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DEPENDENCES OF HELIUM RETENTION IN TUNGSTEN AND TANTALUM COATINGS IRRADIATED WITH He^+ IONS ON FLUENCE AND TEMPERATURE

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The processes of helium accumulation and thermal desorption for tungsten and tantalum coatings deposited on a stainless-steel substrate with an intermediate titanium layer were studied at various temperatures of the samples when irradiated with He^+ ions to various fluences. The dependences of the concentration of captured helium and the form of the spectra of its thermal desorption into vacuum were found both on the fluence of He^+ ions and on the temperature of the samples upon irradiation. Possible mechanisms for the accumulation and thermal desorption of helium, as well as the formation of defects in the crystal lattice of the samples, are discussed.

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INTRODUCTION

Tungsten and tantalum are considered [1-5] as the protective plasma-facing materials for components of thermonuclear reactors with magnetic [1, 3-5] and inertial plasma confinement [2]. They are proposed to be used both as separate materials (W [1, 3, 5] and Ta [2, 3]) and in form of tungsten-tantalum composites [4] (with 10 and 20 at.% Ta fibers in W matrix) and WTa_5 alloys [5] (with 5 wt.% Ta). One of the ways of production of protective coatings for plasma-facing components (PFC) is the plasma sputtering of tungsten or tantalum onto surfaces of PFC [1, 2]. One of the advantages of Ta and W is the negligible accumulation of hydrogen isotopes in them, i.e., the insignificant accumulation of radioactive tritium under plasma irradiation. This problem is intensively studied by the authors of [1-11]. The main aims of such investigations are increasing the radiation resistance of the proposed materials and obtaining new materials with improved parameters. Processes of capture, retention, and thermal desorption of deuterium and helium that ion-implanted into tungsten [6-9, 11] and tantalum [10] coatings at room temperature were studied. In [10] also the processes going at the deuterium implantation into Ta and W coatings irradiated with D^+ ions were studied at elevated temperatures of samples. Formation of radiation defects in W coatings was analyzed with TEM [11]. The influence of the temperature of the samples on the capture, retention and thermal desorption of implanted deuterium from the coatings was shown.

In the present work, we investigated the processes accompanying the implantation of helium ions into tungsten and tantalum coatings at elevated sample temperatures during bombardment with He^+ ions. The influence of the sample temperature on the helium retention and thermal desorption from the coatings into vacuum was shown for different fluences of irradiation with helium ions. Mechanisms of helium capture, retention, and thermal desorption for irradiated tungsten and tantalum coatings and the influence of radiation defects of the crystal lattice of the samples on these processes are discussed.

1. EXPERIMENTS

Tungsten and tantalum coatings obtained by the magnetron sputtering of W and Ta targets in an Ar atmosphere at the pressure of 1.0 Pa were analyzed. The coatings were deposited at $T = 600$ K at the rate of 0.6 nm/s on 0.8 mm-thick stainless-steel substrate with an intermediate Ti layer with the thickness $\leq$ 10 nm predeposited onto it. The thicknesses of the coatings were $\sim$1.0 μm (W) and $\sim$1.5 μm (Ta). They were irradiated with 20-keV He^+ ions at a flux $3.0 \cdot 10^{13}$ $cm^{-2} \cdot s^{-1}$ at T_o: 290, 370, 470, 540, 570, 670 and 870 K and fluences in the range from $1.0 \cdot 10^{17}$ cm^{-2} to $1.0 \cdot 10^{18}$ cm^{-2} for W coatings and at T_o: 290, 370, 670, 770 and 870 K and doses from $1.0 \cdot 10^{17}$ cm^{-2} up to $1.0 \cdot 10^{18}$ cm^{-2} for Ta coatings. Helium ions was generated by a magnetic arc source of positive ions with air cooling [12, 13]. According to [14], the mean projective and total ranges of 20-keV He^+ ions in the W and Ta coatings were $\sim$60 and $\sim$160 nm, respectively.

Thermal-desorption spectrometry (TDS) was used for investigation of the retention as well as thermal desorption of helium from tungsten and tantalum coatings. With TDS method and a small static gas mass spectrometer calibrated with a GELIT-1 leak valve, we measured the TDS spectra of helium. The concentrations C (in cm^{-2}) and capture coefficients $\eta = C/\Phi$ of the implanted gas were determined. The thermal desorption spectrum $S = f(T)$ was the dependence of the amount S (in cm^{-2}) of helium released at a given temperature T of the sample during its heating, on this temperature.

The TDS spectra of helium were registered at heating the irradiated samples with a constant rate $\alpha = 0.8$ K/s in the temperature range 290...1800 K on Ta ribbon. The temperature was measured with welded to it thermocouples from either chromel-alumel or platinum-platinum alloy with 13 % rhodium. The temperature measurement error was $\pm$25 K. The pressure in the chamber was $1.3 \cdot 10^{-4}$ Pa during TDS analysis. The detection sensitivity of the procedure used to determine

the amount of helium particles was no worse than $2.0 \cdot 10^{12}$ cm^{-2}.

2. RESULTS AND DISCUSSION

Thermal desorption of implanted helium was observed during heating samples irradiated with helium. The feature of the thermal desorption spectra depends on the type of material, the temperature of the sample during helium implantation, as well as the fluence of irradiation ions. In Fig. 1 the spectra of helium thermal desorption from W (see Fig. 1,a) and Ta (see Fig. 1,b) coatings He^+ ions irradiated to different fluences at T_o = 290 K are shown. In Fig. 1,a dependencies (1-5, 7) were taken from [9].

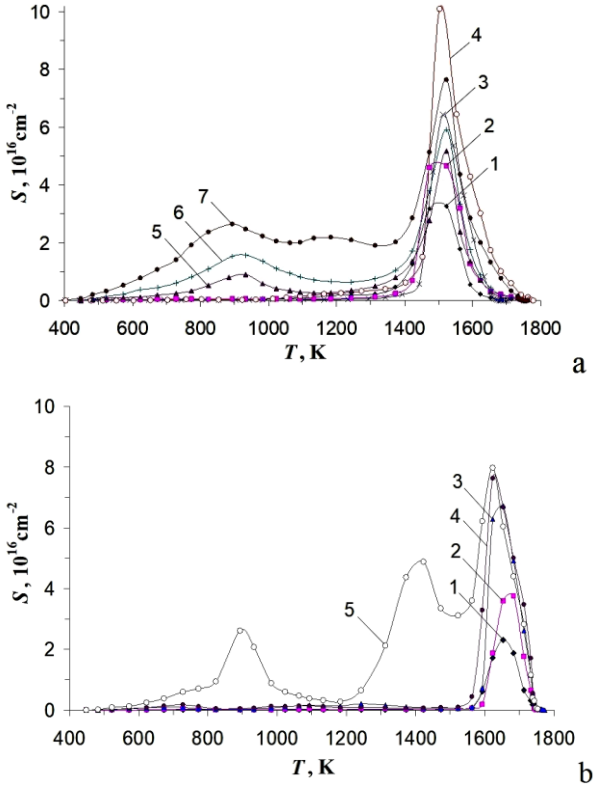

Fig. 1. Spectra of helium thermodesorption from W (a) and Ta (b) coatings, irradiated with 20-keV He^+ ions up to different fluences Φ, 10^{17} cm^{-2}: 1 – 1.0; 2 – 2.0; 3 – 3.1; 4 – 4.0; 5 – 4.7; 6 – 6.1; 7 – 8.3 for (a) and (1) 1.0, (2) 2.0, (3) 4.0, (4) 6.0, (5) 10.0 for (b); T_o = 290 K, α = 0.8 K/s

Fig. 1,a shows, that for $\Phi \le 4.0 \cdot 10^{17}$ cm^{-2} (curves 1-4), helium released from W coatings in vacuum in the temperature range 700 K ≤ ΔT ≤ 1750 K predominantly in the peak with a temperature at the maximum $T_{max} \approx$ 1520 K. The release of helium is observed in the range 450...1750 K for fluences $4.7 \cdot 10^{17}$ cm^{-2} ≤≤ Φ ≤ $8.3 \cdot 10^{17}$ cm^{-2} (curves 5-7). In addition to the main peak of the release of He with T_{max} at 1520 K, low-temperature peaks of its thermal desorption in the range 450…1300 K are observed. The helium release from the Ta coating into vacuum, as is seen in Fig. 1,b, for fluences $\Phi \le 6.0 \cdot 10^{17}$ cm^{-2} (curves 1-4) occurs in the temperature range 500 K ≤ ΔT ≤ 1750 K mainly with the peak at $T_{max} \approx$ 1660 K. For the fluence $\Phi = 1.0 \cdot 10^{18}$ cm^{-2} (curve 5), the release of He is observed in the temperature range 480...1750 K. In addition to the main peak of the release of He with T_{max} at 1660 K, low-temperature peaks of its thermal desorption in the range 480...1550 K also are observed. The results showed that the helium thermal desorption from the above coatings has, in general, the same character. But the value of T_{max} of the helium release peak for a Ta coating shifts to higher temperatures. Also, the change in the spectrum of thermal desorption of helium from a single-peak dependence to a more complex one occurs at high fluences of irradiation with He^+ ions.

The influence of the samples temperature during He^+ ion bombardment on the helium retention and thermal desorption into vacuum was studied for W and Ta coatings at low doses, and for W at higher doses. In Fig. 2 the spectra of helium thermal desorption into vacuum from a W coating irradiated with the same dose from the low range at different temperatures T_o are shown. The spectra of helium thermal desorption into vacuum from Ta coatings He^+ ions irradiated to the same fluences from the range of small values of $\Phi \le 6.0 \cdot 10^{17}$ cm^{-2} have a similar form for different values of the temperature T_o. It is shown that the character of the thermal desorption spectra of helium from W and Ta coatings is retained with an increase in the temperature of the samples during helium implantation into them. The thermal desorption spectra have the form of dependences with one peak for both types of coatings. The maximum of helium thermal desorption was recorded at temperatures $T_{max} \approx$ 1520 K for W and $T_{max} \approx$ 1660 K for Ta.

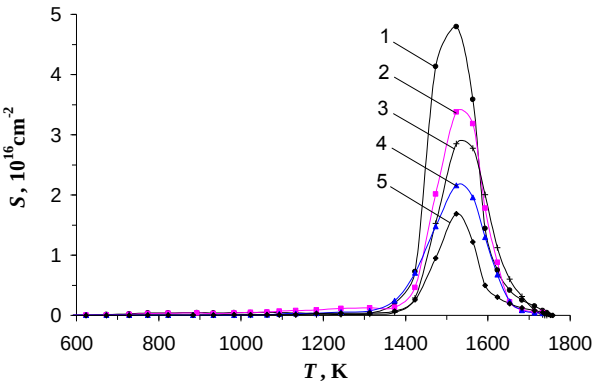

Fig. 2. Spectra of helium thermal desorption from W coating at different temperatures T_o, K: 1 – 290, 2 – 370, 3 – 540, 4 – 670, 5 – 870; Φ = $2.0 \cdot 10^{17}$ cm^{-2}, α = 0.8 K/s, 20-keV He^+ ions

In Fig. 3 the spectra of helium thermal desorption from W coatings irradiated to the fluence ~ $7.0 \cdot 10^{17}$ cm^{-2} for various temperatures T_o are shown. Two temperatures: the room T_o = 290 K (curves 1 and 2) and increased T_o = 570 K (curves 3 and 4) are selected. As seen in Fig. 3, the character of the helium thermal desorption spectra from the W coating changes significantly with increasing temperature T_o. For room temperature (see Fig. 3, curves 1 and 2), the spectra of helium thermal desorption from the W coating have

both the main region of helium release in the peak with $T_{max} \approx 1520$ K and another region of its release in the temperature range 450...1300 K. For an elevated temperature (see Fig. 3, curves 3 and 4), the spectra of helium thermal desorption from W coatings have the form of dependences with one main peak at $T_{max} \approx 1520$ K.

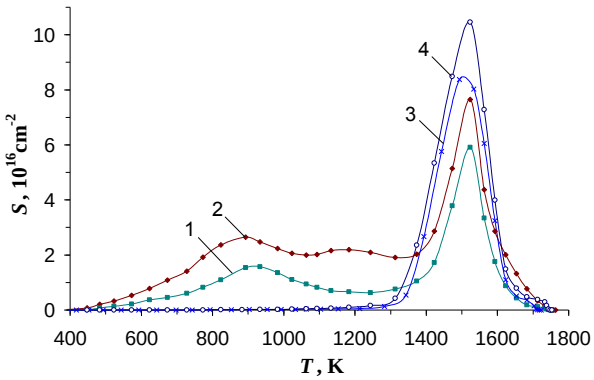

Fig. 3. Spectra of helium thermal desorption from W coating at different temperatures T_o: 1, 2 – 290 K; 3, 4 – 570 K; $\Phi=10^{17}$ cm^{-2}: 1 – 6.1; 2 – 8.3; 3 – 6.5; 4 – 7.0; α = 0.8 K/s, 20-keV He$^+$ ions

In Fig. 4 the dependences of helium concentration C in the tungsten coating at room temperature (curve 1) and elevated temperature (curve 2) on the fluence Φ of He^+ ions irradiation are shown. With an increase in the irradiation fluence, the value of C increases for both T_o = 290 and 570 K.

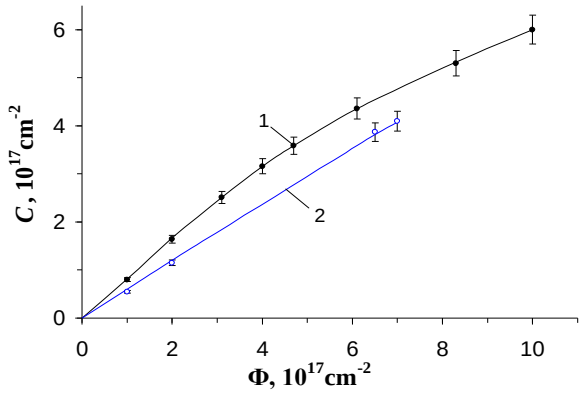

Fig. 4. Dependences of the helium concentration on the fluence of He$^+$ ions irradiation for a tungsten coating at temperatures T_o, K: 1 – 290, 2 – 570; 20-keV He$^+$ ions

In Fig. 5 the dependences of helium concentration C in the W coatings on the temperature T_o of the sample irradiated to low fluences (curves 1, 2) and to high fluences (curves 3, 4) are shown. A decrease in the value of C with an increase in T_o is observed for both low and high Φ. For T_o from the interval 290...570 K, an approximate decrease in the value of C for low Φ is 1.6 times and 1.2 times for high Φ.

For W coatings at T_o = 290 and 570 K, the He^+ ion implantation process cannot be described (within the measurement error of the C measurements) by one and the same dependence $C = f(\Phi)$ (see Fig. 4). That may be due to the different values of the helium capture coefficient η: for W coatings at T_o = 290 K it was ~0.7...0.8 (± 10%), while at T_o = 570 K it was ~0.5…0.6 (± 10%) in the same interval of fluence variation $1.0\cdot10^{17}$ cm^{-2} ≤ Φ ≤ $7.0\cdot10^{17}$ cm^{-2}.

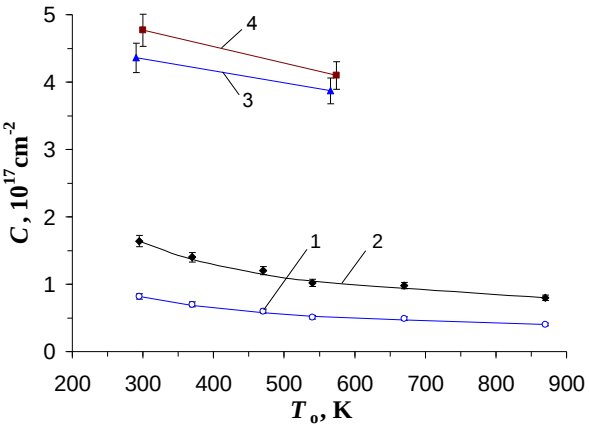

Fig. 5. The dependences $C = f(T_o)$ for W coatings irradiated by 20-keV He$^+$ ions with fluence $\Phi=10^{17}$ cm^{-2}: 1 – 1.0; 2 – 2.0; 3 – 6.5; 4 – 7.0

The authors of this work carried out a TEM study of changes in the microstructure of W coatings containing helium implanted at room temperature [11]. The formation of interstitial dislocation loops and dislocation networks in coatings irradiated with He^+ ions at $\Phi < 7.0\cdot10^{17}$ cm^{-2} was shown. The average diameter of the dislocation loops was 5.0 nm. The formation of helium bubbles was not observed. Helium bubbles were visible at $\Phi \geq 7\cdot10^{17}$ cm^{-2}. They had an average diameter of 2.5 nm and a density of $5\cdot10^{12}$ cm^{-2} for $\Phi = 7\cdot10^{17}$ cm^{-2}. In [15, 16] in metallurgical W (99.95 %) and ultrapure W (99.995%), irradiated with He^+ ions (8 keV, $1\cdot10^{14}$ cm^{-2} ≤ Φ ≤ $6\cdot10^{15}$ cm^{-2}) at T_o = 293 K the formation of interstitial dislocation loops and dislocation networks also was observed. With an increase in Φ, the average size of the loops increased to 5.0 nm, and the density was $3.5\cdot10^{12}$ cm^{-2}. Then they accumulated as entangled dislocations. It was noted in [15] that with an increase in temperature T_o the density of the loops dropped sharply and their size increased. At 873 and 1073 K for fluences of $5.6\cdot10^{14}$ cm^{-2} ≤ Φ ≤ $2.6\cdot10^{15}$ cm^{-2} the dislocation loops grew rapidly and intertwined with each other. According to [15, 16], in the above W samples irradiated by 20-keV He^+ ions to $\Phi \geq 2\cdot10^{17}$ cm^{-2} at room temperature, helium bubbles were noticeable. In [15], the temperature dependence of fluence of the He^+ ions irradiation at which the formation of helium bubbles in metallurgical W was observed, was shown: for T_o= 293 K at $\Phi \geq 2\cdot10^{17}$ cm^{-2}, at $\Phi \geq 1.5\cdot10^{17}$ cm^{-2} for T_o = 873 K, at $\Phi \geq 5.0\cdot10^{15}$ cm^{-2} for T_o = 1073 K. The helium bubbles formed at room temperature had an average diameter of about 2.5 nm and a density greater than the density of dislocation loops.

In our work, it was shown that in W and Ta coatings, irradiated to low fluences $\Phi \leq 4.0\cdot10^{17}$ cm^{-2} (for W) and $\Phi \leq 6.0\cdot10^{17}$ cm^{-2} (for Ta) at room temperature, helium

thermal desorption was observed with a peak at temperatures $T_{max} \approx 1520$ K (for W) and $T_{max} \approx 1660$ K (for Ta). According to the calculations of [11], the activation energy of helium thermal desorption from the tungsten coating was $E_a \approx 4.2$ eV in the indicated peak, close to the dissociation energy of 4.42 eV of a helium-vacancy complex of the HeV type. The dissociation of the HeV complex includes the release of helium from a vacancy with a binding energy of 3.9 eV, the migration of a helium atom through interstices to the surface with a migration energy of 0.28 eV, and He desorption into vacuum. The results of He^+ ions implantation into tungsten and tantalum coatings at elevated sample temperatures show the influence of temperature T_o on the helium capture, retention, and thermal desorption from the coatings. The helium concentrations and capture coefficients in coatings decrease with an increase in the temperature of the samples when they are irradiated with He^+ ions, although the helium thermal desorption spectra are still described by one peak dependences. Basing on saving the character (one peak) of the spectra of helium thermal desorption into vacuum for the investigated ranges of variation of the samples temperature T_o, it can be assumed that the nature of the formed radiation defects and the mechanisms of their annealing can be the same. At high temperatures, by analogy with room temperature [11], in W and Ta coatings He^+ ions irradiation to low fluences produces the following radiation defects: vacancy-type defects, helium-vacancy complexes, and interstitial dislocation loops. Thermal desorption of helium, that is caused by the dissociation of helium-vacancy complexes, migration of helium through interstices to the surface, and release into vacuum for W and Ta coatings irradiated at elevated temperatures, is enhanced. Part of the implanted helium is released during irradiation with He^+ ions.

Helium thermal desorption in vacuum at $T \le 1400$ K from a tungsten coating irradiated with He^+ ions to high fluences $4.7 \cdot 10^{17}\,cm^{-2} \le \Phi \le 1.0 \cdot 10^{18} cm^{-2}$ at room temperature is the result of gas release from other defects in the coating structure after the formation of helium bubbles observed in this work. At high-temperature irradiation of the W coating up to high fluences Φ, the temperature T_o influences on the retention and thermal desorption of helium from the coating. We should especially note the change in the spectra of helium thermal desorption. For room temperature samples and high fluences of He^+ ion irradiation, spectra of helium thermal desorption from W coatings have a multi-peak temperature region of release, which begins at temperatures $T \ge 450$ K. For the elevated samples temperature $T_o = 570$ K and a high fluence $\Phi \approx 7.0 \cdot 10^{17}\,cm^{-2}$ (see Fig. 3, curves 3 and 4), the spectra of helium thermal desorption from W coatings have one peak. The temperature at the peak maximum is $T_{max} \approx 1520$ K. The helium concentration and capture coefficient in the coating decrease with an increase of the samples temperature at the He^+ ions irradiation. Based on the single-peak nature of the spectra of helium thermal desorption for W coatings at high T_o, it can be assumed that the nature of the formed radiation defects and the mechanisms of their annealing can be the same for both high and low fluences of ion irradiation. The following radiation defects are formed: vacancy-type defects, helium-vacancy complexes, and interstitial dislocation loops. During the subsequent heating after implantation, helium thermal releases into vacuum after the dissociation of helium-vacancy complexes and helium migration through interstices to the surface of the sample.

CONCLUSIONS

The processes of helium retention of tungsten and tantalum coatings at various temperatures in the range $290\ K \le T_o \le 870$ K of tungsten and tantalum coatings irradiated with 20-keV He^+ ions to various fluences have been studied. The spectra of helium thermal desorption into vacuum have been analyzed, helium concentrations and capture coefficients in the coatings have been determined.

At low irradiation fluences $\Phi \le 4.0 \cdot 10^{17}\,cm^{-2}$ (for W) and $\Phi \le 6.0 \cdot 10^{17}\,cm^{-2}$ (for Ta), helium thermal desorption spectra from coatings of both types for $290\ K \le T_o \le 870$ K temperature range are described by dependences with one peak. The maximum desorption of helium occurs at $T_{max} \approx 1520$ K for tungsten coatings and $T_{max} \approx 1660$ K for tantalum coatings. The helium concentration and capture coefficient η for coatings of both materials decrease with temperature T_o increasing. In the same comparable intervals of T_o change, the decrease in the value of η for the tungsten coating in comparison with that for the tantalum coating is insignificant (approximately by a factor of 2 and 1.3, respectively).

At irradiation to higher fluences from the range $4.7 \cdot 10^{17}\,cm^{-2} \le \Phi \le 1.0 \cdot 10^{18}\,cm^{-2}$ (for W) and $\Phi = 1.0 \cdot 10^{18}\,cm^{-2}$ (for Ta), the spectra of helium thermal desorption from coatings at $T_o = 290$ K have one peak with a temperature in maximum $T_{max} \approx 1520$ K (for W) and $T_{max} \approx 1660$ K (for Ta). There is also a multi-peak temperature region of helium release, beginning from temperatures $T \ge 450$ K. For the elevated temperature $T_o = 570$ K of the W coating and a fluence of He^+ ions $\Phi \approx 7.0 \cdot 10^{17}\,cm^{-2}$, the spectra of helium thermal desorption from the samples have one peak. The temperature at the peak maximum is $T_{max} \approx 1520$ K.

Possible mechanisms for helium retention and thermal desorption are proposed. The helium retention in the coatings, apparently, occurs with its capture in vacancy-type radiation defects and the formation of helium-vacancy complexes with the subsequent formation of helium bubbles. During post-implantation heating of samples or during their irradiation with He^+ ions at elevated T_o, the observed helium thermal desorption is the result of the dissociation of helium-vacancy complexes, gas migration along interstices to the sample surface. Helium thermal desorption can also be a result of the release from other defects in the structure of coatings after the formation of helium bubbles and the influence of the latter on gas evolution.

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ЗАВИСИМОСТЬ УДЕРЖАНИЯ ГЕЛИЯ В ВОЛЬФРАМОВЫХ И ТАНТАЛОВЫХ ПОКРЫТИЯХ, ОБЛУЧЕННЫХ ИОНАМИ He^+, ОТ ДОЗЫ ОБЛУЧЕНИЯ И ТЕМПЕРАТУРЫ

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Исследованы процессы накопления и термодесорбции гелия для вольфрамового и танталового покрытий, осажденных на подложку из нержавеющей стали с промежуточным слоем титана, с различными температурами образцов при облучении ионами He^+ до различных доз. Обнаружены зависимости концентрации захваченного гелия и вид спектров его термической десорбции в вакуум как от дозы облучения ионами He^+, так и температуры образцов при облучении. Предложены возможные механизмы накопления и термодесорбции гелия, а также образования дефектов кристаллической решетки образцов.

ЗАЛЕЖНІСТЬ УТРИМАННЯ ГЕЛІЮ У ВОЛЬФРАМОВИХ І ТАНТАЛОВИХ ПОКРИТТЯХ, ОПРОМІНЕНИХ ІОНАМИ He^+, ВІД ДОЗИ ОПРОМІНЕННЯ І ТЕМПЕРАТУРИ

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Досліджено процеси накопичення і термодесорбції гелію для вольфрамового і танталового покриттів, осаджених на підкладку з нержавіючої сталі з проміжним шаром титану, з різними температурами зразків при опроміненні іонами He^+ до різних доз. Виявлено залежності концентрації захопленого гелію і вид спектрів його термічної десорбції в вакуум як від дози опромінення іонами He^+, так і температури зразків при опроміненні. Запропоновано можливі механізми накопичення і термодесорбції гелію, а також утворення дефектів кристалічної решітки зразків.