Degradation of the Structure and Properties of Tungsten Carbide Surface Layers under Plasma Effect

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DEPARTMENT OF MATERIALS TESTING

1. Introduction

The relevance of the research is caused by development of a fusion energy, where issues of surface modification of plasma-facing materials of the first wall and divertor of a fusion reactor are of great importance. Therefore, obtaining a reliable database on the properties of materials and other elements in contact with plasma, simulation of the plasma effect on their surfaces using an imitation installation are are highly relevant tasks of material research in modern physics when developing thermonuclear technology, including research at the Kazakhstani Material Testing Tokamak KTM.

The presence of structural materials and their impurities in the installation chamber will lead to the formation of mixed layers in the plasma-facing surfaces during redeposition due to erosion. In particular, carbon in the chamber of fusion reactors entails a number of problems. Carbon acting on the divertor surface can penetrate into the divertor volume and promote erosion and formation of porous layers on the tungsten surface and leads to tungsten carbidization.

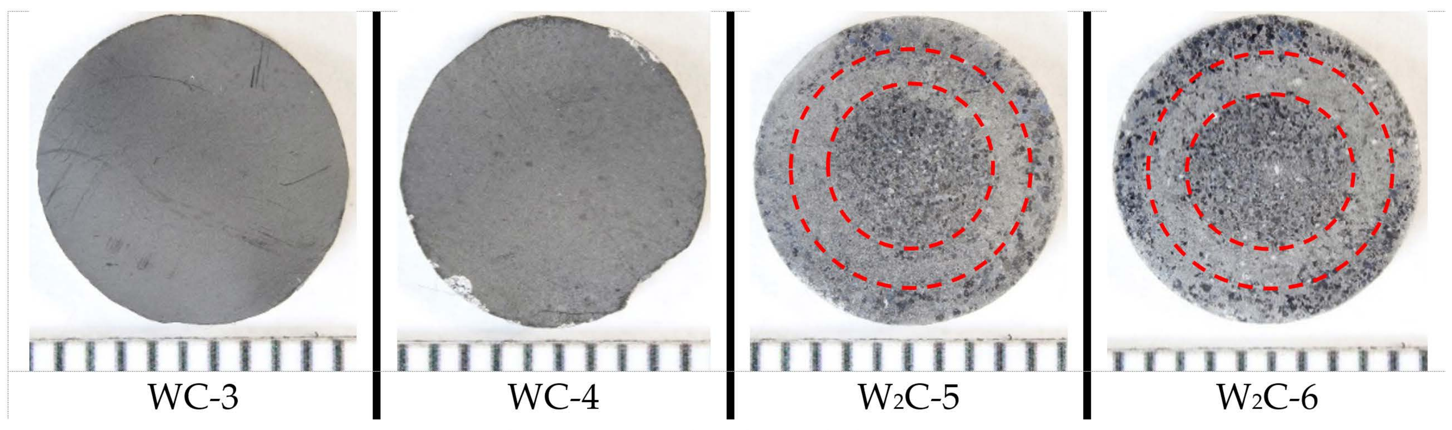
Hence, the goal of the research is to study external effects at a high energy flux and plasma irradiation on the structure and physical and mechanical properties of tungsten with a carbide layer, under conditions close to a fusion installation and the Kazakhstani Material Testing Tokamak KTM.

In this research, the results of experimental studies on the effect of plasma irradiation with a carbidized tungsten surface are obtained. Data on the structure and properties of tungsten with a carbidized near-surface layer have been obtained.

The practical significance of the research lies in the fact that the research results will be used in designing of fusion reactor components, analysis of the resource and degree of erosion effect of divertor components on plasma parameters.

2. Materials and Methods

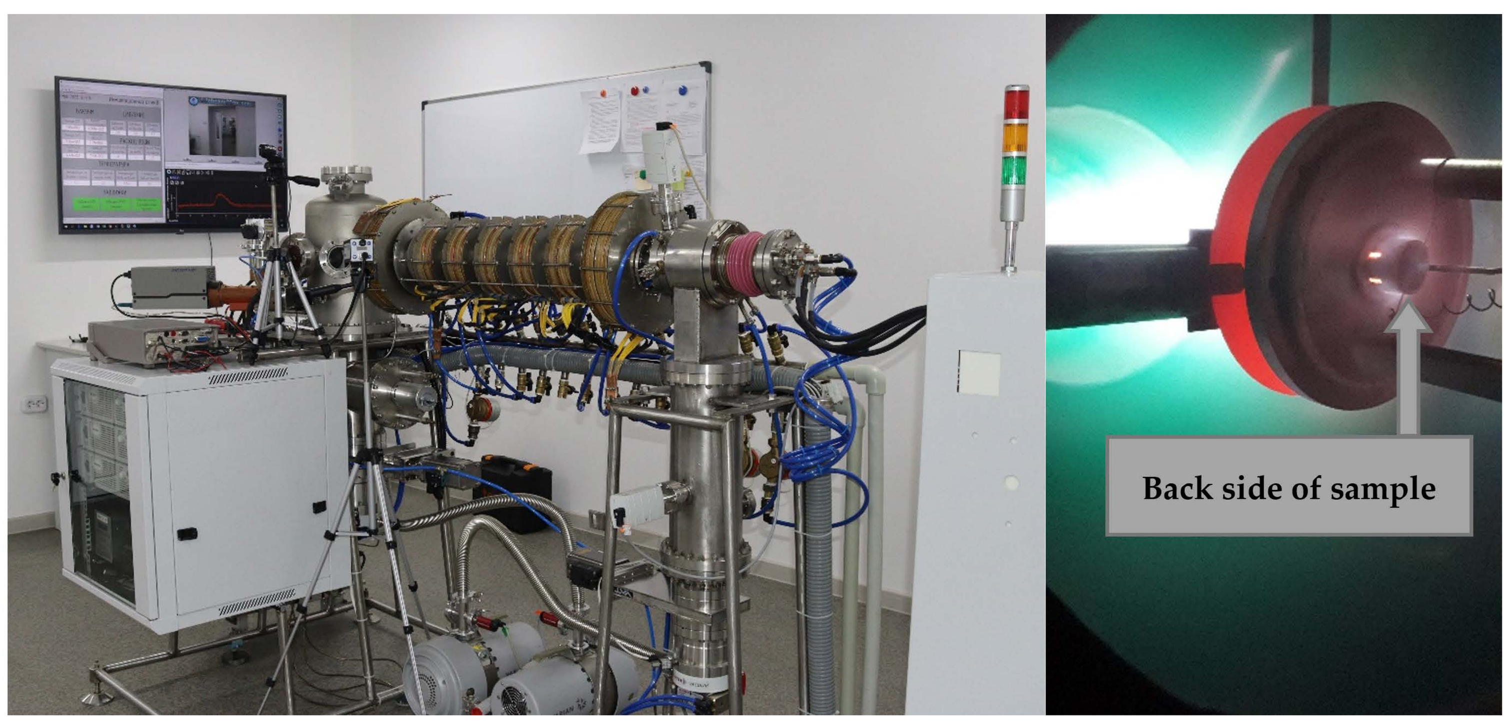
When determining the initial characteristics of the tungsten surface for research, samples were used that were not subjected to surface carbidization (Samples №1 and №2). The total number of tungsten samples in accordance with the results presented in this work is 6 pcs. Of these, 2 samples with a carbide layer of WC and 2 samples with a carbide layer of W_2C. Tungsten samples are disks cut from a VCh grade bar with a height of 2.0 ± 0.1 mm and a diameter of 10 mm (S = 0.78 cm^2) by the electroerosive method. All samples were preliminarily subjected to recrystallization annealing. The appearance of tungsten samples with a carbidized surface is shown in figure. The marking and order of the samples are indicated in accordance with the phase composition of the surface layer of the samples.



Appearance of tungsten samples after carbidization experiments

The study of the influence of the carbidized layer on the processes of interaction of helium plasma with tungsten was accompanied by material science studies of the surface of tungsten samples.The nature of damage to the tested samples was determined by visual inspection using a macrophoto of the surface. Images of the surface of the studied samples were obtained using a Canon EOS 1200D camera. The microstructure and elemental composition of the samples were studied in the topographic and compositional contrast mode using a TescanVega3 scanning electron microscope with an X-Act energy-dispersive spectral analysis attachment. X-ray diffraction patterns of the samples were taken on an Empyrean diffractometer in the PIXcel1D detector mode (scanning linear detector). The diffraction patterns were processed using the HighScore processing and search program. To identify the phase composition, the Crystallography Open Database (hereinafter referred to as COD) and the PDF-2 ICDD Release 2004 database were used.

3. Experiments



Plasma-beam setup and the back side of the sample in the chamber

To study the effect of helium irradiation on the morphology of the tungsten surface, the conditions for carrying out experimental work on a plasma-beam setup were determined. Previously, we carried out work on modeling the thermal load on plasma-reversed tungsten under the conditions of normal operation of ITER. Based on the simulation results, the temperature values were calculated during plasma irradiation to simulate the heat load, corresponding to heat fluxes in ITER, which amounted to 905 °C (corresponding to ~10 MW/m^2 stationary) and 1750 °C (corresponding to ~20 MW/m^2 pulsed). The pressure in the chamber was in the range from $1.03\cdot10^{-3}$ Torr to $1.2\cdot10^{-3}$ Torr. The helium ion flux was about ≥ 10^{22} m^{-2}s^{-1}.Experiments on the implementation of the test parameters for tungsten were carried out on a plasma-beam setup. The tungsten samples were installed in the target assembly, which was placed in the interaction chamber. Figure shows the plasma-beam setup and the sample temperature control process from the rear side of the chamber.

4. Results and Discussions

The thermal load on the surface of the samples was carried out together with the action of helium plasma. Helium was supplied to the interaction chamber, previously evacuated to a high vacuum to form a plasma-beam discharge using an electron beam. Data registration was carried out in real time. The main parameters of the experiments are given in Table.

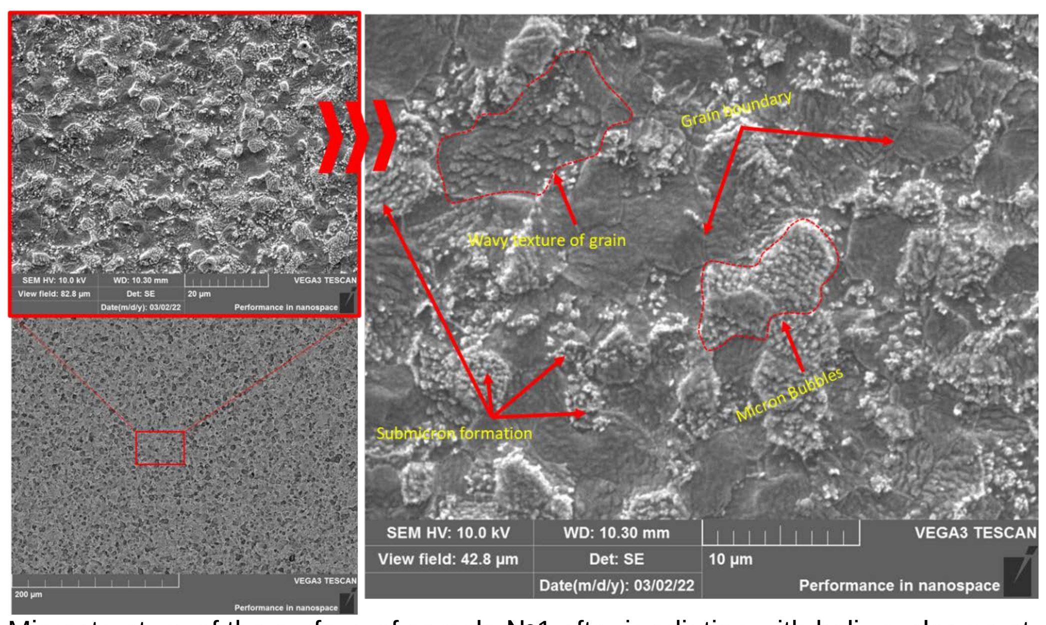
Parameters for conducting experiments on the effect of helium plasma on tungsten

Sample	Layer	Electron beam power, W	Ion current on the sample, mA	Front side temperature, °C
W-1	without	470	78	905±10
W-2	without	1635.1	100	1750±10
WC-3	WC	335.4	27	905±10
WC-4	WC	1050	66	1750±10
W_2C-5	W_2C	322.5	28	905±10
W_2C-6	W_2C	1093.4	79	1750±10

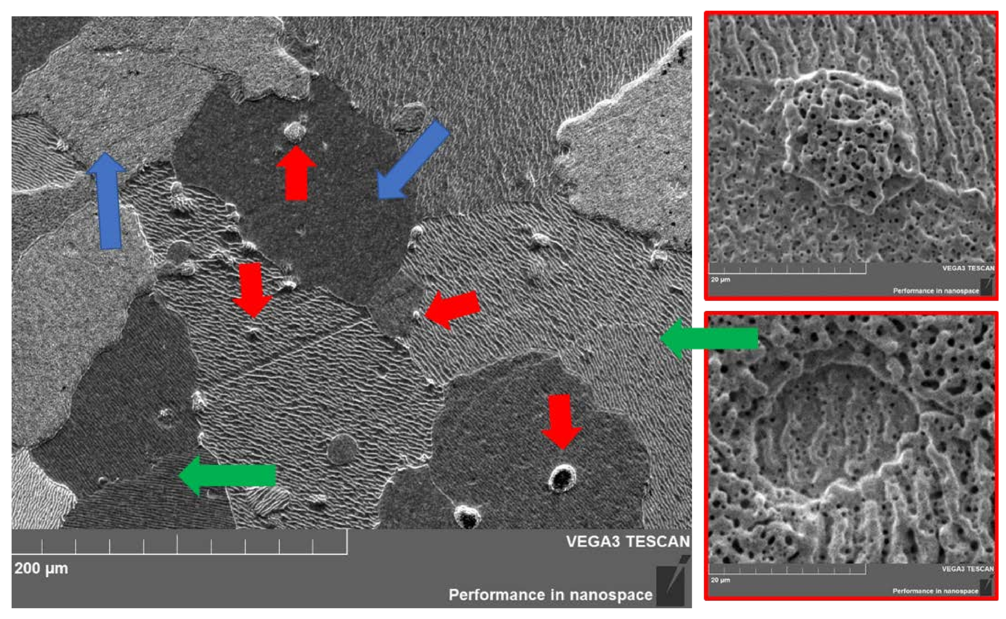
Helium Plasma Parameters Depending on Irradiation Modes

Sample	Surface temperature, °C	Ion concentration, m^{-3}	Electronic temperature, eV	Ion flux, m^{-2}2^{-1}	fluence of ions, m^{-2}
W-1, WC-3, W_2C-5	905	$2.78\cdot10^{18}$	7.39	$7.645\cdot10^{21}$	$8.26\cdot10^{25}$
W-2, WC-4, W_2C-6	1750	$2.52\cdot10^{18}$	7.69	$1.1\cdot10^{21}$	$7.48\cdot10^{25}$

For a comparative analysis, at the initial stage, two initial tungsten samples were irradiated with helium plasma at different temperatures (№1 ~905°C and №2 ~1750°C). The chemical composition of the surface of the samples after irradiation was measured by EDS analysis. The EDS method of the irradiated surface of both samples shows a characteristic pure W spectrum, in addition to a weak oxygen peak. We believe that the presence of oxygen on the surface of the sample is associated with the adsorption of water, which could occur in a vacuum chamber after irradiation with He^+ ions.



Microstructure of the surface of sample №1 after irradiation with helium plasma at ~905 °C



Microstructure of the surface of sample №2 after irradiation with helium plasma at ~1750 °C

Helium plasma irradiation of tungsten samples with a carbid layer based on WC was carried out under similar conditions as for the initial samples, i.e. at an ion energy of 2 keV and temperatures of ~905°C (№3), ~1750°C (№4). The EDS spectrum of the irradiated surface of both samples shows a typical pure W spectrum. Before irradiation, a carbon film was present on the sample surface in addition to the WC-based carbide coating, but after irradiation, the presence of carbon was not detected. Most likely, prolonged exposure to high ion energy and temperature leads to complete evaporation of carbon on the tungsten surface.

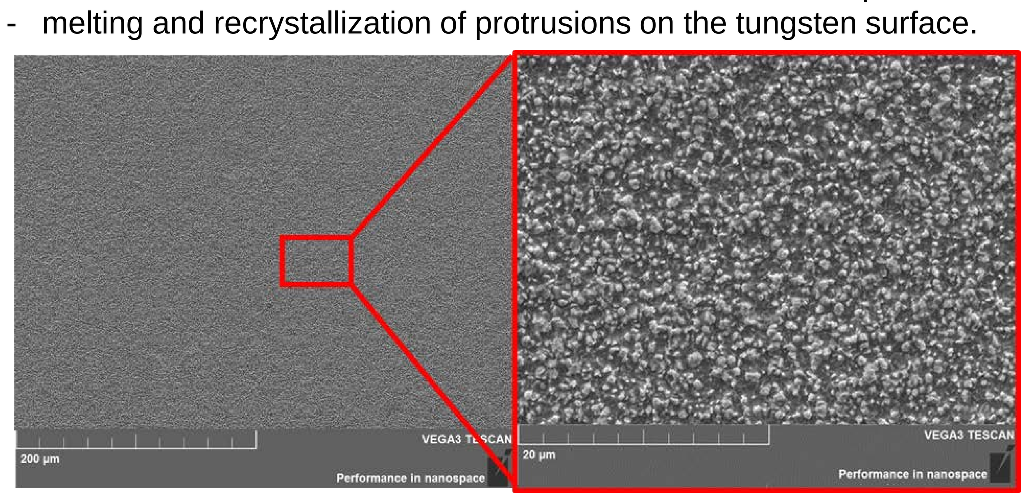
Thus, according to the obtained results of irradiation of the original samples and samples with a carbide coating based on WC, it can be assumed that the carbide coating based on WC negatively affects the properties of the tungsten surface, since after irradiation the surface of the coated samples has a similar structure with the initial sample irradiated at ~1750°C. That is, in the presence of a carbide layer based on WC, already ~905°C is sufficient for the formation of the observed coral structure. A subsequent increase in the irradiation temperature to ~1750°C for sample №4 leads to the predominance of the damaging dose over the grain orientation, and only coral protrusions directed vertically upwards are observed on the surface, which confirms the low resistance of grains with the orientation W{1 0 3}, {1 0 2}, {4 0 7}, and {2 0 3} to damage during exposure to helium. Most likely, with an increase in the exposure temperatures or ion energies for the initial sample irradiated at ~1750°C, we would observe a similar evolution of the structure, as for sample №4.

Subsequently, samples with tungsten carbide based on W_2C were subjected to helium plasma irradiation at similar parameters. The results of EDS analysis of the surface are similar to the previous samples. However, the main difference in the microstructure of the data surface of the samples is the absence of a coral structure. The nature of damage in the structure of samples with a carbide coating based on W_2C is similar to the structure of the original sample №1 irradiated at 905°C. It can be seen that the surface of sample №5, irradiated at ~905 °C, is characterized by high homogeneity with a uniformly distributed volumetric content of submicron blisters. The surface itself has a darker hue and relief morphology. The microstructure of sample №6 irradiated at ~1750°C is characterized by a more developed morphology with a large number of extended cracks over the entire surface of the sample. It should be noted that after carbidization, the surface of the coating was also characterized by a large number of cracks. However, no such cracks were observed in sample №5 after irradiation at a lower temperature. It is possible that the surface layer of the tungsten sample was embrittled due to the high loading temperature.

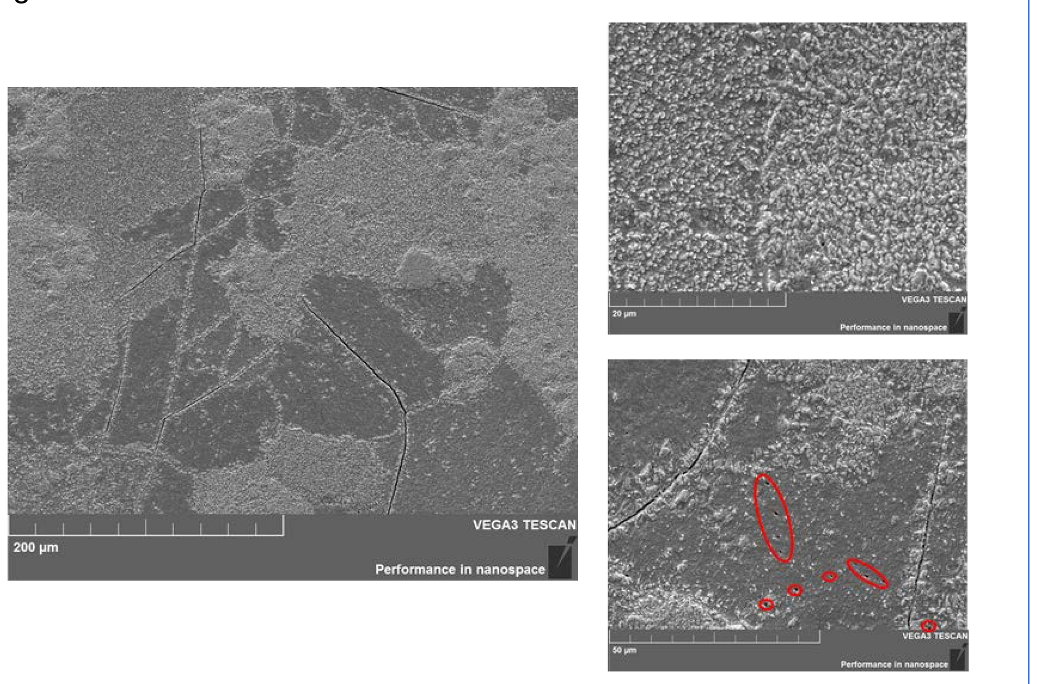
In general, according to preliminary results, the evolution of damage to the surface structure of tungsten samples can be described in the following order:

№1 → №2 → №3 → №4. Where the main characteristic markers of damage are:

- the appearance of helium blisters;
- appearance of submicron helium blisters;
- increase in the size of tungsten grains;
- coral surface structure;
- an increase in the concentration and number of nanosized pits;
- melting and recrystallization of protrusions on the tungsten surface.



Microstructure of the surface of sample №5 after irradiation with helium plasma at ~905° C



Microstructure of the surface of sample №6 after irradiation with helium plasma at ~1750° C

Thus, it can be assumed that the formation of micron and submicron helium blisters on the tungsten surface during irradiation plays a significant role in the subsequent formation of the coral structure. The subsequent growth and coalescence of these helium blisters can significantly change the shape of the exposed surface and subsurface regions of the tungsten sample. According to the results obtained, it is clear that changes in the surface structure directly depend on the composition of the carbidized coating. However, in all cases, we observe the complete absence of both carbon and phases based on it. Most likely, under the influence of high loads, as already noted, carbon and phases completely evaporate or diffuse into the depth of the sample. One of the main factors in the resistance of the tungsten surface to helium exposure is the time required for the decay of the carbidized layer. Apparently, as the irradiation temperature increases, the W_2C-based carbide layer decomposes at shorter time intervals, which leads to a more developed damaged surface. However, in order to confirm this statement and subsequently determine the mechanisms of the beneficial effect of this layer on the tungsten surface during irradiation, it is necessary to conduct experiments with a shorter duration of exposure to helium plasma. Moreover, a similar set of statistics is also necessary for tungsten samples with a WC-based carbide coating, since the issue of low surface resistance to helium plasma exposure in comparison with pure tungsten still remains open.

LABORATORY FOR TESTING MATERIALS IN A CONDITIONS OF THE FUSION REACTOR

4. Conclusions

Thus, based on the analysis of the results obtained in this paper, we can draw the following main conclusions:

Experiments were carried out on the effect of helium plasma on tungsten with carbidized and original surfaces at temperatures of ~905 °C and ~1750 °C. The concentration, electron temperature, ion flux and fluence of helium ions during tungsten irradiation are estimated. The ion fluence during irradiation was $7.48\cdot10^{25}\div8.26\cdot10^{25}$ ions/m^{-2}, which corresponds to the expected operational parameters in ITER. Irradiation of the tungsten surface with an ion energy of 2 keV leads to severe damage to the tungsten surface. A similar evolution of surface morphology due to irradiation with pure He+ ions at elevated temperatures was expected and is consistent with other similar studies.

The WC-based tungsten carbide layer, according to the results obtained, has a weaker resistance to helium damage than pure tungsten under plasma irradiation conditions. An increase in the plasma exposure temperature leads to more severe damage to the surface and the formation of melted and recrystallized protrusions.

According to the obtained results, the W_2C-based tungsten carbid layer has a higher resistance to helium exposure, which is maintained even with an increase in exposure temperature. However, high-temperature exposure at 1750°C leads to embrittlement of the surface layer.

Analysis of the results showed that, depending on the type of carbidized layer, the roughness values differ greatly. It has been established that the surface of samples with the W2C phase is more prone to erosion than the surface with the WC phase.

According to the results of microstructural analysis, it can be assumed that the carbide coating based on WC negatively affects the properties of the tungsten surface, accelerating the formation of a coral structure at low temperatures of irradiation with helium plasma and leads to significant changes in the morphology of the tungsten surface.

Acknowledgement

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