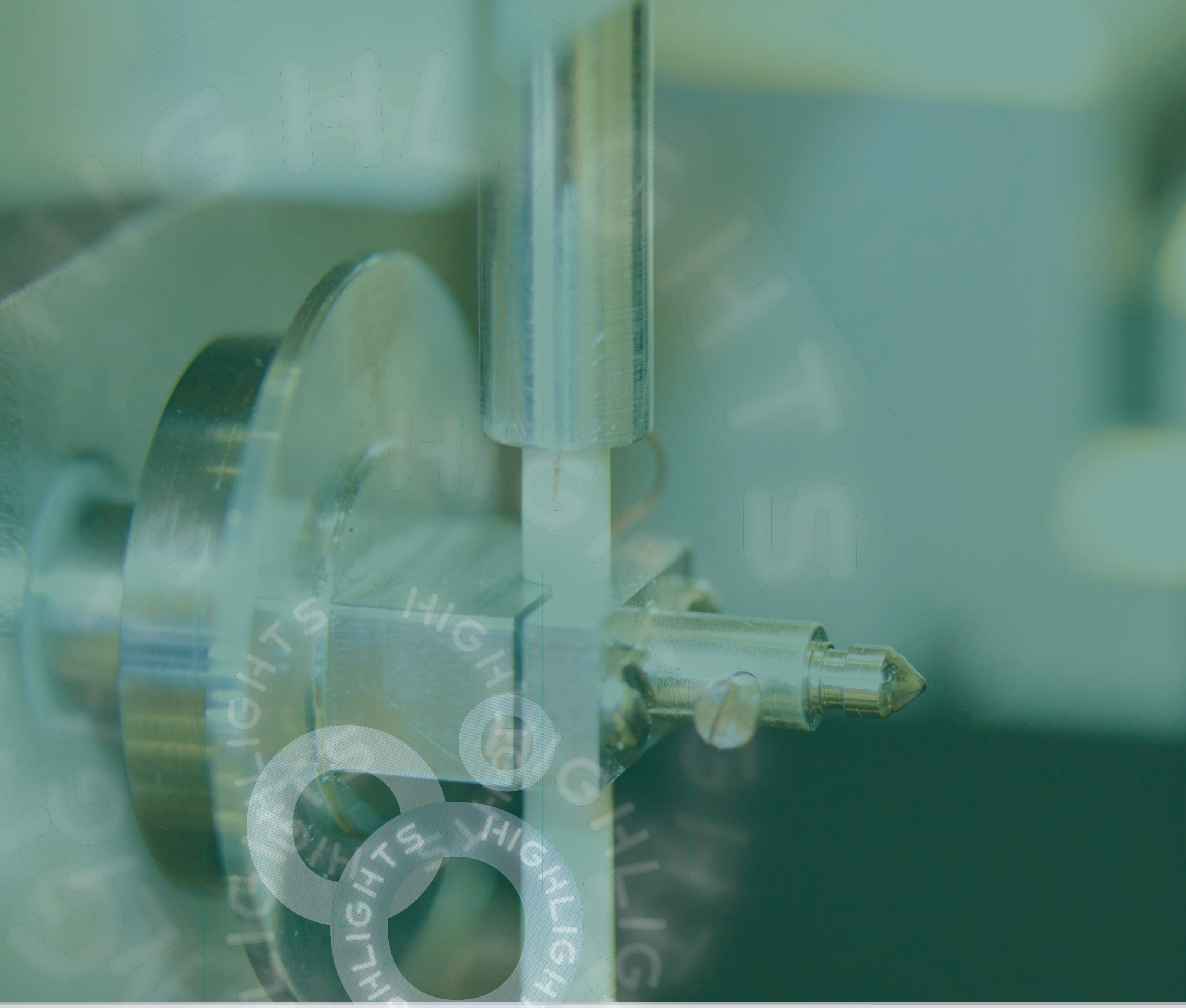

DEPARTMENT

OF MATERIALS PHYSICS

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DEPARTMENT
OF MATERIALS PHYSICS

In 2014 the research activities of the MPD were concentrated on studies of materials expected to be used in a nuclear environment and on the use of nuclear techniques for modification and analysis of solids. The MRL laboratory of MPD is the only facility in Poland disposing of the equipment needed to perform analyses of construction materials irradiated in nuclear reactors. The MRL has the Certificate of Testing Laboratory Accreditation No. AB 025. The Laboratory has also been granted 2nd Degree Approval No LB-038/27 by the Office of Technical Inspection. It also has the License of the National Radiological Protection and Nuclear Safety Department Nr. 1/93/"MET" for investigation of irradiated materials up to 100Ci. The laboratory was designed for testing surveillance specimens from a planned nuclear power plant. The hot laboratory consists of an assembly of 12 lead hot cells arranged in a single line. All cells are designed to handle 3700 GBq (100Ci) of 1 MeV gamma emitter. Each of the cells is equipped with a viewing window and master-slave or tong manipulators. The hot cells are connected by a special inert transport system. The assembly of hot cells is equipped with ventilating and active waste systems.

In 2014 the laboratory of nuclear microanalysis working mainly on Rutherford Basckscattering Spectroscopy was moved from the Pure Research Department to the Plasma and Ion technology Division (FM2) of the MPD.

Among the main research topics carried out in the MPD one may list:

- X-ray diffraction: structure of safe antidepressive alkaloid aptazepine obtained in first enantioselective synthesis and topography investigations of crystal lattice defects in ferroelectric niobates with tungsten bronze structure.
- Neutron scattering: magnetic and atomic short range order in $Mn_{0.3}Ni_{0.3}Cu_{0.4}$ pseudo-binary alloy studied with neutron elastic scattering, studies of the drying process.
- Mechanical properties: studies of strength and hardness of materials used in nuclear engineering, analysis of the role of irradiation on the functional properties of elastomers. Study of the correlation between structural and mechanical properties of the interface between the oxide scale and the bulk.
- Corrosion properties: studies of zirconium corrosion in nuclear reactors, modification of oxidation resistance using plasma or ion-beam doping of steels.
- Doping stainless steel with oxygen reactive elements like Rare Earth Elements (REE) and others for improving surface oxidation resistance at high temperatures.
- Development of new ferromagnetic semiconductors for spintronics.
- Studies of the dependence of specific features of plasma surface engineering methods on the layer structure.
- Optimisation of thin film Pb photocathodes.
- Development of Monte Carlo simulation procedures for channelled ions.
- Study of ZnO semiconductor and the influence of radiation damage on structural properties of zinc oxide.

In 2014 the high temperature option in the nanoindentation tester NanoTest "Vantage" was optimized. In consequence the system is able to perform measurements up to 700^{o}C.

In 2014 the biggest project conducted in MPD was the 4Labs project worth about 40 mln zlotys. The construction of Neutral Beam Injector system elements for the W7-X stellarator at IPP Greifswald was finished (Polish in-kind contribution to the W7-X project). Total value of these projects exceeds 58 mln PLN. Among smaller projects accepted one may list ion-irradiation of advanced graphene-elastomer composites (PBS III Grafel project). Two projects were submitted to the European Commission: BRILLIANT and VINCO. In the latter project NCBJ plays the role of coordinator of the whole consortium.

In 2014 researchers of MPB published 65 scientific publications and presented 71 presentations at scientific conferences.

Jacek Jagielski

CHEMICAL ASPECTS OF PRIMARY IONIZATION MECHANISMS IN MATRIX-ASSISTED LASER DESORPTION IONIZATION

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Up to the present, several mechanisms have been proposed in order to explain the processes actually occurring in MALDI ionization [1–5]. There is a common agreement in invoking a multi-step mechanism in which matrix ions are firstly produced by laser irradiation. However, this view exhibits some weak points: typically, the photon energy for commercial MALDI lasers is 3.5 eV or 3.7 eV (for tripled neodymium yttrium aluminum garnet (Nd:YAG) and nitrogen (N2) lasers, respectively) while matrix ionization energies are in the range 7–10 eV. Therefore, we proposed that the primary ionization mechanism occurring in matrix-assisted laser desorption ionization (MALDI) experiments originates from the presence of matrix dimers in the solid-state matrix-analytes sample. These species are formed by the interaction of carboxylic groups present in the matrix molecules with the formation of strong hydrogen bonds. Theoretical calculations proved that the laser irradiation of these structures leads to one or two H-bridge cleavages (Fig. 1), giving rise to an "open" dimer structure or to disproportionation with the formation of MH+ and $[M–H]^-$ species. The ions thus formed can be considered highly effective in their reactions with analyte ions, leading to their protonation (or deprotonation). To obtain further evidence for these proposals, in the present study the energetics of the reactions of ions from different aromatic carboxylic acids with two amino acids: glycine (see Fig. 2) and lysine and three multipeptides (gly-gly, gly-gly-gly and gly-gly-gly-gly) was investigated. The lowest ΔG values were obtained for 2,5-dihydroxybenzoic acid, widely employed as the MALDI matrix. Also, for p nitrobenzoic acid the reaction is slightly exothermic, while for the other aromatic carboxylic acid derivatives positive values of ΔG are present.

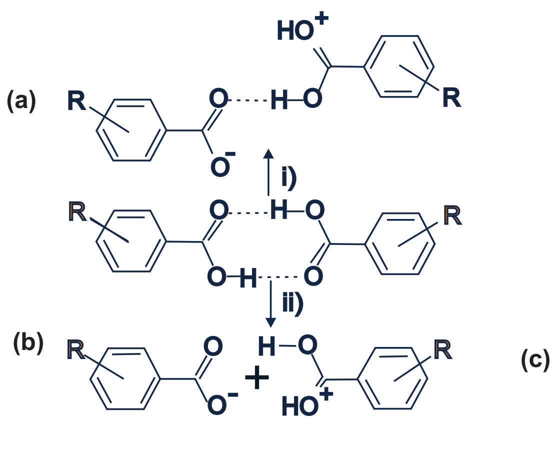

Fig. 1. Dimers of MALDI matrices leading to the formation of species (a) (one photon required) and (c) (two photons required).

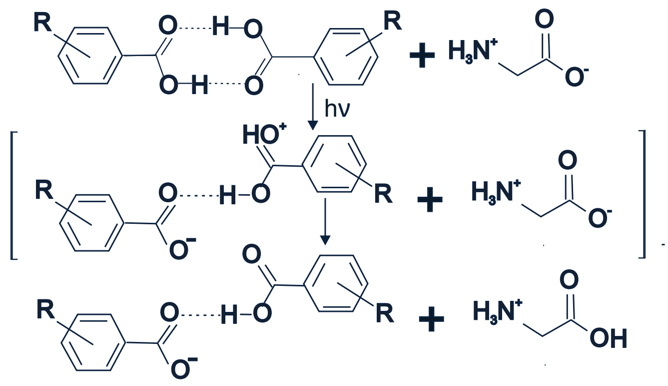

Fig. 2. General reaction among glycine amino acid and a carboxylic acid as matrix.

The experiments performed confirmed the theoretical findings. The "open" form (a) of matrix dimer is a highly reactive species that well justifies the MALDI data, without the need to invoke the pre-existence of ions in the matrix-analyte solid state sample [4] or the occurrence of electronic excitation energy followed by pooling phenomena [5]. Detailed results were published in [6].

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NEUTRON RADIOGRAPHY STUDIES OF ANCIENT OBJECTS FROM POLAND

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Thermal neutron radiography and computed tomography (CT) have become important techniques applied in cultural heritage research. The neutron imaging gets its merits from the small attenuation of neutrons by most common materials like ceramics, bronze and iron. The strong scattering of thermal neutrons on hydrogen nuclei provides another advantage in revealing regions containing organic components.

Our studies were performed on several objects found at the Przeworsk culture burial site at Czersk (central Poland). The main experimental tool was the thermal neutron (white beam) radiography facility at the nuclear research reactor MARIA of NCBJ. The study dealt with a few clearly identified objects like the metal parts of an ancient shield and a burial urn as well as a few initially unidentifiable aggregates of many different objects found at the excavation site. The neutron imaging helped in identification of their components.

One of the heavily corroded aggregates of unidentifiable objects from Czersk drew our particular attention due to its unrecognizable appearance (Fig. 1 a). The object was carefully studied with neutron CT. The 3-D representation of the aggregate was obtained from about 400 tomographic neutron projections. We found that the neutron representation of the external surface of the object revealed more detail of the item`s components than optical pictures (Fig.1 a, b).

More elaborate analysis was performed with segmentation of the object representation into regions of different neutron absorption strength. Three kinds of regions were distinguished corresponding to high, medium and low neutron absorption strength. Application of segmentation procedure revealed some ring shaped structures composed of highly neutron absorbing materials inside the body of the artefact (Fig.1 c). The revealed regions containing the materials of less neutron absorbing power formed some sharp conic structures (Fig 1. d). In effect we proved that an inspection of the neutron images could reveal spear or arrow heads, clip and spur parts clumped together inside the object.

(a) *(b)* *(c)* *(d)*

Fig.1. The pictures of a heavily corroded aggregate from the Czersk excavation site. (a) photo, (b), neutron CT reconstruction of the external surface of the artefact, (c) the most neutron absorbing parts of the items marked in red, (d) the less neutron absorbing parts marked in blue.

ADVANCES IN IMPROVEMENT OF LEAD LAYER-NIOBIUM ADHESION

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The National Centre for Nuclear Research conducted research on thin lead layers for superconducting accelerator photocathodes.

The studies were carried out with the use of fast lead deposition in an arc system connected with subsequent layer melting and recrystallization by high intensity pulsed plasma beams (HIPPB) [1].

The present research was performed to improve adhesion between the layers of lead and the niobium substrate. Creating a connection between lead and niobium is a challenging task, because lead and niobium have different physical and chemical properties (e.g. melting point, boiling point). We decided to use a Rod Plasma Injector (RPI) to resolve the problem of insufficient adhesion (deposition of a Pb layer on a Nb substrate directly or with an additional layer). The RPI generates plasma pulses with energy density from 1 to 8 J/cm^2 at a typical pulse duration of 1 µs. It was estimated using the Energy Transport in Laser Irradiated Targets (ETLIT) code [2], that to melt the niobium surface an ion pulse energy density higher than 4 J/cm^2 is needed (Fig. 1).

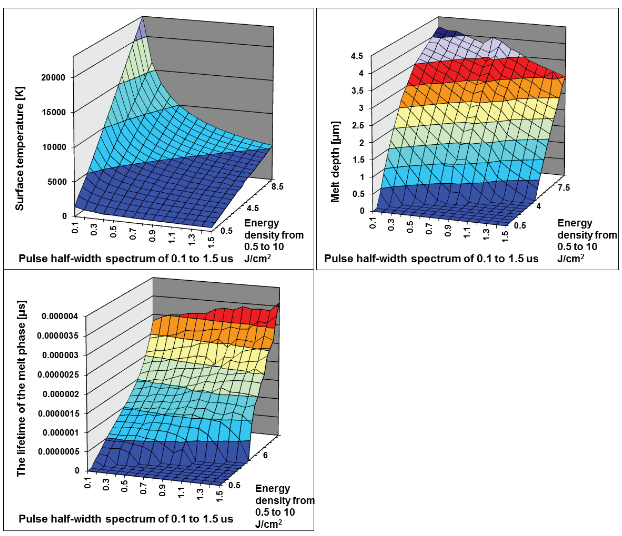

Fig. 1. Surface temperature, melt depth, lifetime of the melt phase as a functions of the pulse energy density and the pulse half-width, the pulse half-width spectrum from 0.1 to 1.5 ms and the energy density from 0.5 to 10 J / cm^2.

Thermodynamic calculations have shown that niobium and lead tend to oxidize. For this reason, nonreactive argon was used as a working gas.

The RPI generator can work in two main regimes, i.e. Pulse Implantation Doping (PID) and Deposition by Pulse Erosion (DPE) [3]. Both (PID and DPE) regimes were used in the research; PID for melting and cleaning the surface and to neutralize the oxygen layer on the niobium surface and DPE for forming layers of lead, titanium or tin.
5-60 ion pulses with 1-7 J/cm^2 energy density were applied for modification of the Nb substrate.

The effects of our investigations were studied using Scanning Electron Microscopy (SEM) and Energy Dispersion Spectroscopy (EDS).

Fig. 2 shows the main results of scanning observations of Pb and Ti layers deposited on a Nb substrate, using 5, 15 and 40 plasma pulses. It can be seen that, in all cases the substrate was melted. In contrast to Pb the Ti layer is continuous, smoother and homogeneous, without pores and drops. At this moment, the improvement of the adhesion of Pb on Nb is better using an additional transition Ti layer (as opposed to direct deposition of Pb).

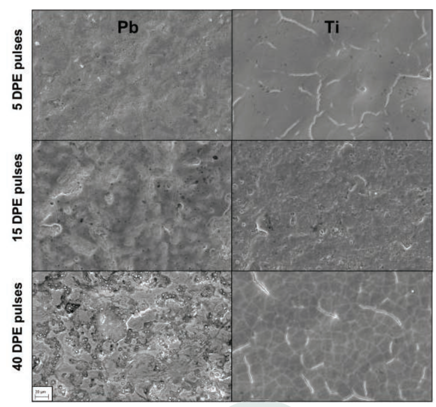

Fig. 2. Pb and Ti layers deposited on a Nb substrate using 5 plasma pulses with 5 J/cm^2 energy density, 15 plasma pulses with 5 J/cm^2, and 40 plasma pulses with energy density decreasing from 7 J/cm^2 to 1 J/cm^2.

In future we plan to deposit a Pb layer on an additional Ti and Sn layer, and to optimize the modification process.

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CHARACTERIZATION OF PULSED PLASMA GENERATED UNDER GAS INJECTION DURING THE IMPULSE PLASMA DEPOSITION METHOD

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We focus on the pulsed plasma generated in the modified Impulse Plasma Deposition (IPD) method [1,2], in which the main attention was paid to the role of pulsed changeable concentration of the working gas particles during the synthesis process. It is known that decrease of pressure contributes to increase of energy of plasma particles. The standard way of gas delivery by continuous flow through the synthesis environment leads to energy dissipation of plasma species, as a result of inelastic collisions with particles of neutral gas. Combining use of an impulse valve, dosing the gas directly into the discharge region, and a high vacuum pump system capable of fast evacuation of pulsed gas portions, reduces the dissipation of plasma species energy on impacts with the „cold" gas and provides optimum conditions for igniting the electric discharge. The concept of applying pulsed changeable concentration of the plasma formative (working) gas was reposted in one of our previous publications [3]. We used the modified IPD method in the synthesis of TiN layers on cutting tools. The results obtained showed a significant increase in anti-abrasive properties, a 16-fold prolongation of the tool life time. Additionally, our late at spectroscopic studies of this plasma generated under modified gas delivery conditions showed the effects of effective sputtering of the substrate material exposed to the pulsed plasma streams [3]. We supposed that these results were an effect of carrying out the synthesis under the modified conditions of gas delivery using pulse changeable gas concentration which affects the state of the plasma.

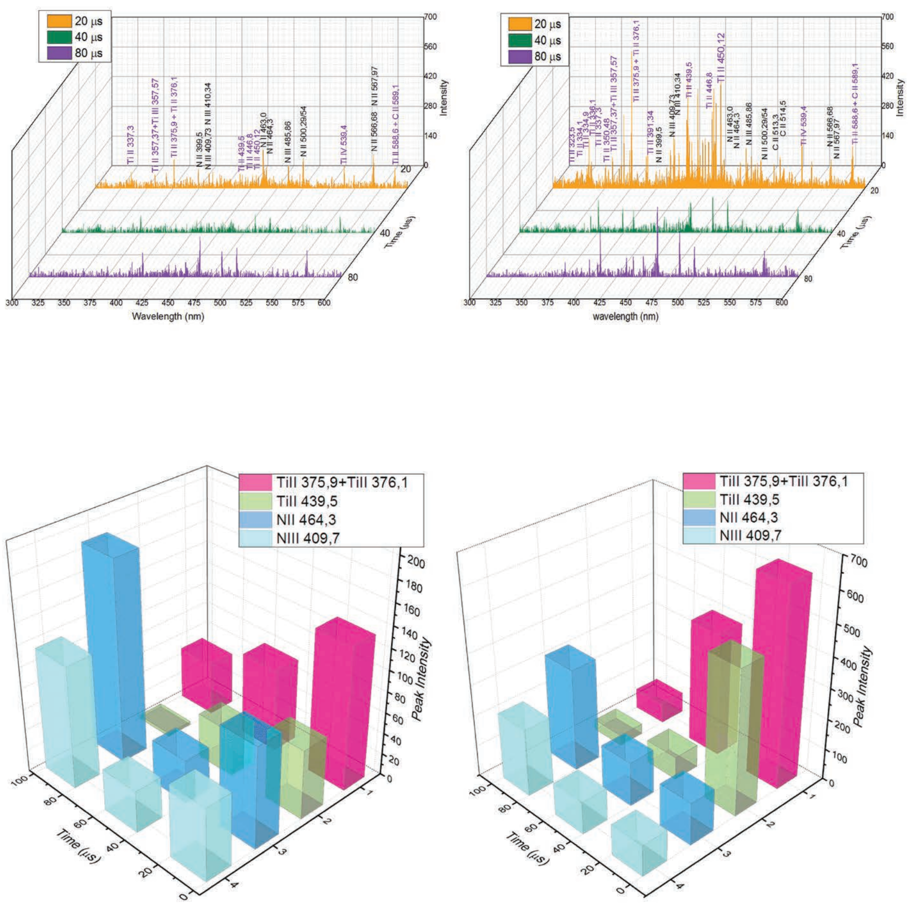

Fig. 1. Time resolved distribution of pulsed plasma for a 3 kV discharge (left graph) and for a 4 kV discharge (right graph). Time resolved distribution of chosen spectral lines (bottom).

Spectroscopic studies of pulsed plasma using the modified IPD method showed a two zone structure of spreading plasmoids. In the case of the products of erosion of electrode material, the intensity of titanium ions is greater in the early stages of plasma spreading (the spectrum is dominated by titanium ions). In the case of products of working gas ionization (nitrogen), the intensity and quantity increases with time. This means that the species originating from working gas ionization ("gaseous" plasma) follow the electrode erosion products ("metallic" plasma). The results obtained, are in contradiction to existing knowledge of the IPD method operating under standard conditions of continuous gas flow, where "metallic" plasma follows „gaseous" plasma. The time resolved structures of the pulsed plasma generated in the modified IPD method are shown in Figure 1.

Computational modelling of discharges within the IPD accelerator [4] reveal qualitative difference between the dynamics of plasmoids accelerated under standard conditions (constant concentration of gas particles during the synthesis process - standard IPD method [1]), and the dynamics of plasmoids accelerated under pulsed gas injection (pulsed changeable concentration of the working gas particles during the synthesis process - modified IPD method [2,3]). The impulse plasma pulse generated and spread in the dynamic conditions of gas dosing is more energetic, compared to the impulse plasma generated under the „standard" conditions. In the case of plasma that is generated under standard conditions, the electron temperature reaches a value of 2-3 eV, while in the case of a simulation under variable gas concentration conditions, up to 15 eV, near to the values from the experiments (Figure 2).

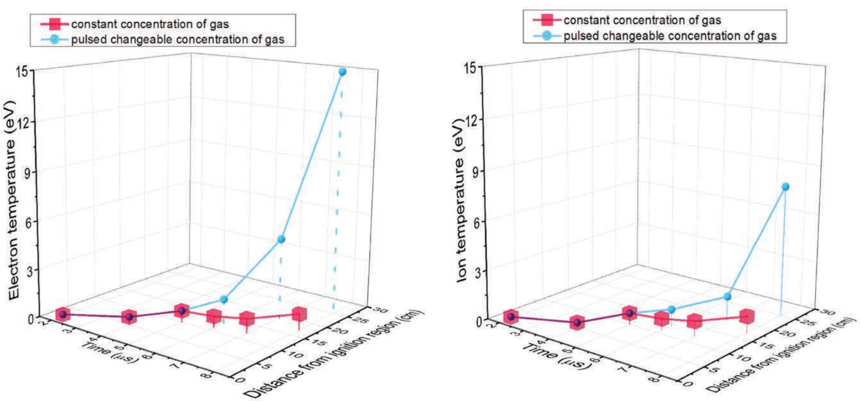

Fig. 2. Electron and ion temperatures of plasma generated in the dynamic conditions of the gas dosing and under the „standard" condi-tions.

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MOLECULAR DYNAMICS SIMULATIONS OF DEFECT TRANSFORMATIONS AT VARIOUS STRESS LEVELS

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Molecular Dynamics offers a huge potential in the analysis of atomic displacements on the nanometer and femtosecond scale. However, the analysis is usually performed on model systems with only limited experimental validation. The main objective of this collaboration project established between NCBJ and CEA is to perform MD simulations in conditions that mimic real experiments, especially the strain evolution that triggers defect transformation from point defects to dislocation and dislocation loops.

The problem of defect transformations in irradiated materials constitutes one of the main axes of research on materials expected to be used in a radiative environment. Numerous experimental works have permitted a complete picture composed of quantitative analysis of damage level (mainly using the Rutherford Backscattering/Channelling (RBS/C) method) to be drawn and a detailed description of defect structure (essentially by Transmission Electron Microscopy (TEM)). Several reviews of the experiments have been published, see e.g. Refs. 1 and 2. The experimental results were analyzed in the framework of the Multi Step Damage Accumulation (MSDA) model [3]. The next step of the analysis is to obtain deeper insight into the mechanisms responsible for defect formation and transformation. This can be done using Molecular Dynamics simulations, allowing one to reproduce atomic transformations occurring in displacement cascades produced by atomic collisions. The main objective of the project is to study the role of strain related to the formation of a free volume in irradiated material on defect transformations. Magnesia (MgO) has been selected as a test case for the analysis. MD simulations were performed using the LAMMPS computer code installed at the CIŚ computer centre in Swierk. Simulations of displacement cascades created by Primary Knock-on Atoms (PKA) of various energies and various directions were performed at increasing stress levels varying from 0 to 260 GPa. In parallel, calculations of the system enthalpy were performed for virgin and defected structures, in the latter case both point defects and dislocations were taken into account. The most notable result is the determination of a threshold stress level at which the structure containing point defects becomes less energetically favourable than the structure containing dislocations. The mean enthalpy change per atom in a structure containing point defects compared to a structure contained dislocations is shown in Fig.1.

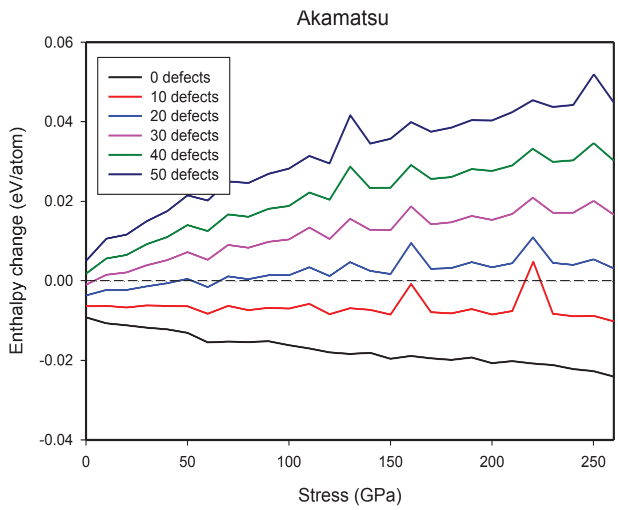

Fig.1. Variation of mean enthalpy per atom in MgO structure containing point defects as a function of the number of defects and stress level. Positive values mean that point defects are less energetically favourable than dislocations.

The results obtained confirmed the experimentally observed destabilization of point defect clusters and stress-triggered transformation of defects into dislocations and dislocation loops. Further analysis is in progress.

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INVESTIGATIONS OF THE RADIATION RESISTANCE OF STRUCTURAL MATERIALS DESTINED FOR FUSION REACTORS

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Tungsten, due to its properties, is proposed for use as a structural material in fusion reactors. Being in direct contact with thermal plasma and intensive radiation from the stream of neutrons it must withstand great heat loads.

For the experiments a thin tungsten foil was used. The inpurity content was determined using X-ray spectrometry: <2,5ppm Fe, <4,9ppm Cu.
Firstly, a microscopic investigation was carried out. A close-grained banded structure with small intermetallic inclusions, probable of oxides, was observed (Fig.1).

Fig.1. The microstructure of the tungsten foil.

The mechanical properties of the tungsten foil were studied using an Instron 8501 system with miniature specimens: gauge length l_0=12mm, width b_0=5mm and thickness a_0=0,22mm. The tensile test showed: R_m=1800MPa, $R_{t0,2}$=1700MPa and A=1,5%.

The hardness of the foil was measured by the Vickers method using a Wolpert Dia-Testor 7524 as 470HV30.
The mechanical properties of the foil were also studied using small punch tests (SPT). This method was selected because of the specimen dimensions connected with the small place in the reactor and in the plasma accelerator.

The tests were performed using special equipment on the Instron 8501 system, as the effect of the test the force –deflection curve of the specimen is obtained. In the work samples with a diameter of 3mm of tungsten metal sheet of nominal thickness about 0,22mm were inspected. The determined coefficients were:

$$P_y/h^2=3,06kN/mm^2 \equiv f(R_{t0,2})$$

$$P_m/h^2=7,81kN/mm^2 \equiv f(R_m)$$

The small specimens for SPT tests were irradiated in the MARIA reactor and exposed to a high-energy deuterium plasma, generated by a PF 1000 gun. Some specimens were also irradiated in the accelerator with argon ions. Further SPT tests were performed. The conditions of the experiments as well as the results of the SPT tests are given in Table 1.

Table 1. Results of SPT tests

Material	Number of plasma shocks	F_y/h^2		F_m/h^2	
		Mean value	Standard deviation	Mean value	Standard deviation
Initial state	-	3,0610	0,4494	7,8134	0,9374
Plasma interaction	3 times - $0,11 \cdot 10^{11}$	1,9372	0,7151	3,1411	1,2619
	3 times - $0,22 \cdot 10^{11}$	1,5288	0,5296	2,2953	0,1682
	3 times - $0,95 \cdot 10^{11}$	1,3959	0,3494	2,2509	0,2668
	14 times - $1,30 \cdot 10^{11}$	1,1652	0,3180	1,4030	0,5327
After Ar ion irradiation		1,8717	0,4546	2,8359	0,3910
After plasma shocks 0,22 10^{11}n. and Ar ion irradiation		1,1790	0,3809	1,8764	0,2755
After irradiation with neutrons in the MARIA reactor $6x10^{20}$ n_{th}/cm^2 $5x10^{19}$ n_f/cm^2		1,8140	0,2134	2,0316	0,1688

The force – deflection curves are shown in Fig.2.

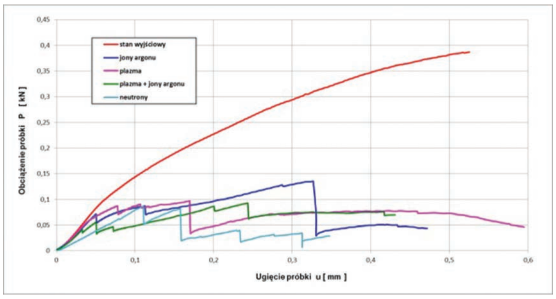

Fig.2. The force – deflection curves for different material states.

In addition, fractographic observations of the fracture surfaces of all specimens were performed (Fig.3).

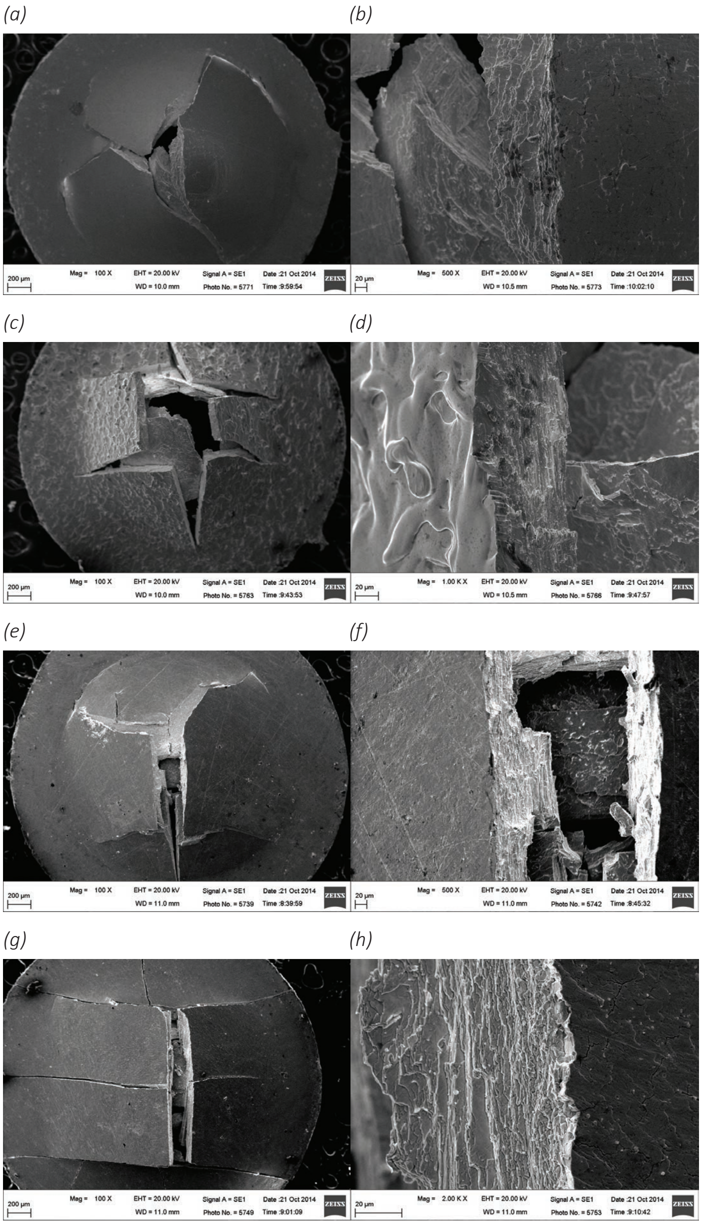

Fig.3. Fracture surfaces – SEM-photos; a, b/ initial state, c, d/ after plasma acction, e, f/ after Ar ion irradiation, g, h/ after plasma acction and Ar ion irradiation.

Summary

The structure of the tungsten foil was characteristic of a high degree of cold work. The material had a high tensile strength and high yield point, while the elongation was very low.

Irradiation in the reactor with neutron flux and with argon ions induced degrees of mechanical properties of tungsten foil under investigation. The same effect was observed after plasma action.

<u>The most important conclusion is:</u>

The SPT tests are useful for the investigation of the irradiation influence on the mechanical properties of metals.
The experiments showed that the effect of irradiation with ions is a good simulation of radiation defects.

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