INTERACTION OF LOW-ENERGY PROTONS WITH ALUMINUM SURFACE

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The article presents the results of researches on the protons interaction with energy of 260 eV with a surface of aluminum foil at the temperature of 300 °C, ion current density of $\sim 1.5 \text{ mA/cm}^2$ with an exposure of 11 hours. The surface is covered with bubbles and microcracks. There is a phenomenon of surface blistering. The substantial cleaning of the surface from oxides occurs due to sputtering of the surface, as well as because of their repair in hydrogen plasma. The hydrogen content substantially increases at the depth of the sample after irradiation of aluminum with protons. The sputtering plasma-chemical reactor materials is observed.

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INTRODUCTION

In the conditions of the work of nuclear power plants structural materials are exposed to the intense irradiation of hydrogen ions and its isotopes. This leads to a change in the structure, physical and mechanical properties of these materials.

Aluminum is one of the most important technical materials. This is a fairly common structural material, so its interaction with hydrogen is an urgent task for research. Aluminum has a melting point of 658.7 °C and in nature there is a single stable isotope ²⁷Al. The advantage of aluminum and its alloys is a low density (2.7 g/cm³), relatively high technical characteristics, good heat and electrical conductivity, high performance, high corrosion resistance, and others.

The solubility of hydrogen for Al in the solid state is rather small and has a value of about $0.01 \text{ cm}^3/100 \text{ g}$ at the temperature of 500 °C [1]. Aluminum does not react chemically under normal conditions with hydrogen, thus not forming hydrides. In this regard, it has a high enough hydrogen corrosion resistance. Therefore, aluminum was selected as one of the materials for research on the interaction of low-energy protons with its surface.

The proton irradiation was carried out on a modernized source of ions at an RF discharge of 5.5 A, a negative bias voltage of 260 V [2, 3]. Exposure was 11 hours, the temperature of additional heating of 300 °C. The working pressure in PCR was 0.13 Torr, magnetic field intensity $2.5 \cdot 10^4$ A/m. The average energy of protons was 260 eV and the ion current density was 1.5 mA/cm². After irradiation with protons, surface studies were conducted using secondary ion mass spectrometry (SIMS) and raster electron microscopy (REM) [2]. The SIMS method makes it possible to determine the isotopic composition of various materials (metals and alloys) before and after irradiation, as well as by layer-by-layer removal of surface in order to determine the depth of penetration

and hydrogen concentrations in the materials under study, depending on the interaction time, proton energy, and dose sample temperature.

1. EXPERIMENTAL RESULTS AND DISCUSSION

1.1. INFLUENCE OF HYDROGEN PLASMA TREATMENT ON SURFACE OF ALUMINUM SAMPLES

In the Fig. 1 shows a comparative SIMS spectrum of irradiated and nonirradiated samples of aluminum in the range of 1...70 m/z.



Fig. 1. The survey SIMS spectrum of aluminum to treatment (asterisk) and after treatment (circles)

The amount of atomic hydrogen increases by more than two orders of magnitude on the surface of an aluminum specimen. The amplitude of the mass of aluminum (m/z = 27) is practically unchanged, and the amount of atomic oxygen on the surface is also little changed (m/z = 16). But after the irradiation, the amplitudes of the elements belonging to the alkaline and alkaline earth elements increase. The amplitude of mass ⁷Li, ²³Na, ³⁹K, ^{24,25,26}Mg, as well as isotopes of ^{40,42,43,44}Ca increases. The peaks of the three isotopes of

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silicon are increasing, since the part of the sample was covered with a silicon wafer during treatment.

For determine the localization of hydrogen in Al before and after irradiation the research was conducted on the distribution of hydrogen and oxygen in depth using the SIMS method [2]. The research results are presented in Fig. 2,a,b.



Fig. 2. The hydrogen distribution (a) and oxygen distribution (b) in depth in an aluminum sample

The sputtering depth is 1 μ m at energy of an ionic beam of argon 5 kV for an 800 s. The researches have shown that the concentration of hydrogen in the surface layer of the material increases by about an order of magnitude after irradiation. In this case molecular hydrogen is not observed in samples either to no after treatment with protons.

After treatment in hydrogen plasma, the concentration of oxygen in the irradiated portion of the sample decreases. Thus, there is a decrease in oxides in the aluminum sample after treatment. The reduction of oxides can occur due two mechanisms. The first is the repair in the hydrogen plasma, and the second is due to the physical sputtering of oxygen from the surface of aluminum.

The masses numbers of metal atoms is observed, which are part of the stainless steel (12X18N10T) from which the substrate holder is made. The distribution of individual masses of H, O, Al, Cr, Si, Ta, Nb, Al, Cu, C depths due to diffusion in the sample volume is investigated (Fig. 3).

Most impurities behave approximately the same: on the surface their concentration almost an order of magnitude more than at a depth of $1.5 \ \mu\text{m}$. There is a

smooth decrease in the intensity (amplitude) of impurities with an increase in the penetration depth in aluminum sample.



Fig. 3. The repositioned elements distribution in depth in a sample of aluminum

1.2. INVESTIGATION OF SURFACE CHANGES AFTER IRRADIATION

The surface of aluminum after treatment is significantly different from the untreated. After processing, the surface of the material becomes more uneven with the appearance of fine structure. The color of the surface changes from dark to light substantially. That fact confirms previously marked reduction of oxides of aluminum and their physical sputtering due to ion hydrogen bombardment (Fig. 4).



Fig. 4. The surface image of Al before treatment (from the left) and after treatment (on the right) with hydrogen ions

In the Fig. 5 the results of the research of the REM of the surface of aluminum are presented. The surface of the material is covered with bubbles, the formation of which is associated with the diffusion of hydrogen ions into the material, as well as its accumulation in hidden defects in the surface layers of the material.

The diffusion of hydrogen into the crystal lattice leads to the accumulation of hydrogen in the bubbles and increase the size of these bubbles. Subsequent diffusion of hydrogen into bubbles leads to their "bursting", which is responsible for the formation of microcracks on the surface. The coalescence is observed with the growth of bubbles, that is, the merging of several small bubbles into one large one.

We can say that there is a surface blistering phenomenon. The energy threshold for blistering in aluminum is near 20 keV and the fluence is 1 mA/cm^2 , so the observation of this phenomenon is rather unexpected. In our experiment, the energy of hydrogen ions was 260 eV and the ion current density was 1.5 mA/cm^2 . The main factor may be the temperature of additional heating up to 300 °C. Since in the previous experiments carried out for similar parameters of the discharge without additional heating such the phenomenon was not observed.







Fig. 5. The structure of the Al surface before treatment (a) and after treatment (b) with hydrogen ions (c)

1.3. INVESTIGATION OF THE INTERACTION OF HELIUM IONS WITH ALUMINUM

Investigation of low-energy helium ions is one of the important problems in researching the interaction of ions with materials. Such investigation were carried out at energies of ions energy 260 eV. The irradiation of aluminum foil was carried out for 36 hours. An electronic image of non-irradiated and irradiated specimens is shown in Fig. 6.

Minor sputtering of aluminum is observed in the interaction of its surface with helium ions, and not uniform, and we get island structures. X-ray fluorescence analysis of the surface before and after irradiation was performed.



Fig. 6. The electronic image of non-irradiated and irradiated aluminum sample (right – non-irradiated area, left – irradiated area)

The X-ray fluorescence analysis showed a significant decrease in impurities on the surface of an aluminum specimen after its treatment. The percentage of impurities is given in the Table.

Spectrum name	С	0	Al	Si	Fe	Cr
irradiated area						
№ 20	9.4	1.1	89.6	_	_	0.0
№ 21	11.2	0.8	87.9	1	1	0.0
non-irradiated area						
№ 23	23.7	5.9	68.7	0.8	0.7	0.0
<u>№</u> 25	24.2	9.1	63.5	0.7	0.5	0.2

CONCLUSIONS

The researches of aluminum treated with protons showed that hydrogen samples accumulate near the surface in the samples, and also a phenomenon of surface blistering at ions energies 260 eV is observed. Compared with tungsten, the results of which are published in [2], aluminum, hydrogen substantially adsorbs. In this case, molecular hydrogen is not observed.

There are planned to investigate the effect of additional heating of samples in order to determine the

main parameter responsible for its formation for further researches of the phenomenon of low-energy surface blistering.

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ВЗАИМОДЕЙСТВИЕ НИЗКОЭНЕРГЕТИЧНЫХ ПРОТОНОВ С ПОВЕРХНОСТЬЮ АЛЮМИНИЯ

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Приведены результаты исследований взаимодействия протонов с энергией 260 эВ с поверхностью алюминиевой фольги при температуре 300 °С и плотности ионного тока ~ 1,5 мА/см² с экспозицией 11 часов. Поверхность алюминия значительно меняется после процесса облучения, покрывается пузырьками и микрократерами. Наблюдается явление поверхностного блистеринга. После облучения алюминия протонами существенно увеличивается содержание водорода вблизи поверхности образца, содержание которого плавно уменьшается по глубине.

ВЗАЄМОДІЯ НИЗЬКОЕНЕРГЕТИЧНИХ ПРОТОНІВ З ПОВЕРХНЕЮ АЛЮМІНІЮ

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Приведено результати досліджень взаємодії протонів з енергією 260 еВ з поверхнею алюмінієвої фольги при температурі 300 °С, щільності іонного струму ~ 1,5 мА/см² з експозицією 11 годин. Поверхня алюмінію значно змінюється після процесу опромінення, покривається бульбашками та мікрократерами. Спостерігається явище поверхневого блістерінгу. Після опромінення алюмінію протонами істотно збільшується вміст водню поблизу поверхні зразка, вміст якого плавно зменшується за глибиною.