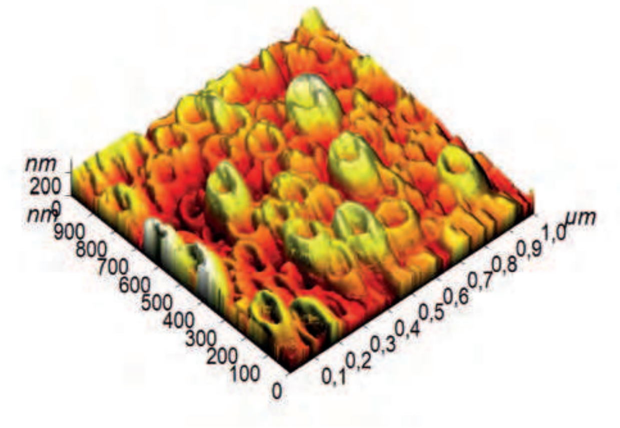

Figure 4: Titanium oxide nanotubes of diameter 100 nm, as observed by atomic force microscopy (AFM).

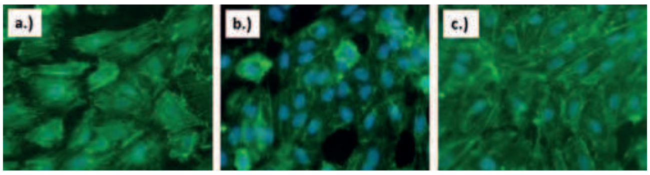

Figure 5: Fluorescent images of endothelial cells (HCAEC) on a Ti foil (left), as synthesized TiO_2 nanotubes (middle) and plasma treated TiO_2 nanotubes.

Low-pressure gaseous plasma can also be used for modifying the surfaces of biomedical materials made from polymers. While the best anti-thrombogenic material is a coating made from heparin, such coatings are difficult to apply because they are quickly removed in a real environment, so we just functionalized the polyethylene terephthalate surface with sulfonate groups in order to mimic heparin. We used inductively coupled plasma in mixtures of SO_2 and O_2. X-ray photoelectron spectroscopy and atomic force microscopy showed weak functionalization of the samples' surfaces with sulphur-containing groups and revealed the highly altered morphology of plasma-treated samples. The samples were then incubated with human umbilical vein endothelial cells (HUVECs) and various biological tests were performed. The biocompatibility demonstrated a well-pronounced maximum versus gas composition, which correlated well with the development of the surface morphology. The best proliferation was observed in the case of nearly the same amounts of both mentioned gasses what was explained by the formation of $-SO_x$ radicals on the surface of the polyethylene terephthalate – similar to the groups in heparin. Such a surface finish seems to be stable so the innovative treatment with SO_2/O_2 plasma represents an interesting alternative to classic heparin coatings.

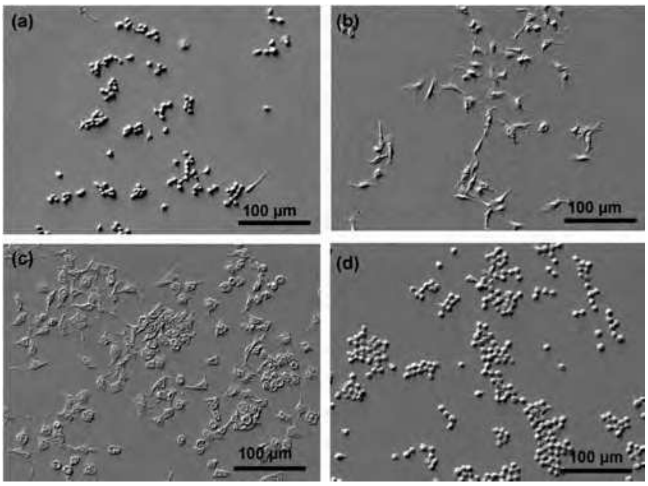

Figure 6: SEM images of cells on PET surfaces treated in various SO_2 + O_2 mixtures after 24 hours of incubation: (a) 100% SO_2; (b) 90% SO_2 + 10% O_2; (c) 60% SO_2 + 40% O_2; (d) 10% SO_2 + 90% O_2.

Atmospheric pressure plasmas, especially atmospheric pressure plasma jets (APPJs), offer new possibilities in medical applications, such as the treatment of cancer cells and wound treatment. Furthermore, APPJs also proved to be useful in various fields of dentistry, including the surface modifications of dental implants, adhesion of protective materials to teeth, caries treatment, endodontic treatment and tooth bleaching. Our study was focused on the stability of the chemical surface modification of the human dental enamel and dentine by using helium single-electrode multiple-harmonic APPJs. Modification of the enamel and dentine surface was observed using contact-angle measurements and X-ray photoelectron spectroscopy. The results of this study showed increased wettability of the enamel and dentine, increased Ca/P ratio, which was close to an ideal ratio for the highest volume of re-mineralization, and an increased O/C ratio, which confirmed the oxidation of transparent organic matrix in the enamel – one of the major processes in tooth bleaching.

Use of atmospheric pressure plasmas was also extended to other areas of biomedical research. We explored atmospheric pressure plasma depositions of antibacterial coatings, the treatment of model wounds, decontamination of natural toxins and the selective deactivation of cancer cells. Biomedical applications of plasma require its efficacy for specific purposes, whereas its safety is equally important. These safety aspects of cold plasma with simple atmospheric pressure plasma jet produced with helium gas were evaluated in skin damage on a

Figure 7: Cover image of Applied Physics Reviews with our illustration.

mouse, for different durations of exposure and gas-flow rates. The extent of skin damage was systematically evaluated using a stereomicroscope, labelling with fluorescent dyes, histology, infrared imaging and optical emission spectroscopy. The analyses revealed early and late skin damages as a consequence of plasma treatment, and were attributed to the direct and indirect effects of plasma. The results indicate that direct skin damage progresses with longer treatment times and increasing gas flow rates. With increasing flow rates, the temperature of the treated skin increases and so do the reactive oxygen and nitrogen species (RONSs). The direct effects depended on plasma parameters, whereas the secondary effects were rather independent of the discharge parameters and related to the diffusion of RONS. Thermal effects and skin heating were related to plasma coupling and were separated from the effects of RONS. It was demonstrated that a cumulative treatment with a helium plasma jet could lead to the skin damage. Our results provide guidance for researchers working on atmospheric pressure plasma jets for skin treatments worldwide.

Atmospheric pressure plasma was also used as a tool for the removal of mycotoxins, which are secondary metabolites produced by several filamentous fungi. These toxins frequently contaminate our food, and can result in human diseases affecting vital systems such as the nervous and immune systems. Intensive food production is contributing to the incorrect handling, transport and storage of the food, resulting in increased levels of mycotoxin contamination. Mycotoxins are structurally very diverse molecules necessitating versatile food-decontamination approaches, which are grouped into physical, chemical and biological techniques. Our new and promising approach demonstrated a high mycotoxin destruction efficiency as compared to classic techniques.

Similarly, we used plasma treatment as an alternative therapy for bone cancer, either primary or metastatic. The classic treatments are difficult to implement and not always effective. An alternative therapy could be cold plasma generated at atmospheric pressure, which has already demonstrated selective anti-tumour action in a number of carcinomas and in rather rare brain tumours. An atmospheric pressure plasma jet was employed to validate its selectivity towards osteosarcoma cells versus human mesenchymal stem cells. Effects on cells during the direct interaction of plasma jet with cells were compared with indirect interaction when only the liquid medium was treated and subsequently added to the cells. The delayed effects led to 100% bone cancer cell death through apoptosis stages, while healthy cells remained almost fully viable and unaffected by the treatment. The high efficiency of the indirect treatment indicates that an important role is played by the RONS in the gaseous plasma, which are transmitted into the liquid phase, which in turn led to lethal and selective action towards osteosarcoma cells. These initial findings might open another pathway for the treatment of metastatic bone disease with a minimal invasive approach.

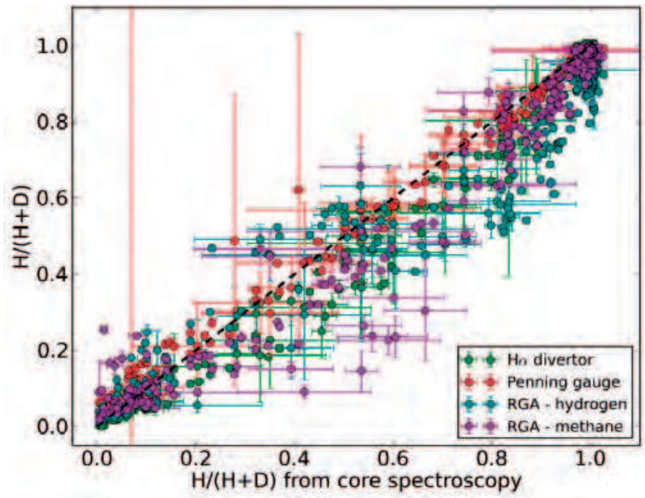

Figure 8: Average values of H/(H+D) ratios from the sub-divertor Penning gauge, H-alpha divertor spectroscopy and RGA hydrogen as well as methane signals in JET discharges.

In recent years we occasionally participated in fusion experiments in the two European tokamaks: JET and ASDEX-U. JET (Joint European Torus) is currently the biggest operating fusion reactor in the world, located in the Culham Centre for Fusion Energy in Oxfordshire, UK. ASDEX-U is a divertor tokamak located in the Max-Planck-Institut für Plasmaphysik, Garching, and it is Germany's second largest fusion device after the newly-built stellarator Wendelstein 7X in Greifswald. Our main interest was the investigation of ammonia production in nitrogen-seeded fusion-plasma discharges. The detection of ammonia with Residual Gas Analysers (RGA), mounted either in the tokamak divertor or the mid-plane, was impeded by the presence of water and methane, which, in a mixed hydrogen-deuterium system, leave signatures in the same range of the mass spectra. We suggested a new statistical model and fitting procedure, and applied it successfully to experimental data from nitrogen-seeded campaigns at ASDEX-U and JET. Our novel method of RGA spectra analysis was also used for the evaluation of the hydrogen isotope content at both tokamaks.

Tritium retention studies in future nuclear fusion reactors represent an important activity as maximum tolerated absorbed tritium dose determines the reactor's lifetime. To avoid risky experiments with radioactive tritium, many experiments are performed using hydrogen or deuterium instead. To compensate for the lower detection capability of these two hydrogen isotopes, all contributions which influence the accuracy of the results should be well recognized. In our very precise measurements of hydrogen permeation through impermeable membranes or in Thermal Desorption Spectroscopy (TDS) experiments, we determined hydrogen/deuterium absorption within oxide layers of an ultra-high-vacuum system. Pressure measurements were performed by non-ionizing gauges as we had to eliminate unacceptably high influence of positively charged gaseous ions. We performed hydrogen exposures at conditions that simulated true conditions in fusion reactors. Results were applied in fusion-related studies of mixed Be/W layers with oxygen and carbon. Namely, the formation of Be/W layers during plasma operation is evident from JET experiments and the retention of tritium in these layers should be predictable. Unfortunately, mixed Be/W layers in a real device will also contain oxygen and carbon, which further increases the number of chemical states for hydrogen retention. Several samples of mixed layers on pure tungsten substrates were investigated by TDS cycles performed up to 700°C.

Some outstanding publications in the past three years

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3. Tatarova, E., et al. Towards large-scale in free-standing graphene and N-graphene sheets. *Scientific reports*, 2017, 7: p. 10175-1-10175-15.
4. Recek, N., et al., Cell Proliferation on Polyethylene Terephthalate Treated in Plasma Created in SO_2/O_2 Mixtures. *Polymers,* 2017. 9(3): p. 82.
5. Šantak, V., et al., Surface Treatment of Human Hard Dental Tissues with Atmospheric Pressure Plasma Jet. *Plasma Chemistry and Plasma Processing*, 2017. 37(2): p. 401-413.
6. Hojnik, N., et al., Mycotoxin decontamination of food: cold atmospheric pressure plasma versus "classic" decontamination. *Toxins* 2017, 9(5): p. 151-1-151-19,
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9. Drenik, A., et al., Detection of ammonia by residual gas analysis in AUG and JET. *Fusion Engineering and Design*, 2017, 124: p. 239-243.
10. Nemanič, V., et al., Hydrogen interactions with oxidized austenitic stainless steel. *Journal of vacuum science & technology. A*, 2017, 35(2): p. 2017/35-1-2017/35-6.

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INTERNATIONAL PROJECTS

1. COST TD1208; Electrical Discharges with Liquids for Future Applications; COST Training School on Liquid Discharges
   Prof. Uroš Cvelbar
   Cost Office
2. NATO Grant; SPS 984555; Atmospheric Pressure Plasma Jet for Neutralisation of CBW
   Prof. Uroš Cvelbar
   Nato - North Atlantic Treaty Organisation
3. COST CA15114; Anti-Microbal Coating Innovations to prevent Infectious Diseases (AMICI)
   Prof. Uroš Cvelbar
   Cost Office
4. COST TD1305; Improved Protection of Medical Devices Against Infection (IPROMEDAI)
   Dr. Martina Modic
   Cost Office
5. H2020 - PEGASUS; Plasma Enabled and Graphene Allowed Synthesis of Unique nano Structures
   Prof. Uroš Cvelbar
   European Commission
6. H2020-EUROfusion-Plasma Facing Components-1-IPH-FU, EUROFUSION
   Asst. Prof. Rok Zaplotnik
   European Commission
7. H2020 EUROfusion - Education-ED-FU
   Prof. Miran Mozetič
   European Commission
8. H2020 EUROfusion - Medium Size Tokamak Campaigns-MST1-FU
   Asst. Prof. Rok Zaplotnik
   European Commission
9. Sniffing for Carcinogenic Substances - Research for Toxic Gas Molecule Sensing with Networks of Carbon Nanowalls
   Prof. Uroš Cvelbar
   Slovenian Research Agency
10. Biocompatible Nanostructured Tetragonal Zirconium Oxide Thin Films with Alternative Stabilization Dopants
    Prof. Miran Mozetič
    Slovenian Research Agency
11. Innovative Method for Synthesis of Thin Absorption Films for Photovoltaics
    Asst. Prof. Alenka Vesel
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12. Sterilization of Heat-sensitive Materials with Innovative Plasma Source of UV Radiation
    Prof. Miran Mozetič
    Slovenian Research Agency
13. Dust in Plasmas (DIP)
    Prof. Miran Mozetič
    Slovenian Research Agency

14. Making Luminescent C-dots and GQDs Based on Atmospheric Pressure Microplasma-Liquid Interaction
Prof. Uroš Cvelbar
Slovenian Research Agency
15. Quantitative Depth Profiling of Ultra-Thin Films
Asst. Prof. Janez Kovač
Slovenian Research Agency
16. Catalytic Activity of Nanomaterials for Elimination of Sulfur
Prof. Uroš Cvelbar
Slovenian Research Agency
17. Determination of Neutral-Atom Densities in Large Plasma Reactors
Prof. Miran Mozetič
Slovenian Research Agency
18. Plasma Assisted-Deposition of Antibacterial Coatings and their Testing
Dr. Martina Modic
Slovenian Research Agency
19. Plasma-Assisted Design of Multifunctional Carbon Nanowalls Bio-Sensor
Prof. Uroš Cvelbar
Slovenian Research Agency
20. Membership Chair of ECS - Division Dielectric Science and Technology Electrochemical Society
Prof. Uroš Cvelbar
Slovenian Research Agency
21. Innovative Coatings for Bare Metallic Vascular Stents for Reduction of Restenosis and Acceleration of Natural Endothelization
Prof. Miran Mozetič
Slovenian Research Agency
22. Consequences of electron emission from hot plasma-facing components in nuclear fusion reactors
Prof. Miran Mozetič
Slovenian Research Agency

RESEARCH PROGRAMS

1. Vacuum technique and materials for electronics
Dr. Vincenc Nemanič
2. Thin film structures and plasma surface engineering
Prof. Miran Mozetič

R & D GRANTS AND CONTRACTS

1. Nanoscale engineering of the contract interfaces for green lubrication technology
Asst. Prof. Janez Kovač
2. Multifunctional electrospunned nanofibers development and dynamic interaction studies with pathogen bacteria
Prof. Miran Mozetič
3. Understanding plasma processes and thin film growth in High Power Impulse Magnetron Sputtering
Prof. Uroš Cvelbar
4. Plasma-assisted wound treatment and topical introduction of molecules
Prof. Uroš Cvelbar
5. New generation of superior creep resistant steels with nanoparticles modified microstructure
Prof. Uroš Cvelbar
6. Interaction between fully dissociated moderately ionized ammonia plasma and glass-fiber reinforced polymers
Prof. Miran Mozetič
7. Nanostructures and related composites for detection of hazardous gaseous molecules
Prof. Uroš Cvelbar
8. Functionalization of polymer cardiovascular implants for optimal hemocompatibility
Asst. Prof. Alenka Vesel
9. Advanced hydrodesulphurisation with catalyst nanomaterials
Prof. Uroš Cvelbar
10. Advanced hemocompatible surfaces of vascular stents
Dr. Ita Junkar
11. Evaluation of the range of plasma parameters suitable for nanostructuring of polymers on industrial scale
Prof. Miran Mozetič
12. Selective plasma oxidation of FeCrAl alloys for extended-lifetime of glow plugs for diesel engines
Asst. Prof. Janez Kovač
13. Food for future - F4F
Asst. Prof. Alenka Vesel
14. Potential of biomass for development of advanced materials and bio-based products
Dr. Ita Junkar
15. Building blocks, tools and systems for the Factories of the Future – GOSTOP
Prof. Miran Mozetič
16. Strategic Research & Innovation Partnership Factories of the Future (SRIP FoF)
Prof. Miran Mozetič
17. Development of nanostructured biosensors for diagnosis/treatment of cancer and surfaces with antibacterial
Prof. Miran Mozetič
18. Novel type of antibacterial coatings on textile materials and plastics with controllable release of antibacterial agent
Prof. Uroš Cvelbar
19. Hydrogen Permeation Measurements on PVD-Coated Eurofer
Dr. Vincenc Nemanič
20. Tof-SIMS surface spectrometry
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1. Prof Dr Reinhard H. Schwarz, Institut Superior Tecnico, Lisbon, Portugal, 12–13 January 2017
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3. Dr Tomislava Vukušić, Faculty of food technology and biotechnology, Zagreb, Croatia, 23–28 January 2017
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7. Dr Christian Nöbauer, Technical University of Wien, Wien, Austria, 14–17 May 2017
8. Prof Dr Masaru Hori, University of Nagoya, Nagoya, Japan, 5–9 July 2017
9. Dr Kenji Ishikawa, University of Nagoya, Nagoya, Japan, 6–8 July 2017
10. Dr Petr Humpoliček, Tomas Bata University, Zlin, Czech Republic, 14–18 August 2017
11. Dr James Walsh, University of Liverpool, Liverpool, UK, 31 August–13 September 2017
12. Dr Danijela Vujošević, Institute for Public Health of Montenegro, Podgorica, Montenegro, 13–15 October 2017
13. Andreas Pelster, ION-TOF, Münster, Germany, 16–20 October 2017
14. Dr Oleg Baranov, National Aerospace University, Kharkiv, Ukraine, 4–31 December 2017

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BIBLIOGRAPHY

ORIGINAL ARTICLE

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