STATUS OF EXPERIMENTS ON SURFACE MODIFICATION OF MATERIALS ON THE ACCELERATOR HIP-1

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Abstract

Ion implantation is an effective method for materials surface modification for various technological applications. The most common examples of its use are an increase of the durability, corrosion resistance, heat resistance for various industrial steels and special alloys for applications in biology and medicine, strengthening and changes in the morphology of the surface layers of polymers. Work in this direction is underway at the accelerator HIP-1 in ITEP. To provide the experiments, the beams of iron, vanadium and titanium generated by vacuum-arc metal ion source, as well as ion beams of nitrogen generated by duoplasmatron ion source are used. Several sets of experiments for the modification of the surface features were carried out. The transmission electron microscopy (TEM) and tomographic atom-probe microscopy (TAP) were used for samples analysis after ion beam treatment.

INTRODUCTION

Ion implantation is widely used as a method for modification of materials in order to improve their physical and chemical parameters. The ions used can be divided into light ones (typically nitrogen, carbon, oxygen and boron) and heavy ones (chrome, titanium, and tungsten). For biomedical applications, it is effective to use silver ions, copper and other elements to improve the bactericidal properties of steels, titanium alloys, CoCr alloys without losing their strength and corrosion resistance [1]. To improve the wear resistance and durability of industrial steels, implantation of nitrogen ions is used widespread [2, 3]. It is shown that the hardening depends not only on the dose of implanted nitrogen, but also on the chemical composition of the material to be modified and formed as a result of nitride complexes [4]. Furthermore, implantation of high doses of carbon (about 8×10^{17} ions / cm ^{2}) allows achieving a significant increase in durability by forming a carbon layer on the product surface [5]. Improvement of the corrosion resistance can be obtained by implantation of chromium and rare earth metals ions [6]. Since the implantation of different chemical element ions into material can significantly modify the properties of their structure in sub-surface layers, the use of composite beams may be the best technology in the preparation of materials for specialized applications.

To provide experimental activity, the beams of iron, vanadium and titanium generated by vacuum-arc metal ion source, and beams of nitrogen generated duoplasmatron were accelerated in the heavy ion HIP-1 (Heavy Ion Prototype). Several sets of experiments for the modification of the surface features were carried out. The results of the first experiments of surface modification by ion beams are presented.

FACILITY AND EXPERIMENT

The scheme of RFQ HIP-1 is shown on Fig.1. The accelerator assembly consists of the 100 kV terminal (1), low energy beam transport (LEBT) line with two electrostatic Einzel lenses (3) and diagnostic chamber (2), 27 MHz RFQ section and channel with 3 magnetic quadruple lenses (L1,L2,L3) and diagnostic station (5) at the output of the accelerator.

The accelerator allows the two types of experiments: at "low energy" when the irradiation of samples is carried out at the output of the injector inside the diagnostic chamber A (fig.1. pos.2) for further study by transmission electron microscopy (TEM) and tomographic atomprobe microscopy (TAP). Another type is the samples irradiation by "high energy" beams at the output of the accelerator (fig.1. pos. 5) for the further samples testing by transmission electron microscopy [7].

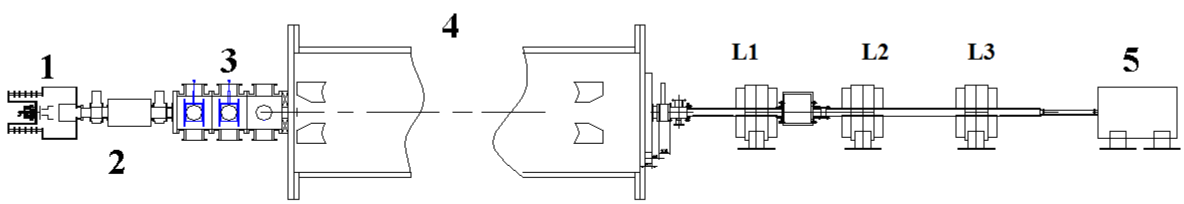

Figure 1: ITEP RFQ HIP-1. 1 - injector, 2 - diagnostic chamber A, 3 - electrostatic lens, 4 - RFQ, 5-diagnostic chamber B, L1,L2, L3 –quadruple lenses.

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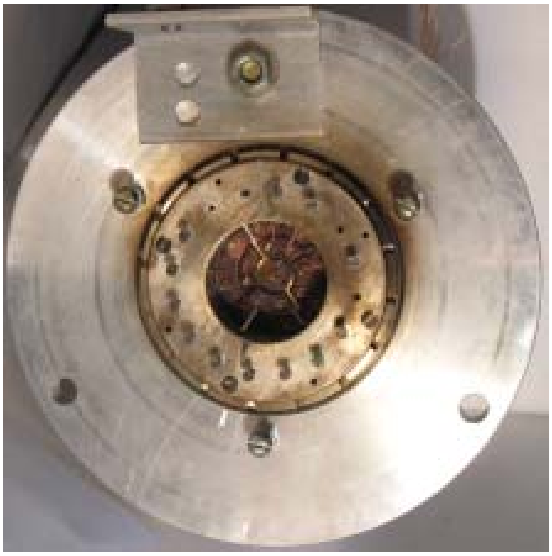

Figure 2: Target holder for TAP samples.

In the experiments at the output of the injector (acceleration of ions performed by electrostatic tube) the final energy of the ion beam is up to Z • 75 kV, where Z - ion charge. For the experiments with material surface treatment an existing target holder (Figure 2), established for the irradiation of the samples prepared for the following investigation by the atomic probe microscope is used. Those samples have a shape of needles with the cap radii about 100 nm. The additional target block was constructed and installed for another kind of samples irradiation. As samples the cylinders with the diameter of 3 mm and the thickness of 0.1 mm are used. The block enables simultaneous installation up to seven samples for ion beam irradiation (Figure 3). Irradiations at the output of the injector are held by Fe^{2+} ions with energy of 150 keV, and by the N^{1+}ion with energy of 75 keV.

For the experiments with high energy, a target assembly with samples heating system is used (Figure 4).

Figure 3: Target holder for TEM samples.

The construction of the sample holder allows samples irradiation with heating to a temperature of ~ 700^{0}C. The set-up of the sample holder consists of the copper heating block 2, sample holder assembly 1, placed on the butt copper block, two stainless steel rings 3 fixing the construction inside the water cooling screen 4 [8].

During irradiation, it is vital to control the samples temperature with tolerance ± 3^{0}C in the range from 300 to 500^{0}C. Heating of the samples is currently being implemented with the use of a proportional-integral-derivative controller (PID controller) TRM101. For the implementation of sample heating, in a copper holder (Figure 4. Pos 2) the axial blind hole was drilled for installation of the heating elements of 200 W. To control the samples temperature, two thermocouples were installed in the longitudinal blind holes located at the same distance but opposite side from heating element. One thermocouple is used to provide the PID regulation, while the second thermocouple is used as a control one

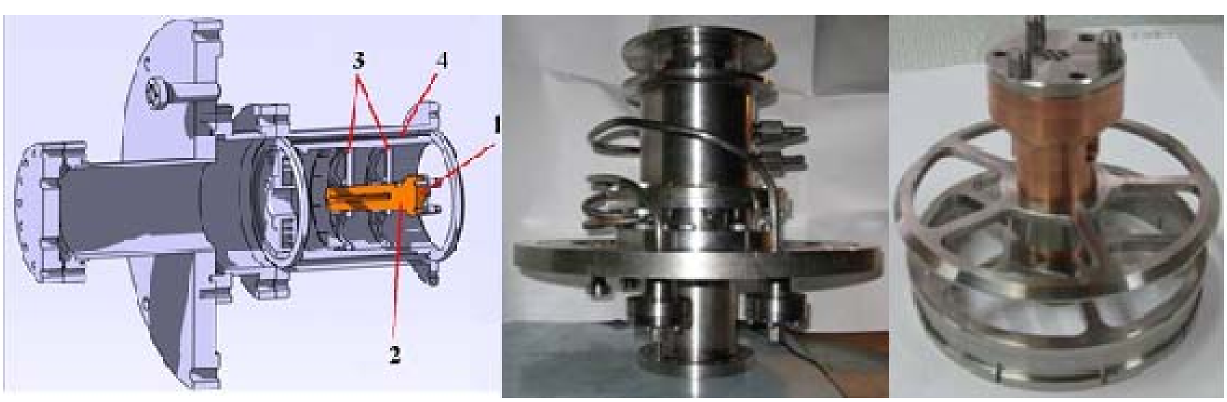

Figure 4: A) Target chamber construction: 1 – sample holder; 2 – copper ingot; 3 – stainless steel rings, 4 – water cooling system. B) Photo of system of sample holder C) Photo of copper ingot and stainless steel rings

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Figure 5 shows a graph of samples heating up to 300°C. The desired temperature is reached in about 20 minutes, the temperature stability of the sample holder after the release in normal mode is better than ± 1°C. The maximum overheating is less than 2^0C. The heating test showed required stability of temperature and revealed new opportunities for further experimental works when heating is sufficient.

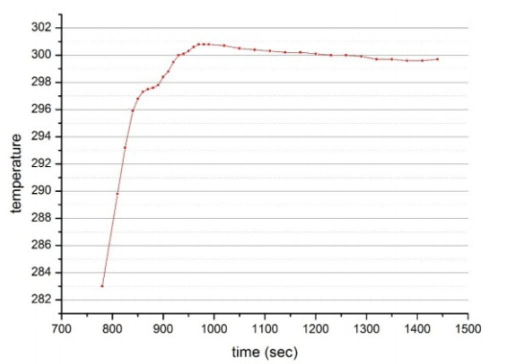
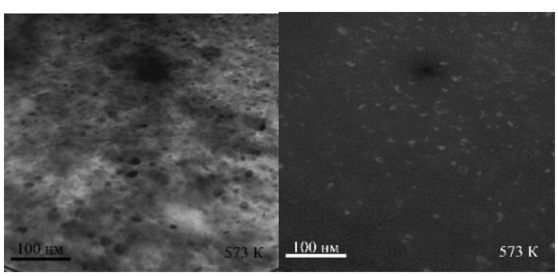


Figure 5: A plot of the T (t).

The first experiments on the irradiation of the samples of ADS Eurofer 97 steel by the Fe^{2+} ions accelerated up to 101 keV/n with simultaneous heating to a temperature of 300^0C was carried up to total flux of 1×10^{15} ions/sm^2. The final dose is ~3 DPA. After irradiation, samples were investigated by using at TEM. Results are shown in Fig.6.



a b

Figure 6: Layout of papers. Photography of irradiated sample, obtained with TEM.

Results are presented in two different modes light field (Fig.6.a) and dark field (Fig.6.b). In Fig.6.a one can see that the carbide formation (black dots) with size of ~ 30 nmare is present in irradiated steel. White dots presented on Fig.6.b are appeared to be the result of clusters formation process. This result is in a good agreement with results of [9] taken as a reference to test the method we develop.

CONCLUSIONS

In ITEP the experiments for material surface modification is underway at the accelerator HIP-1. To provide the experiments, the beams of iron, vanadium and titanium generated by vacuum-arc metal ion source, as well as ion beams of nitrogen generated by duoplasmatron are used. Several sets of experiments for the modification of the surface features were carried out. The transmission electron microscopy (TEM) and tomographic atom-probe microscopy (TAP) were used for samples analysis after ion beam treatment.

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