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Abstract. The influence of irradiation in hydrogen plasma with oxygen impurity on the penetration of hydrogen isotopes through the surface of tungsten, through the surface of beryllium and aluminum layers on tungsten, and through the tungsten-coating interface was studied. It was shown that all these processes are accelerated in the presence of oxygen impurity in plasma. A conclusion is made that detritisation of the surface of both in the conditions of a fusion reactor tungsten and beryllium is possible.

1. Introduction

Beryllium and tungsten are chosen as the materials for the first wall and the diverter of the ITER tokamak, respectively. Beryllium and tungsten have oxide layers formed on their surfaces. The surface of tungsten and beryllium will be coated by oxides if the ITER plasma would have an oxygen impurity. Sputtered beryllium atoms will be deposited on the surface of tungsten tiles and form oxide layers under these conditions. It is known [1–3] that the oxide layers function as barriers for hydrogen diffusion and lead to accumulation of hydrogen isotopes in metals. This fact makes it relevant to study the features of accumulation of hydrogen isotopes, in particular tritium, in materials in contact with plasma.

The authors of paper [4] discovered the effect of low-temperature ($T \approx 320$ K) hydrogen isotopes penetration through the surface of stainless steel (phenomenon of Activated Surface Penetration – ASP) under the irradiation by hydrogen ions in hydrogen atmosphere with oxygen impurity, as well as hydrogen plasma with oxygen impurity. This effect is explained by the processes initiated by inelastic collisions of deuterium and oxygen atoms/ions with the chromium oxide layer on the surface of stainless steel [5].

The aim of this work was to study the possibility of developing the ASP phenomenon on the oxidized surface of tungsten and tungsten with beryllium and aluminum layers, as well as reveal the features of hydrogen isotope transport between tungsten and coatings upon irradiation with ions of hydrogen plasma with oxygen addition and analyze the distribution of trapped atoms between tungsten and coatings.

The experiments were carried out using samples made of pure tungsten and tungsten coated with beryllium or aluminum layers on one or both sides. The number of tungsten samples with beryllium coating at our disposal was insufficient to carry out the entire program of experiments. Therefore, after

we made sure that the results of experiments on tungsten samples with beryllium and aluminum layers were similar, the latter were used to study the features of the ASP phenomenon in detail.

2. Experimental facility and experimental method

Beryllium layers 100 ± 10 nm thick were deposited on both sides of samples made of Plansee-grade polycrystalline tungsten of ≈ 0.2 mm thick at the National Institute of Laser, Plasma and Radiation Physics (Bucharest, Romania). Tungsten samples were mechanically polished, cleaned in acetone and alcohol ultrasonic baths. Layers were formed by atoms evaporated from a molten beryllium target. The residual gas pressure during the deposition did not exceed 1.2×10^{-3} Pa. The deposition rate was 0.5 nm/s. The temperature of tungsten substrate during the deposition process increased from room temperature to 370–420 K due to radiation from beryllium targets.

Aluminum layers 200 ± 10 nm thick were deposited on samples made of Plansee-grade polycrystalline tungsten ≈ 1.0 mm thick at NRNU MEPhI (Moscow, Russia). Tungsten samples were mechanically polished, cleaned using acetone and alcohol ultrasonic baths and then annealed in vacuum with the residual pressure of $\leq 5\times10^{-6}$ Pa at a relatively low temperature (1600K) to remove the "technological" hydrogen. The coating was formed by aluminum atoms sputtered from the aluminum target with ions of argon plasma with hydrogen impurity. The residual gas pressure during the deposition did not exceed 5×10^{-4} Pa. The deposition rate was 0.055 nm/s. The temperature of tungsten substrate during the deposition was 450-500 K. Samples of tungsten with aluminum layer from one and both sides were made.

The X-ray Energy Dispersion Spectroscopy (EDS) and the Thermal Desorption Spectroscopy (TDS) analysis has shown that the layers deposited under the conditions described above contained hydrogen and oxygen with concentrations of 40 and 9 at. %, respectively, in beryllium layers and 35 and 13 at. % in aluminum layers. It should also be noted that an oxide film was formed on the surface of the deposited layers after contact with the atmosphere.

The experiments were conducted on the "MIKMA" stand [6]. The pumping system allows for the residual pressure in both radiation chambers to not exceed 1×10^{-4} Pa, as well as pump the TDS chamber down to 1×10^{-5} Pa.

In the first series of experiments, deuterium was implanted in the samples of tungsten coated from both sides with beryllium and aluminum layer by irradiating them with 50 eV/at energy deuterium plasma ions. Plasma consisted mostly of D_2^+ (82-85%) ions, with the rest being D^+ and D_3^+ ions. For this reason, the experimental results were analyzed assuming that the samples were irradiated by D_2^+ ions only. The parameters of implantation were as follows: ion flux density $j=3.7\times10^{19}$ at/m^{2}s, fluence $F=1.3\times10^{23}$ at/m^2, the temperature of the samples during the irradiation T=500 K. The first sample with implanted deuterium was used to measure the number of deuterium and hydrogen atoms after the deuterium implantation into the samples of tungsten coated from both sides with beryllium and aluminum layers. The second sample was kept in vacuum for 1 hour after the implantation at 500 K i.e. for the same duration at the same temperature as the one irradiated by H_2+1 at. % O_2 plasma. This was made to see whether simply exposing the sample to vacuum would lead to desorption of the implanted deuterium. The measurement of the number of deuterium atoms was then conducted using TDS. The third sample was irradiated from the same side by ions with energy of 50 eV/at. (mostly H_2^+) of H_2+1 at. % O_2 plasma. The parameters of irradiation were the following: ion flux density $j=3.7\times10^{19}$ at/m^{2}s, fluence $\Phi=1.3\times10^{23}$ at/m^2, the temperature of the samples during the irradiation T=500 K. Measurement of the number of deuterium and hydrogen atoms after the H_2+1 at. % O_2 plasma irradiation was then conducted using TDS.

In the second series of experiments, tungsten samples coated from one side with aluminum layer were used. For the first group of coated samples, deuterium was implanted from the tungsten side. For the second group, implantation was performed from the aluminum-coated side. In both cases, the implantation conditions were the same as in the previous series of experiments.

The first sample with implanted deuterium from each group was analyzed using TDS method to measure the amount of deuterium atoms captured and how the amount of hydrogen atoms in the

samples changed after implantation of deuterium. The second sample from each group was kept in vacuum for 1 hour after implantation of deuterium at 500 K i.e. for the same duration at the same temperature as the one irradiated by H_2+1 at. % O_2 plasma. The TDS method was used to determine how the content of hydrogen and deuterium in the samples changed after being exposed to vacuum. After implantation of deuterium, the third and fourth samples of each group were irradiated from the tungsten side and from the aluminum layer side by ions with energy of 50 eV/at. (mostly H_2^+) of H_2+1 at. % O_2 plasma. The parameters of irradiation were the following: ion flux density $j=3.7\times10^{19}$ at/m^{2}s, fluence $F=1.3\times10^{23}$ at/m^2, T=500 K. The measurement of the number of deuterium and hydrogen atoms after the H_2+1 at. % O_2 plasma irradiation was then conducted using TDS.

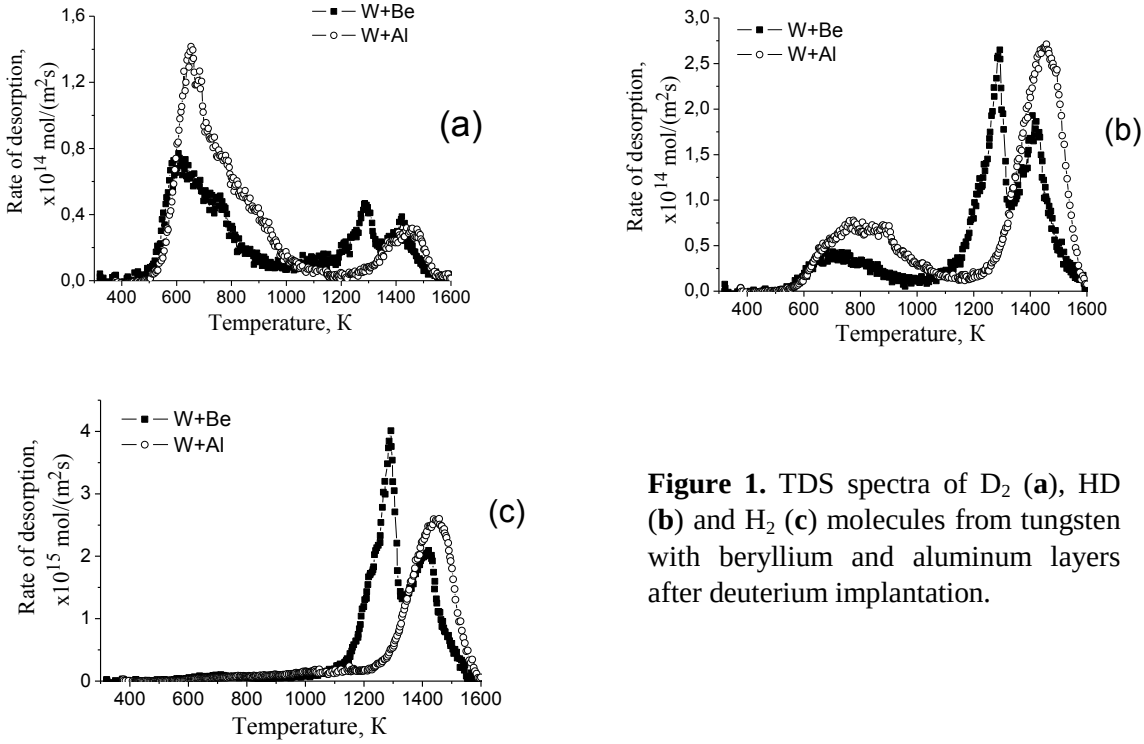

Figure 1. TDS spectra of D_2 (**a**), HD (**b**) and H_2 (**c**) molecules from tungsten with beryllium and aluminum layers after deuterium implantation.

The heating rate of the samples during TDS was 5 K/s. The temperature of the samples during plasma irradiation and the TDS analysis was measured using W-Re thermocouple welded to the samples. Desorption signals of H_2, HD, D_2, H_2O, HDO and D_2O molecules were measured during TDS. Desorption of another hydrogen isotopes-containing molecules was negligible.

3. Results and discussion

The experimental results are presented in figure 1 and table 1. A comparison of the TDS spectra from tungsten with aluminum layer (figures 1a, b) and from metallic tungsten and aluminum (figure 2) allows us to state that deuterium desorption in the temperature range 600–1000 K occurs both from tungsten and from aluminum layer. We didn't have the opportunity to study desorption from metallic beryllium, but figure 1b suggests that deuterium trapping in tungsten with beryllium layer during implantation also occurs in tungsten and in beryllium layer. More than 90% of the hydrogen atoms trapped in the samples during deposition of layers desorb in the temperature range 1300-1600K. Possible reason for desorption of hydrogen isotopes from tungsten with beryllium and aluminum layer in the temperature range 1300-1600K can be formation of W-Be and W-Al alloys. The possibility of diffusion of aluminum atoms into tungsten and the formation of alloy of the type W-Be and W-Al are indicated in a number of literature [7, 8].

It can be seen (table 1) that deposition of beryllium and aluminum layers on tungsten leads to an increase in the trapping of deuterium and hydrogen atoms (table 1, columns 2 and 4). At the same time, upon subsequent irradiation of samples with ions of H_2 + 1 at.% O_2 plasma with energy of 50 eV / at (practically not sputtering the surface of the samples), approximately the same amount of deuterium is desorbed from tungsten and from tungsten with beryllium and aluminum layers (table 1 columns 2 and 3).

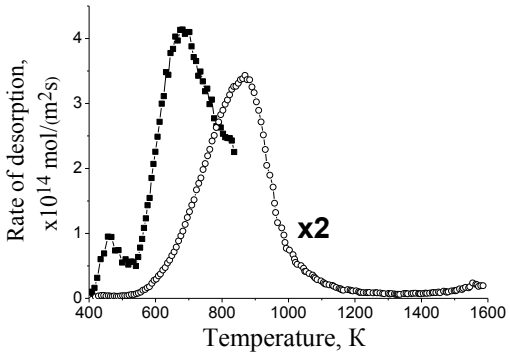

Figure 2. TDS spectrum of D_2 molecules from aluminum and tungsten after deuterium implantation (Implantation parameters: ion energy E= 650 eV/at, ion flux density j=3.7×10^{19} at/m^2s, fluence Φ=1.3×10^{23} at/m2, T=500 K).

It can be concluded that the ASP phenomenon develops both on the oxidized surface of tungsten and on tungsten with beryllium and aluminum layers. Moreover, the intensities of the ASP phenomenon on the surface of beryllium and aluminum layers are close. Therefore, further the features of the development of the ASP phenomenon were studied on tungsten samples with aluminum layer.

Table 1. The number of deuterium and hydrogen atoms in tungsten samples (W) and in tungsten samples with beryllium (W_{Be}) and aluminum (W_{Al}) layers after implantation of deuterium and subsequent irradiation in H_2 + 1at. % O_2 plasma.

	Amount of deuterium atoms, ×10^{19} at/m^2		Amount of hydrogen atoms, ×10^{20} at/m^2	
Sample	Before irradiation by H_2+1 at. % O_2 plasma	After irradiation by H_2+1 at. % O_2 plasma	Before irradiation by H_2+1 at. % O_2 plasma	After irradiation by H_2+1 at. % O_2 plasma
W	9.4	1.6	0.6	0.4
W_{Be}	16.0	3.1	6.5	4.1
W_{Al}	28.0	13.0	21.4	48.2

4. Conclusion

Study of the features of developing the ASP phenomenon (Activated Surface Penetration of hydrogen isotopes under irradiation with hydrogen atoms/ions and oxygen) on the surface of tungsten and tungsten with beryllium and aluminum layers has been conducted. Also transport of hydrogen isotopes between tungsten and aluminum layer during irradiation by hydrogen plasma with oxygen addition has been studied.

It is shown that the ASP phenomenon develops on the oxidized surface of tungsten, as well as on tungsten with beryllium and aluminum layers. The intensities of the ASP phenomenon on the surface

of beryllium and aluminum layers are close, and hydrogen isotopes are trapped in tungsten with beryllium and aluminum layers in the same traps.

The low-energy H_2 + 1 at. % O_2 plasma irradiation of both the oxidized surface of tungsten and the oxidized surface of aluminum layer on tungsten initiates penetration of deuterium previously implanted into the tungsten / aluminum layer into the aluminum layer / tungsten and desorption from 24 to 76% of deuterium atoms.

It is concluded that the ASP phenomenon can be used for detritization of divertor tiles coated with beryllium layers and beryllium tiles of ITER first wall.

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