FEATURES OF THE LOW ENERGY PROTONS INTERACTION WITH MOLIBDEN SURFACE

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The surface of molybdenum changes significantly after the proton irradiation process. The sputtering of the surface leads to a substantial cleaning of oxides. It is also possible to reduce oxides in a hydrogen plasma. The surface of molybdenum becomes more uneven with the manifestation of the fine structure after treatment. Molybdenum is sputtering at the rate ~ 0.1 μm/hour at the temperature of 300 °C and an ion current density ~ 1.5 mA/cm^2. The hydrogen content near the surface of molybdenum practically does not increase after irradiation with protons. The content of hydrogen in molybdenum does not change in depth, it is constant before and after irradiation. Molecular hydrogen is not observed in the both samples. The masses numbers of metal atoms is observed, which are part of the stainless steel (12X18N10T) from which the substrate holder is made.

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INTRODUCTION

The research of non-magnetic materials and their alloys with high melting points and low hydrogen sorption is one of the most important problems facing the developers of fusion devices.

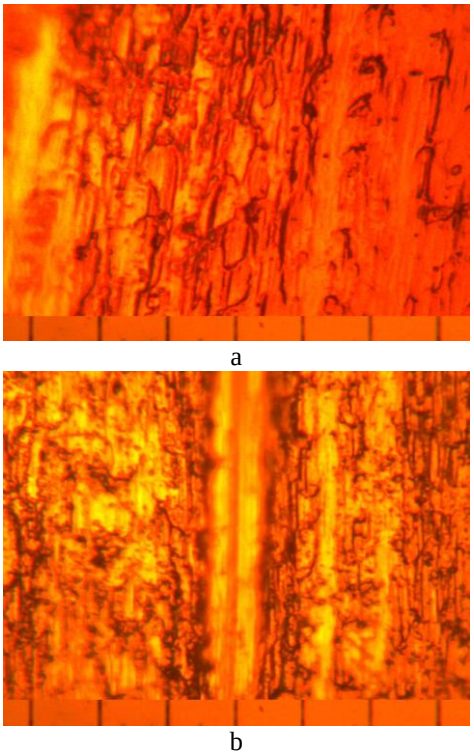
In comparison with traditional power plants, materials in nuclear and especially in thermonuclear plants operate in much more difficult conditions [1, 2]. When neutrons and protons irradiate the first wall of a thermonuclear reactor, they are transformed into atoms of hydrogen. An increase in the diffusion of hydrogen atoms into the depth of the material occurs at elevated temperatures (up to 150 °C), high (~ 800 °C) and especially ultrahigh temperatures (more than 800 °C). This reduces corrosion resistance. As a result of interaction with the material of investigation sample, the protons are converted to atomic hydrogen, which leads to gas sputtering and the appearance of hydrogen brittleness of materials.

Also important carry out investigations of structural materials that will be in contact with thermonuclear plasma and should be as inert as possible with respect to it. Molybdenum is one such material. It has a melting point 2896 K (boiling point 4912 K), high resistance to deformation and a small cross section for the capture of thermal neutrons. Therefore, molybdenum was chosen as one of the materials for research the interaction of low-energy protons with it.

EXPERIMENTAL RESULTS AND DISCUSSION

The proton irradiation was carried out on a modernized source of ions at an RF discharge 5.5 A, a negative bias voltage 260 V [3, 4]. Treatment was carried out for 11 hours, the temperature of additional heating was 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/cm^2.

The research of the surface were carried out on the optical microscope after irradiation with molybdenum protons. The surface of molybdenum changes significantly after the irradiation process. The sputtering of the surface leads to a substantial cleaning of oxides. It is also possible to reduce oxides in a hydrogen plasma. The surface of molybdenum becomes more uneven with the manifestation of a fine structure after treatment (Fig. 1,a,b). The molybdenum is sputtering at the rate ~ 0.1 μm/hour at the temperature 300 °C and an ion current density ~ 1 mA/cm^2.



a

b

Fig. 1. Microscopic photograph of the structure of the molybdenum surface before treatment (a) and after treatment with protons (b) (distance between marks 10 μm)

The comparative research of the mass spectra of the processed and untreated samples in the mass range m/z = 1… 250 atomic mass units (amu) were carried out. The samples were sputtering with argon ions with an energy of 500 eV with a primary ion current 4 mA. The analysis is based on the INA-3 instrument with a common ion-optical system, which uses a quadrupole mass analyzer. More detailed features of the methods of obtaining and processing of secondary ion mass spectra shown in [3].

Fig. 2 shows the secondary mass spectra obtained before and after irradiation a sample with protons (in the range 90…120 amu). Molecular hydrogen is not observed in both samples. The seven isotopes of Mo are separated, and as many of its oxides and dioxides with amplitudes of 20 and 2000 times smaller. The masses numbers of metal atoms is observed, which are part of the stainless steel (12X18N10T) from which the substrate holder is made. The peaks of the three isotopes of silicon are increasing, since the part of the sample was covered with a silicon wafer during treatment. The number of lines on the mass spectra has drastically decreased when replacing a stainless steel substrate holder with an aluminum substrate holder, which greatly facilitated the processing of spectra (Fig. 3).

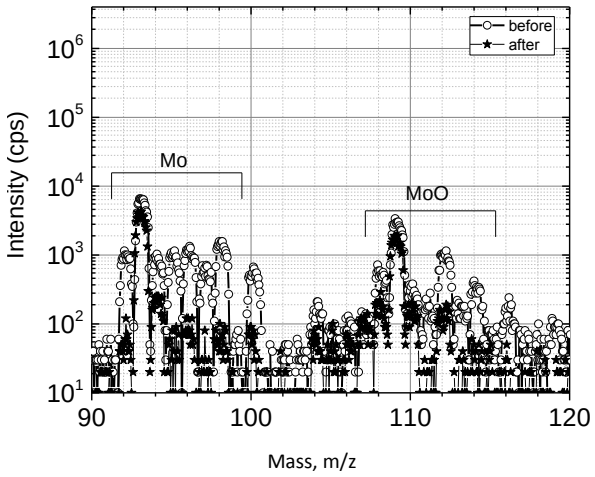


Fig. 2. The comparative secondary ion mass spectra of molybdenum surface before and after irradiation with 260 eV protons, at the temperature of 300 °C (circles indicate the masses before irradiation, asterisks after irradiation with protons)

The amount of impurities on the surface of molybdenum and their oxides substantially decreases, which significantly facilitates the processing of secondary ionic mass spectra with using duralumin substrate holder. This is due to the fact that aluminum has only one isotope and the alloying elements are copper (4.4 % has 2 stable isotopes), magnesium (1.5%, 3 isotopes) and manganese (0.6 %, 1 isotope). The seven impurity isotopes are manifested in the spectra from the duralumin substrate holder. The aluminum emissions in the form of individual droplets arise on the surface of molybdenum during the combustion of the discharge on such a cathode.

The research of the distribution of many impurities over the depth of the molybdenum sample were carried out. In Fig. 4 shows the results of the distribution of impurities of hydrogen, oxygen, chromium and aluminum over the depth of the molybdenum sample before and after its treatment with protons.

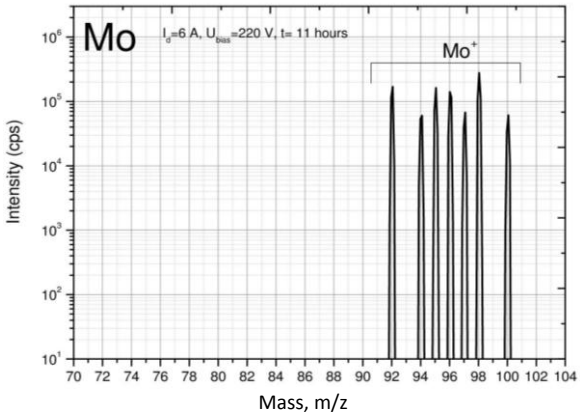


Fig. 3. The SIMS mass spectrum of molybdenum with using the duralumin holder

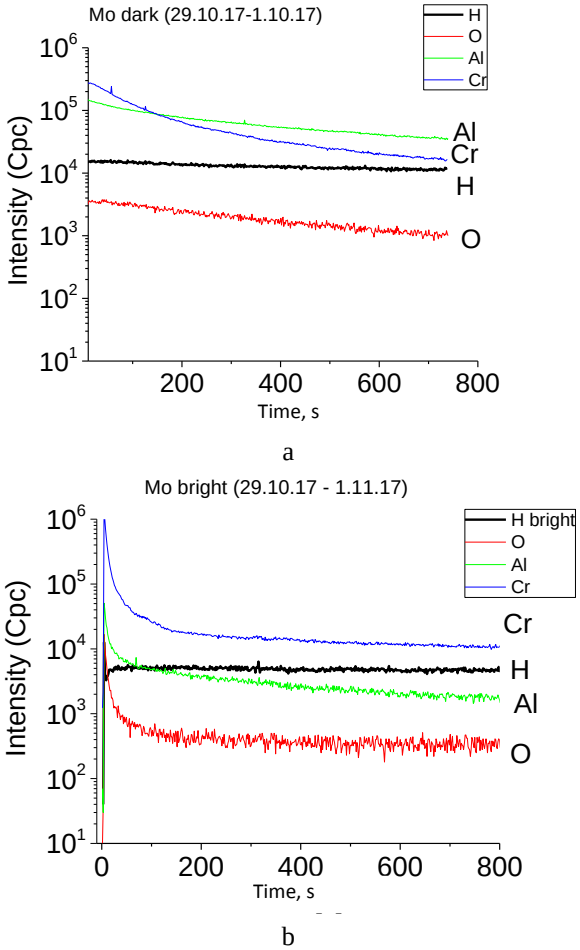


Fig. 4. The distribution of some materials by depth in molybdenum (a – before irradiation; b – after irradiation with protons)

The depth distribution of atomic hydrogen in a sample of molybdenum is almost uniform before and after proton irradiation. Moreover, the amount of hydrogen after irradiation becomes three times less than before irradiation with protons. The amount of impurities of oxygen, chromium and aluminum increases of two orders of magnitude on the surface of

molybdenum after irradiation. This indicates their reprecipitation on the surface after sputtering from the substrate holder. The hydrogen content near the sample surface practically does not increase after molybdenum is irradiated with protons.

The depth distribution of other impurities behaves similarly to the above (Fig. 5).

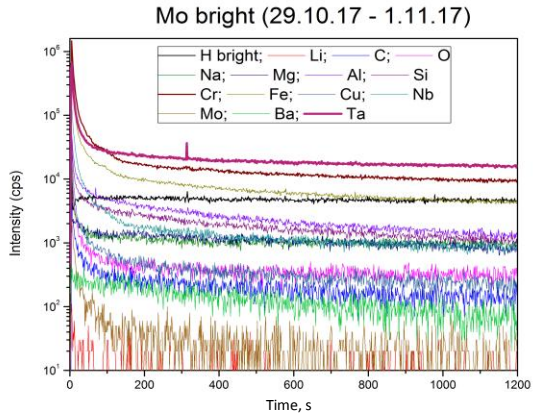


Fig. 5. The depth distributions of individual materials after molybdenum treatment with protons (holder of stainless steel substrates)

The distribution of individual masses over the depth of penetration into the samples during their sputtering with a beam of argon ions up to 1200 s (to a depth of 2 μm) was also investigated (see Fig. 3). The research were conducted on the depth of H, O, Al, Cr, Mo, Ta, Nb, Al, Cu, etc.

In Fig. 6 shows the results of the distribution of several impurities in depth with using the holder of the substrate of duralumin.

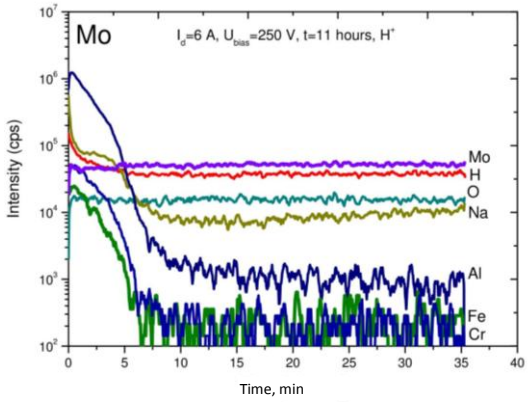


Fig. 6. The distributions of molybdenum, hydrogen, oxygen, and other impurities in depth with using a duralumin substrate holder

The similar pattern is observed, but the deposition of impurities on the surface substantially increases. The molybdenum and hydrogen are distributed almost evenly from the surface itself. Other impurities are distributed mainly on the surface, which indicates their reprecipitation from the surface of the substrate holder. In the case of aluminum, chromium, iron and calcium, their amount on the surface increases by three orders of magnitude compared with their amount in molybdenum itself.

CONCLUSIONS

The content of hydrogen in molybdenum does not change in depth, it is constant both before and after irradiation. The surface of molybdenum changes significantly after the irradiation process. The sputtering of the surface leads to a substantial cleaning of oxides. The surface of molybdenum becomes more uneven with the manifestation of a fine structure after treatment. The molybdenum is sputtering at the rate ~ 0.1 μm/hour at the temperature 300 °C and an ion current density ~ 1.5 mA/cm^2. Molecular hydrogen is not observed in the both samples. The masses numbers of metal atonMass, m/z d, which are part of the stainless steel (12X18N10T) from which the substrate holder is made.

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ОСОБЕННОСТИ ВЗАИМОДЕЙСТВИЯ НИЗКОЭНЕРГЕТИЧНЫХ ПРОТОНОВ С ПОВЕРХНОСТЬЮ МОЛИБДЕНА

О.А. Федорович, В.В. Гладковский, Б.П. Полозов, Л.М. Войтенко, Е.Г. Костин, А.А. Рокицкий, А.С. Оберемок

Поверхность молибдена значительно изменяется после процесса облучения протонами. Происходит существенная очистка поверхности от оксидов из-за распыления поверхности. Возможно также восстановление оксидов в водородной плазме. Поверхность молибдена после обработки становится более

неровной с проявлением мелкой структуры. Происходит распыление молибдена со скоростью ~ 0,1 мкм/ч при температуре 300 °С и плотности ионного тока ~ 1,5 мА/см^2. Содержание водорода вблизи поверхности образца после облучения молибдена протонами практически не увеличивается. Содержание водорода в молибдене по глубине не меняется, а является постоянным как до облучения, так и после. Молекулярный водород не наблюдается в обоих образцах. Разделяются 7 изотопов Mo и столько же его оксидов. На поверхности Mo после обработки наблюдается ряд масс атомов, принадлежащих материалу держателя подложки из нержавеющей стали (12Х18Н10Т) (изотопы Cr, Fe, Ni, Ti, Mn и др.). Наблюдаются также изотопы других примесей, которые могут перераспыляться с держателя подложки и кремния, которым прикрывается необрабатываемый участок образца.

ОСОБЛИВОСТІ ВЗАЄМОДІЇ НИЗЬКОЕНЕРГЕТИЧНИХ ПРОТОНІВ З ПОВЕРХНЕЮ МОЛІБДЕНУ

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Поверхня молібдену значно змінюється після процесу опромінення. Відбувається істотне очищення поверхні від оксидів через розпилення поверхні. Можливе також відновлення оксидів у водневій плазмі. Поверхня молібдену після обробки стає більш нерівною з проявом дрібної структури. Відбувається розпорошення молібдену зі швидкістю ~ 0,1 мкм/год при температурі 300 °С і щільності іонного струму ~ 1,5 мА/см^2. Вміст водню поблизу поверхні зразка після опромінення молібдену протонами практично не збільшується. Вміст водню в молібдені за глибиною не змінюється, а є постійним як до опромінення, так і після. Молекулярний водень не спостерігається в обох зразках. Розділяються 7 ізотопів Mo і стільки ж його оксидів. На поверхні Mo після обробки спостерігається ряд мас атомів, які належать матеріалу утримувача підкладки з нержавіючої сталі (12Х18Н10Т) (ізотопи Cr, Fe, Ni, Ti, Mn і ін.). Спостерігаються також ізотопи інших домішок, які можуть перерозпилятися з тримача підкладки і кремнію, яким прикривається необроблювана ділянка зразка.