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 a ultra-high-vacuum environment, vacuum optoelectronics, and basic research in the field of surface and thin-film characterization by electron spectroscopy techniques.

Head:
Prof. Miran Mozetič

A highly dissociated cold plasma created by inductively coupled radiofrequency discharges was applied either as a source of neutral oxygen or nitrogen atoms, or as a powerful tool for the modification of solid materials. The plasma created in such electrode-less discharges is extremely non-equilibrium and thus suitable for the treatment of both inorganic and delicate organic samples. The research group developed a method for the suppression of tritium retention in current and future fusion reactors with carbon limiters or divertors. Important results towards the minimization of hydrogenated carbon-deposit formation were obtained in collaboration with research groups from Madrid, Spain, and Nieuwegein, The Netherlands. The application of reactive nitrogen particles created in electrode-less discharges effectively interact with hydrogenated carbon radicals in the gas phase, forming stable volatile molecules that do not stick to surfaces at room or elevated temperature and are thus easily pumped away from thermal plasma reactors. Furthermore, N and HN radicals that are formed in an ammonia plasma interact with the deposits already at room temperature, allowing for removal of the deposits.

A hypothesis has been launched to explain the rapid oxidation of metals in an aggressive oxygen plasma resulting in the spontaneous growth of single-crystalline metal oxide nanowires.

Plasma nanoscience is a new, attractive and rapidly expanding field of interdisciplinary research where the research group is extremely active. A specialized workshop was organized at Lake Bohinj in the Slovenian Alps under the supervision of the International Union for Vacuum Science, Techniques and Applications: 62nd IUVSTA Workshop on Plasma Synthesis and Modification of Nanomaterials, 14th – 18th June 2010. The workshop gathered worldwide-renowned scientists and represented one of the most important events in nanoscience. The research group applies oxygen plasma for the direct synthesis of metal oxide nanoparticles on metallic substrates. Since the original invention of the synthesis of niobium oxide nanowires published in Advanced Materials in 2005, the method has been expanded to a variety of metal-oxygen systems and the research group obtained important results on the way to explaining this unusual effect. Although an internationally acceptable theory on the growth of metal oxide nanoparticles under extremely non-equilibrium conditions has not yet been recognized, our recent results employing both theoretical and experimental approaches show possible mechanisms involved in this strange happening. Appropriate hypotheses were published in a new, high-quality international journal – Nanoscale – published by the Royal Society of Chemistry.

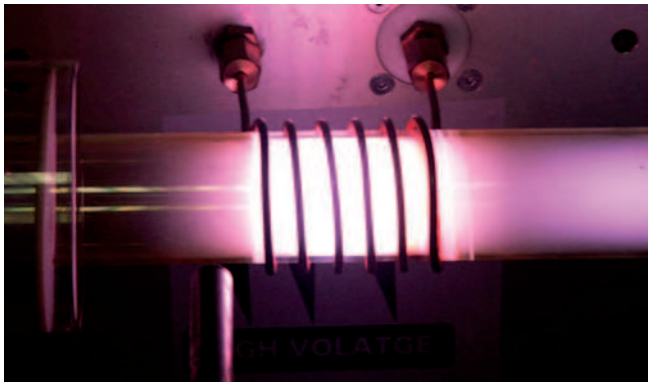

Figure 1: Low-pressure plasma created within a quartz-glass tube by inductively coupled radiofrequency discharge

Oxygen plasma created by powerful electrode-less discharges is too aggressive for the modification of organic materials, but serves as an excellent source of neutral oxygen atoms in the ground state. While most reactive plasma particles recombine on the walls of the vacuum system, the lifetime of the neutral O atoms is very long, providing the pressure is low enough to prevent gas-phase recombination during three-body collision events (in practice it means below a few hundred Pa) and as long as the walls are made of materials with a low coefficient for heterogeneous surface recombination. A late afterglow of plasma created at moderately low pressure and in glass systems is therefore a suitable source of neutral oxygen atoms at room temperature. Such atoms do not cause substantial modification of the bulk

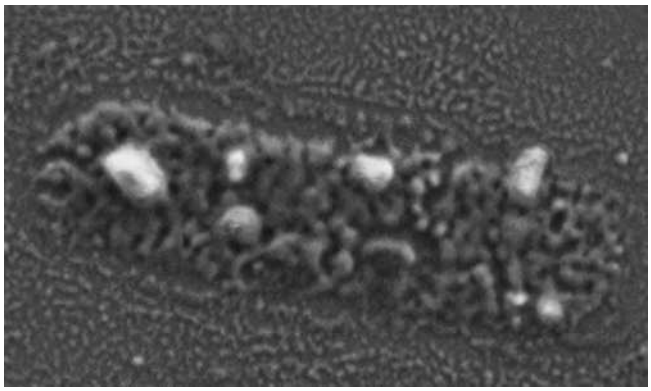

Figure 2: SEM image of bacillus stearothermophilus after treatment with oxygen atoms reveals the non-homogeneous structure of the bacterial cytoplasm.

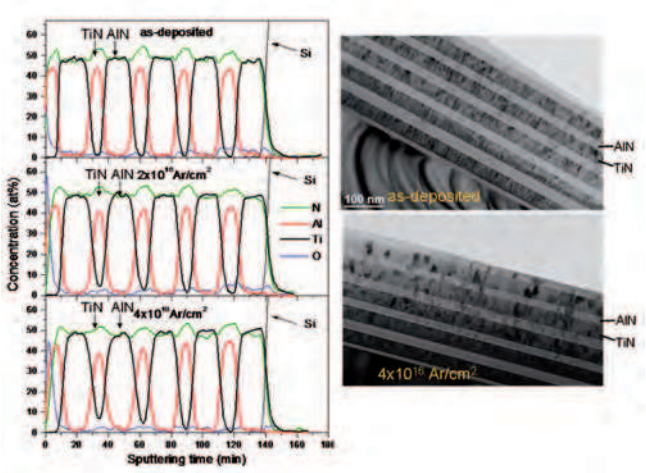

Figure 3: Very high ion radiation stability observed in the multilayered (AlN/TiN)x5/Si nanocomposites upon Ar ion irradiation at 200 keV. The AlN and TiN layers remain well separated, with sharp interfaces. XPS depth profiles are shown on left and TEM images on right. The work was performed in collaboration with the Institute for Nuclear Sciences Vinča, Serbia.

properties on organic materials, but do affect the surface properties. Numerous organic materials are treated by such an afterglow in order to obtain the required properties of polymer materials. The afterglow is also suitable for the slow and selective removal of organic materials from live biological cells, including bacteria. A treatment by neutral oxygen atoms at room temperature causes the removal of the outermost parts of the bacteria, revealing its internal structure.

The characterization of the surfaces and interfaces of bulk materials, layered structures and nanomaterials requires the application of modern surface-analytical techniques. X-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES) and atomic force microscopy (AFM) have been used successfully, all for basic research and for the characterization of technological samples. Our research group is worldwide recognized for the depth profiling of thin films and multilayers at a high depth resolution. Applying the XPS method we investigated the structural changes in multilayered AlN/TiN nanocomposites upon Ar ion irradiation in collaboration with the Institute for Nuclear Sciences from Vinča, Serbia. Reactive sputtering was used to deposit (AlN/TiN) × 5 multilayers on a Si substrate with a thickness of the individual layer equal to 27 nm. Argon was implanted at 200 keV at doses of 5×10^{15} to 4×10^{16} ions/cm^2. It was found that this immiscible system exhibits a high ion radiation stability, the AlN and TiN layers remaining well separated with sharp interfaces. Ion irradiation induced small local density changes and only a slight increase in the size of individual grains. Due to these small structural changes, ion irradiation enhanced the mechanical strength of the multilayered nanocomposites.

The existence of ferromagnetism at room temperature in the Zn–Mn–O semiconductor samples and its dependence on the preparation conditions were investigated. We systematically examined the samples with a manganese concentration ranging from 0 to 10 at.%, prepared by a solid-state reaction route. The XPS surface composition, chemical analysis and depth profiling were successfully employed on powder, revealing the chemical composition at the surface of the grains and underneath. The present investigation shows that the physical properties and the observed room-temperature ferromagnetism is due to grain surface effects. It seems that the ferromagnetic phase is correlated with an oxygen build up at the surface.

A method for the suppression of tritium retention has been elaborated in collaboration within the EURATOM association.

Important results were obtained in collaboration with 15 partners of the European project PlasmaNice – Atmospheric plasmas for nanoscale industrial surface processing – funded under the EU's7th FP. The main objective of the PlasmaNice project is to develop equipment and technology for the industrial in-line atmospheric plasma deposition of functional nanocoatings on various fibre- and polymer-based substrates for packaging. The project aims at improving the recyclability of conventional fossil-fuel-based plastics and/or their replacement by renewable, bio-based and biodegradable materials. Our research group performs accurate surface characterizations of plasma-deposited sol-gel coatings using XPS, AFM and SEM methods. We determined the correlation between the plasma process parameters, the degree of surface fuctionalization and the thickness of the deposited coatings. Our second task is the development of a new method for the fast and in-line monitoring of the plasma-coating deposition process at a very high velocity. We tested different optical, electrical and spectroscopic methods and identified the most suitable one.

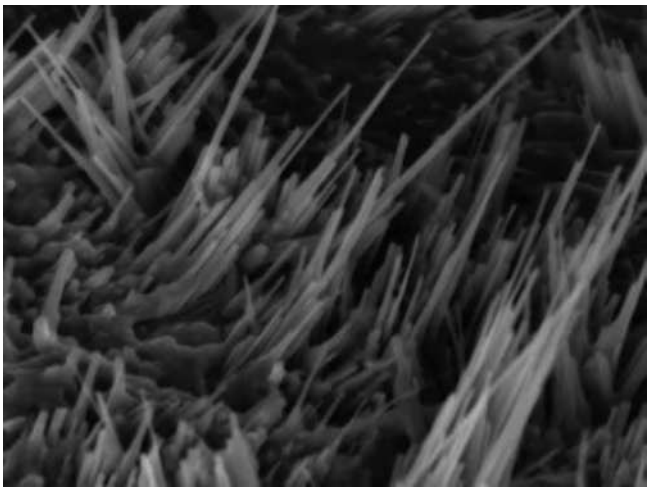

Figure 4: Single-crystalline nanowires of niobium pentoxide growing from the surface of polycrystalline niobium foil during exposure to a fully dissociated oxygen plasma.

An extremely sensitive method for the quantitative analysis of hydrogen in ultra-high-vacuum systems was developed. The system allows for an accurate quantitative analysis of gas compositions. We can determine the composition of a very small amount (10^{-10} mol) of gas which resides in the gas-accumulating vessel. Special attention was devoted to the suppression of the hydrogen background from the heated sample holder. This was achieved by a careful selection of materials and a pre-treatment procedure using an original construction. The quantitative analysis of the composition of a gas mixture using a mass spectrometer is the basic method giving a rough insight into the reactions in gases and on surfaces. Our improved setup was successfully applied in an investigation of the breakdown voltage drift with time in gas surge arresters. In the frame of Physics laboratory IV, the training

exercise on mass spectrometry of gas mixtures for students of physics at the Faculty of Mathematics and Physics, University of Ljubljana, was established.

Permeation measurements through coated Eurofer membranes were important activities related to an EU fusion project within Euratom and another EU project. It was found that 5-micrometer-thick TiAlN films (prepared at department F-3 at JSI) have the highest permeation barrier reduction factor (PRF) reported so far. In cooperation with Joanneum research centre in Leoben we found that silicon oxy-nitride and silicon nitride films can be prepared in the form that has also very high PRF, even at a thickness below 1 micrometer. No quantitative data on such films existed prior to our measurements. Beryllium and tungsten films on Eurofer were investigated in this context too. Both metals will be applied for the first wall in future large fusion reactors. Beryllium films (8 mm thick) were deposited in Dr. Cristian Lungu's lab at the National Institute for Laser, Plasma and Radiation Physics - NILPRP from Bucharest, Romania. We obtained results that were reproducible within relatively wide margins. However, they could not be compared to published data since these data do not exist. The unexpected kinetics could be well explained by our better model and respecting the fact that data on bulk beryllium are rather old and probably inaccurate. Similar conclusions could be drawn from experiments with tungsten films on Eurofer, although the reproducibility was much better than for beryllium. Tungsten films (10 mm thick) were deposited in dr. Cristian Ruset's lab at NILPRP, which also developed and successfully tested identical films in fusion reactors like JET.

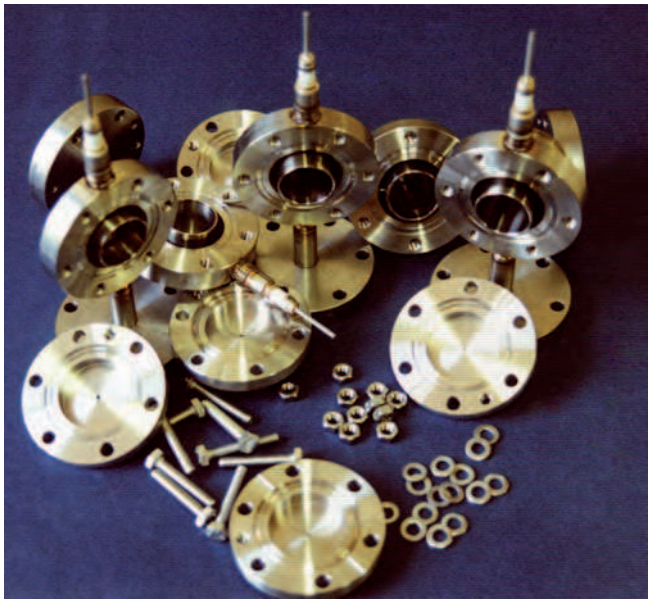

Figure 5: Ultra-high-vacuum pressure gauges in the development stage. The corresponding US patent has recently been granted.

The patent US7800376B2 entitled "Method and device for measuring ultrahigh vacuum" was granted in September 2010. The patent protects our original device for accurate pressure measurements in the range from 10^{-4} to 10^{-8} Pa.

Some outstanding publications in the past year

1. Francisco L. Tabarés, Jose Ferreira, G. van Rooij, J. Rapp, Aleksander Drenik, Miran Mozetič. Suppression of tritium retention in remote areas of ITER by nonperturbative reactive gas injection. Phys. rev. lett., 2010, vol. 105, no. 17, p. 175006-1-175006-4.
2. Kostya Ostrikov, Igor Levchenko, Uroš Cvelbar, Mahendra K. Sunkara, Miran Mozetič. From nucleation to nanowires : a single-step process in reactive plasmas. Nanoscale (Print), 2010, vol. 2, no. 10, p. 2012-2027.
3. Momir Milosavljević, Dalibor Peruško, Velimir Milinović, Zoran Stojanović, Janez Kovač, Chris Jeynes. Ion irradiation stability of multilayered AlN/TiN nanocomposites. J. phys., D, Appl. phys., 2010, vol. 43, no. 6, p. 065302-1-065302-6.

Patents granted

1. Method of treatment of biomedical polymeric prostheses for improvement of their antithrombogenous properties
   Ita Junkar, Miran Mozetič, Alenka Vesel, Uroš Cvelbar, Metka Krašna, Dragoslav Domanovič
   Patent No. SI 23021 (A)
2. Method and device for measuring ultrahigh vacuum
   Alenka Vesel, Miran Mozetič
   Patent No. US 7800376 (B2)

Organization of conferences, congress and meetings

1. 3rd International Conference on Advanced Plasma Technologies (ICAPT-III), Bohinj, Slovenia, 15.-18.6.2010

INTERNATIONAL PROJECTS

1. Atmospheric Plasmas for Nanoscale Industrial Surface Processing
   PlasmaNice
   7. FP
   EC; Dr. Johanna Lahti, Tampere University of Technology, Paper Converting and Packaging Technology, Tampere, Finland
   Asst. Prof. Janez Kovač
2. Removal of Deposits by Neutral Oxygen and Nitrogen Atoms - 1.4.2. – FU
   WP10-PWI-02-02/MHEST/PS; Detailed Characterization of Reaction Products from Removal of A-C:H with Mixed H2/N2 Plasmas
   EURATOM – MHEST
   7. FP, EURATOM, Slovenian Fusion Association – SFA
   3211-08-000102, FU07-CT-2007-00065
   EC; Republic of Slovenia, Ministry of Higher Education, Science and Technology, Ljubljana, Slovenia
   Prof. Miran Mozetič

3. Deuterium Retention and Release from Metal Surfaces - 1.4.4. – FU
   EURATOM – MHEST
   7. FP, EURATOM, Slovenian Fusion Association – SFA
   3211-08-000102, FU07-CT-2007-00065
   EC; Republic of Slovenia, Ministry of Higher Education, Science and Technology, Ljubljana, Slovenia
   Dr. Vincenc Nemanič
4. Plasma Sterilization and Decontamination of Water
   NATO CLG. REF.983580
   Dr. Fausto Pedrazziini, NATO - North Atlantic Treaty Organisation, Brussels, Belgium
   Asst. Prof. Uroš Cvelbar
5. Development of Bioactive Packaging
   BIOPACKAGING
   EUREKA
   Univerza v Mariboru, Inštitut za inženirske materiale in oblikovanje, Maribor, Slovenia
   Prof. Miran Mozetič
6. Vascular Graft Interfaces
   VaGrint
   MNT ERA NET, 3211-07-000024
   University of Maribor, Faculty of Mechanical Engineering, Maribor, Slovenia
   Prof. Miran Mozetič
7. Hydrogen Impermeable Nano-material Coatings for Steels
   Hy - Nano – IM
   MNT ERA NET
   Dr. Vincenc Nemanič, Dr. Paul McGuiness, Dr. Miha Čekada
8. Introduction Consulting to define Targets and Specify Methods; Measurement of Three Samples at Room Temperature
   Research Agreement
   Dr. Vincenc Nemanič
9. Characterization of Microorganism Structures by Advanced Analytical Techniques
   BI-ME/10-11-1
   Dr. Zoran Vratnica, Institute of Public Health, Podgorica, Montenegro
   Prof. Miran Mozetič
10. Sterilization of Medicine Materials with Gaseous Plasma
    BI-ME/10-11-3
    Dr. Danijela Vujošević, Institute of Public Health, Podgorica, Montenegro
    Asst. Prof. Uroš Cvelbar
11. Interaction of Highly Dissociated CO2 Plasma with Materials Suitable as Outer Protective Layers of Future Space Vehichles
    PROTEUS 2010 – 2011, BI-FR/10-11-PROTEUS-005
    Dr. Marianne Balat-Pichelin, Processes, Materials and Solar Energy Laboratory (PROMES-CNRS), Font-Romeu Odeillo, France
    Asst. Prof. Alenka Vesel
12. Improvement of Adhesive Properties of Biomedical Materials by Plasma Treatment
    BI-HR/10-11-020
    Dr. Morana Jaganjac, Ruđer Bošković Institute, Zagreb, Croatia
    Asst. Prof. Alenka Vesel
13. Modification of Cardiovascular Implants by Gaseous Plasma
    BI-HR/09-10-001
    Dr. Slobodan Milošević, Institut za fiziku - Institute of Physics, Zagreb, Croatia
    Prof. Miran Mozetič
14. Quantum Dots for Solar Cells
    BI-CN/09-11-003
    Dr. Xiaoxia Zhong, Shanghai Jiao Tong University, Shanghai, China
    Asst. Prof. Uroš Cvelbar
15. Study of Ion Mixing caused by FIB
    BI-HU/09-10-004
    Dr. Miklos Menyhard, Research Institute for Technical Physics and Materials Sciences, Budapest, Hungary
    Asst. Prof. Janez Kovač
16. Dissociation Kinetics in Technological Plasmas
    BI-SR/10-11-001
    Prof. Zoran Petrovič, Institute of Physics, Beograd - Zemun, Serbia
    Prof. Miran Mozetič
17. Thermoionic Energy Conversion
    BI-US/09-12-021
    Prof. Robert Nemanich, Arizona State University, (ASU), Tempe, Arizona, USA
    Dr. Vincenc Nemanič
18. Metal Oxide Nanowire/Nanotube Arrays for Electrochemical Energy Conversion Applications
    BI-US/08-10-030
    Prof. K. Mahendra Sukara, Oddelek za kemijsko inženirstvo, Univerza v Louisvillu, Louisville, KY, USA
    Asst. Prof. Uroš Cvelbar

R &D GRANTS AND CONTRACTS

1. Plasma Treatment of Vascular Grafts
   Prof. Miran Mozetič
2. Research and Development of Integrated Overvoltage Protection Devices Based on Gas Discharger Toward a Reliable Miniature Technical Solution
   Dr. Vincenc Nemanič
3. Study of Gaseous Deuterium Retention and Release from Metals Relevant to ITER
   Dr. Bojan Zajec
4. Investigation of Gaseous Diascharges for Introduction of New Enviromentally Friendly Technology for Functionalization of Semiproduct in Capacitor Production
   Prof. Miran Mozetič
5. Development of Treatments and Procedures for Improvement of Hemocompatibility of Polyethyleneterephtalate Surfaces
   Prof. Miran Mozetič
6. Synthesis and Functionalization of Composite Nanobeads for Early Diagnosis of Neurodegenerative Diseases
   Asst. Prof. Alenka Vesel
7. Superhydrophilicity of Surfaces and its Application in Technological Processes for Industrial Application
   Asst. Prof. Uroš Cvelbar
8. Printed Passive Electronic Components for Smart Packaging
   Asst. Prof. Alenka Vesel
9. Multifunctional Nanocomposite Coatings and Paints
   Asst. Prof. Janez Kovač, Asst. Prof. Alenka Vesel
10. Ignition and Eelf-extinguishing of arc in a Gas Surge Arrester at High Overvoltages
    Dr. Vincenc Nemanič

RESEARCH PROGRAMS

1. Vacuum Technique and Materials for Electronics
   Dr. Vincenc Nemanič
2. Thin Film Structures and Plasma Surface Engineering
   Prof. Miran Mozetič

MENTORING

Ph. D. Thesis

1. Ita Junkar, *Plasma treatment of polymers for biomedical applications* (mentor Miran Mozetič; co-mentor Uroš Cvelbar)

VISITORS FROM ABROAD

1. Dr. Slobodan Milošević, dr. Marijan Bišćan, Nikša Krstulović, Zlatko Kregar, Krešimir Salamon, Institute of Physics, Zagreb, Croatia, several times
2. Dr. Primož Eiselt, Plasmabull, Lebring, Austria, several times
3. Dr. Zoran Vratnica, dr. Danijela Vujošević, Institute of public health, Podgorica, Montenegro, several times
4. Dr. Nevena Puač, dr. Željka Nikitović, Institute of Physics, Belgrade, Serbia, several times
5. Dr. Momir Milosavljević, dr. Davor Peruško, Institute of nuclear sciences, Vinča, Belgrade, Serbia, 24. 5. – 28. 5 . 2010
6. Dr. Cristian Lungu, National Institute for Lasers, Bucharest, Romania, 13. 6. -15. 6. 2010
7. Dr. David Ruzic, University of Illinois, USA, 14. 6. – 20. 6. 2010
8. Prof. dr. Antony B. Murphy, CSIRO, Sydney, Australia, 14. 6. – 20. 6. 2010
9. Prof. dr. Giorgios Evangelakis, University of Ioannina, Ioannina, Greece, 14. 6. – 20. 6. 2010
10. Prof. dr. Tom Stara, J.P.Speed School of Engineering, University of Louisville, Louisville, Kentucky, USA, 14. 7. 2010 – 15. 7. 2010
11. Prof. Marian Lehocky and A/Prof. dr. Aleš Mraček, University Tomas Bata, Zlin, Czech Republic, 11. 11. 201 – 13. 11.2010
12. Dr. Iacono Jonathan, CNRS, Laboratory Promes, Odeillo, France, 7.12. 2010 – 14. 12.2010

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BIBLIOGRAPHY

Original Articles

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2. Andrew Das Arulsamy, Kristina Eleršič, Martina Modic, Uroš Cvelbar, Miran Mozetič, "Reversible carrier-type transitions in gas-sensing oxides and nanostructures", *ChemPhysChem*, vol. 11, no. 17, pp. 3704-3712, 2010.
3. Ahmad Asadinezhad, Igor Novák, Marián Lehocký, František Bílek, Alenka Vesel, Ita Junkar, Petr Sáha, Anton Popelka, "Polysaccharides coatings on medical-grade PVC: a probe into surface characteristics and the extent of bacterial adhesion", *Molecules (Basel)*, vol. 15, no. 2, pp. 1007-1027, 2010.
4. Ahmad Asadinezhad, Igor Novák, Marián Lehocký, Vladimír Sedlarik, Alenka Vesel, Ita Junkar, Petr Sáha, Ivan Chodák, Ivan Chodák, "A physicochemical approach to render antibacterial surfaces on plasma-treated medical-grade PVC: irgasan coating", *Plasma processes polym. (Print)*, vol. 7, no. 6, pp. 504-514, 2010.
5. Ahmad Asadinezhad, Igor Novák, Marián Lehocký, Vladimír Sedlarik, Alenka Vesel, Petr Sáha, Ivan Chodák, "An in vitro bacterial adhesion assessment of surface-modified medical-grade PVC", *Colloids surf., B Biointerfaces*, vol. 77, no. 2, pp. 246-256, 2010.
6. Marianne Balat-Pichelin, Marc Passarelli, Alenka Vesel, "Recombination of atomic oxygen on sintered zirconia at high temperature in non-equilibrium air plasma", *Mater. chem. phys.*, vol. 123, no. 1, pp. 40-46, 2010.
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10. Aleksander Drenik, Andrej Tomeljak, Miran Mozetič, Alenka Vesel, Dušan Babič, Marianne Balat-Pichelin, "Behaviour of neutral hydrogen atom density in the presence of a sample holder in a plasma reactor", In: *Proceedings of the 12th Joint Vacuum Conference, 10th European Vacuum Conference and 7th Annual Meeting of the German Vacuum Society (JVC-12/EVC-10/AMDVG-7), Balatonalmadi, Hungary, 22 - 26 September 2008*, (Vacuum, vol. 84, no. 1), Sándor Bohátka, ed., Béla Pécz, ed., András Berkó, ed., Oxford, New York, Pergamon Press, 2010, pp. 90-93.
11. Kristina Eleršič, Ita Junkar, Aleš Špes, Nina Hauptman, Marta Klanjšek Gunde, Alenka Vesel, "Degradation of bacteria Escherichia coli by treatment with Ar ion beam and neutral oxygen atoms", *Mater. tehnol.*, vol. 44, no. 3, pp. 153-156, 2010.
12. Kristina Eleršič, Janez Kovač, Martina Modic, Miran Mozetič, "ARXPS-analiza bakterij Escherichia coli, obdelanih v kisikovi plazmi", *Vakuumist*, vol. 30, no. 2, pp. 4-7, 2010.
13. Matjaž Finšgar, Janez Kovač, Ingrid Milošev, "Surface analysis of 1-hydroxybenzotriazole and benzotriazole adsorbed on Cu by X-ray photoelectron spectroscopy", *J. Electrochem. Soc.*, vol. 157, no. 2, pp. C52-C60, 2010.
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16. Marija Gorenšek, Marija Gorjanc, Vili Bukošek, Janez Kovač, Zoran Lj. Petrović, Nevena Puač, "Functionalization of polyester fabric by Ar/N2 plasma and silver", *Tex. res. j.*, vol. 80, no. 16, pp. 1633-1642, 2010.
17. Marija Gorenšek, Marija Gorjanc, Janez Kovač, "Preiskava kemijskih sprememb na površini PET pletiva z rentgensko fotoelektronsko spektroskopijo po obdelavi s korona plazmo in po staranju pletiva", *Tekstilec*, vol. 53, no. 4/6, pp. 103-112, 2010.
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19. Marija Gorjanc, Vili Bukošek, Marija Gorenšek, Alenka Vesel, "The influence of water vapor plasma treatment on specific properties of bleached and mercerized cotton fabric", *Tex. res. j.*, vol. 80, no. 6, pp. 557-567, 2010.
20. Marija Gorjanc, Janez Kovač, Marija Gorenšek, "Rentgenska fotoelektronska spektroskopija za določanje kemijskih sprememb na površini bombaža po obdelavi s korona in nizkotlačno plazmo", *Tekstilec*, vol. 53, no. 7/9, pp. 194-204, 2010.
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