Chapter 7

In Situ Renewable Coating of Boron Carbide (B_4C) for Plasma Materials for Plasma-Technological and Fusion Devices

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Additional information is available at the end of the chapter

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Abstract

The application of the in situ renewable protecting boron carbide (B_4C) coating can prevent plasma-facing materials of plasma technology and thermonuclear devices from plasma irradiation and by this means prevents their destruction and plasma contamination by materials of their erosion. At the same time, the regimes and conditions of high adhesive deposition of B_4C on tungsten and the B_4C coating ability to withstand the thermal cycling and high-power density irradiation by plasma ions have not been investigated yet. The chapter considers the results of ion irradiation and thermal cycling of boron carbide coating on tungsten sample in Stand for Coating Deposition and Material Testing—CODMATT (NRNU MEPhI) and plasma irradiation during a plasma disruption in fusion device—T-10 tokamak (NRC "Kurchatov Institute"). Boron carbide coating withstands the thermal cycling and high-power density irradiation by plasma ions. It retains uniformity and adhesion to tungsten and protects it from direct plasma interaction for temperatures up to melting point of tungsten. The retaining of uniform coating in contact with tungsten substrate allows renewing the coating on its surface even after high-energy plasma loads.

Keywords: investigation, boron carbide, coating, ion cycling, plasma

1. Introduction

Tungsten is used as the plasma-facing material of the contemporary fusion devices and for the divertor of the International Thermonuclear Experimental Reactor (ITER) under construction.

Last year investigations show that high-power density plasma irradiation initiates cracking of tungsten surface, blister formation, flaking, macroscopic particle emission, etc. (see, for instance, [1, 2]). These phenomena can cause accelerated destruction of tungsten tiles of ITER divertor.

It was shown [3] that application of the in situ renewable protecting boron carbide (B_4C) coating can prevent plasma-facing materials of plasma technology and thermonuclear devices from plasma irradiation and by this means prevents their destruction and plasma contamination by materials of their erosion. B_4C coating has high melting temperature (2800 K). The erosion rate of B_4C changes only slightly up to 1400°C and appears to be much less than the erosion rate of dense graphites under similar irradiation conditions [4]. The B_4C coating being used as the protective coating of the plasma-facing components of fusion devices will not have thickness higher than 30–50 μm; therefore, accumulation of remarkable amount of tritium in the plasma-facing materials is prevented.

Investigations showed that near stoichiometric B_4C coating can be deposited in high-temperature plasma providing total dissociation of the molecules of initial substance. The conclusion was confirmed experimentally, when B_4C coating had been successfully deposited in plasma devices (PISCEC-B [5] and tokamak T11-M [6]). The vapor of nontoxic, nonexplosive, and nonhazardous carborane ($C_2B_{10}H_{12}$) was used as the initial substance for coating deposition in the course of regular discharge in the plasma devices.

At the same time, some of the important aspects of the B_4C coating application in fusion devices have not been investigated yet. Among them, there are the regimes and conditions of high adhesive deposition of B_4C on tungsten and the B_4C coating ability to withstand the thermal cycling and high-power density irradiation by plasma ions.

The results of these topics' investigations are the subject of this presentation. The paper considers the results of ion irradiation and thermal cycling of boron carbide coating on tungsten sample in Stand for Coating Deposition and Material Testing, CODMATT (National Research Nuclear University (MEPhI)) [7], and plasma irradiation during a plasma disruption in fusion device (T-10 tokamak, National Research Center "Kurchatov Institute," Moscow).

2. Boron carbide deposition and testing in the laboratory stand

The method of B_4C coating deposition on tungsten, reproducing in general terms the coating regime in conditions of fusion devices, was developed on the CODMATT facility. The coating was formed by atoms of boron and carbon sputtered by plasma ions from corresponding targets. For tests in T-10, a coating of boron carbide was deposited on tungsten samples measuring 15 × 15 × 1 mm^3, 5 μm in thickness. The photograph of the coating is shown in **Figure 1**.

It is seen that coatings had a smooth surface, cracks, and peeling and other signs of a violation of adhesion to the substrate were not observed. Using X-ray energy dispersive spectrometry (EDS), it was found that the coating practically corresponds to the stoichiometric composition of boron carbide (B76%, C22%).

Several cycles of pulsed irradiation of tungsten samples coated with hydrogen ions on the CODMATT facility were carried out. In the first cycle, the irradiation flux power density was

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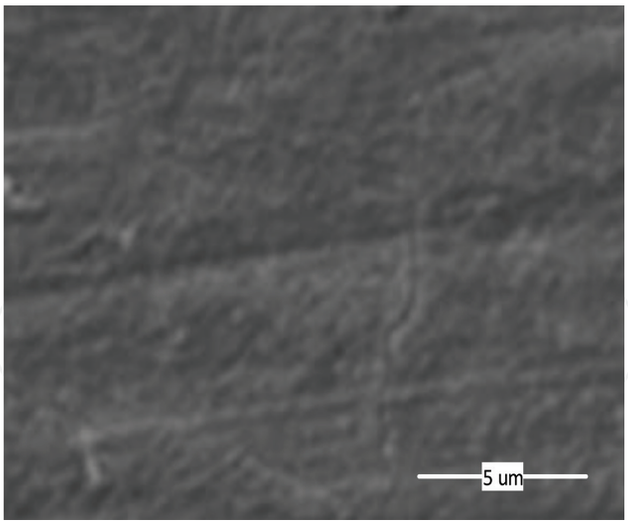

Figure 1. Boron carbide coating on tungsten substrate.

0.3 MW/m^2. The energy of the irradiating ions E_i = 10 kV/at, the duration of irradiation τ = 0.6 s, the total cycle time t = 300 s, and the temperature of the sample during the test varied in the range T = 100–400°C. One thousand pulses were conducted. The second cycle was performed using irradiation flux power density which equals 5 MW/m^2 (E_i = 15 kV/at, W = 5.0 MW/m^2, τ = 0.4 s, t = 30 s, T = 600–900°C). One hundred pulses were conducted. During the third cycle irradiation, flux power density was elevated up to 5 MW/m^2 (E_i = 15 kV/at, W = 5.0 MW/m^2, τ = 0.6 s, t = 30 s, T = 700–1200°C). Fifty pulses were conducted.

None of the test cycles led to the peeling of the coatings, the appearance of caverns, cracks, and changes in the composition of the coating. Noticeable traces of etching of the surface appeared only after the last cycle.

3. T-10 boron carbide coating tests in a T-10 tokamak

A tungsten sample with a coating located on the diagnostic input of the T-10 tokamak was exposed to plasma irradiation during a plasma disruption.

Part of the sample subjected to the most intense plasma irradiation melted out and left the sample (**Figure 2**). Surface morphology analysis of the remaining part of the sample after the irradiation was done on a TESCAN VEGA 3 scanning electron microscope (SEM). Five areas are distinctly visible on a sample around the molten part: the area of tungsten that melted but stayed from the sample; the area of melted coating, the upper level of which collected in separate globules; the area of partial coating melting; the area of cracked non-molten coating; and the area covered by the sample holder.

An SEM image of the molten tungsten area of the sample is shown in **Figure 3**. Distinct micro-crystals are visible which were formed due to solidification of the molten tungsten, as well as pores and trenches on the tungsten grain boundaries. Measurements performed by EDS have not shown the presence of boron and carbon in a layer about 0.8 μm thick, which indicates complete or almost complete removal of coating from the surface of molten tungsten.

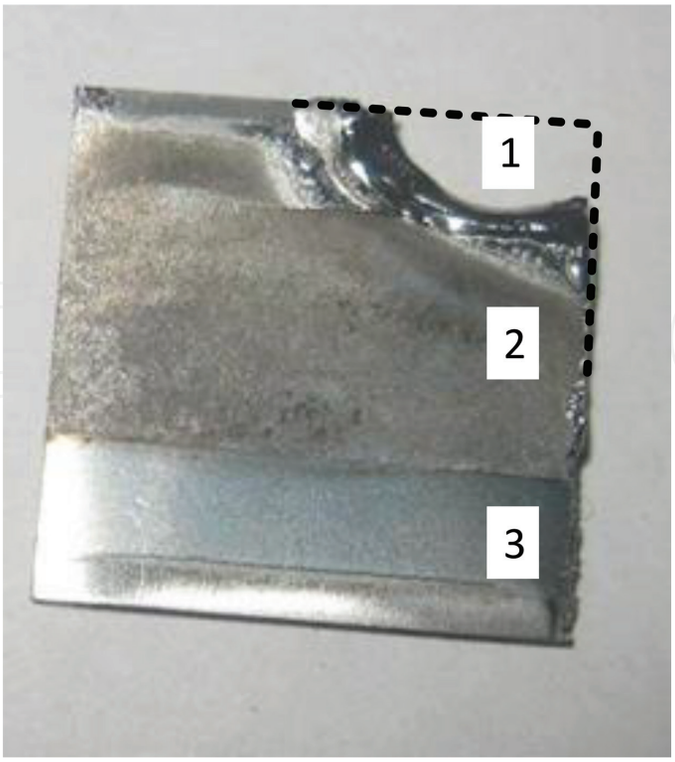

Figure 2. Sample after tests: (1) tungsten melting area, (2) irradiated part of the coating, and (3) sample holder area.

Area of a molten coating is shown in **Figure 4**. Multiple globules with sizes of tens of micrometers are of notice on this area, which were formed, supposedly, under surface tension force on the upper part of the coating. The coating left on the surface of the sample is a uniform layer with small cracks, but no signs of exfoliation.

For measuring the thickness of coating left on a sample, a cross section was performed using a FEI Scios DualBeam device with a focused ion beam method. The cross section went through one of the cracks on the area free of globules (**Figure 5**).

A substrate, an intermediate layer, the remaining part of the coating (approx. 1.8 μm), and a crack can be seen on the cross section of the sample. The crack is visible in the tungsten substrate, which indicates that this and, supposedly, other cracks were initiated in the substrate, with cracks forming during the cooling of tungsten at temperatures lower than boron carbide freezing point and going on the crystal grain boundaries, as seen in **Figure 5**. An SEM image of coating remaining on the sample, done in a large magnification (**Figure 6**), shows its porosity, with pores that probably form during the solidification of the molten coating.

Globules and coating remaining on the surface of the substrate, according to EDS analysis, included approximately identical parts of boron and carbon. Their ratios ranged from 1:1 to 1.2:1 boron to carbon; however, it is known that boron carbide structure remains even at a ratio of B:C = 1:1. Some percentage in globule and remaining coating content was stainless steel components (Fe, Ni, Cr) and oxygen, which were probably the tokamak's working gas impurities introduced into the coating during plasma irradiation.

The area of a partially molten coating is shown in **Figure 7**. A number of characteristic areas can be observed on this part, such as initial coating with cracks, molten parts, and points of

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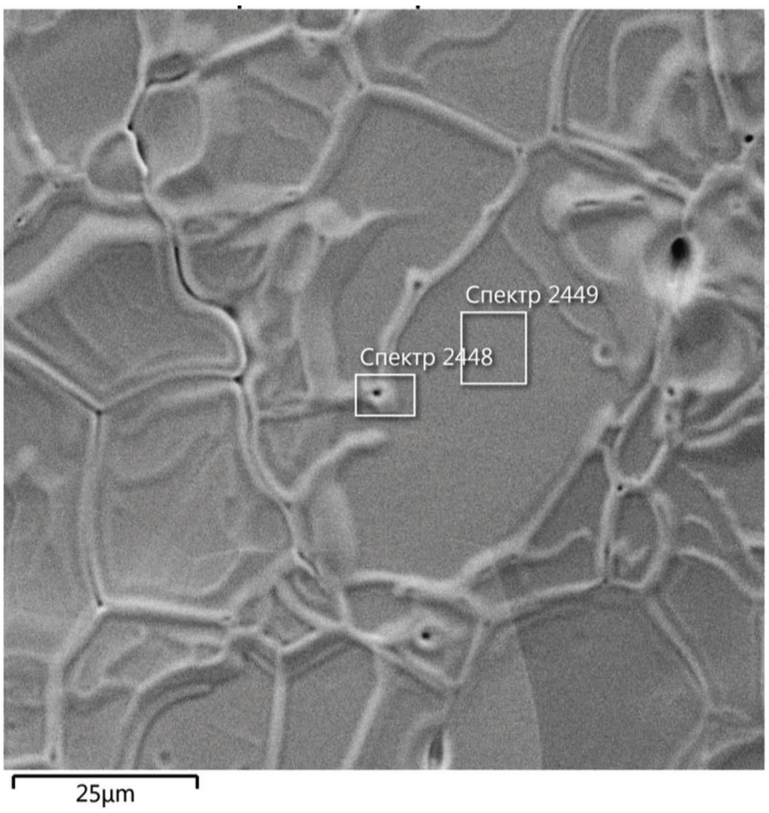

Figure 3. Molten tungsten area of a sample.

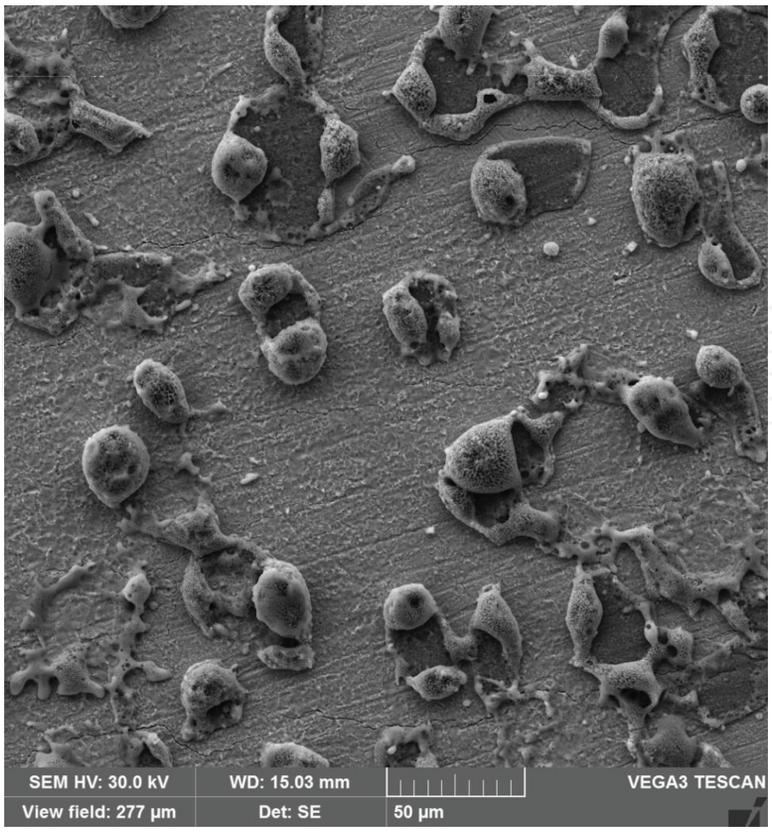

Figure 4. Area of a molten coating and globules.

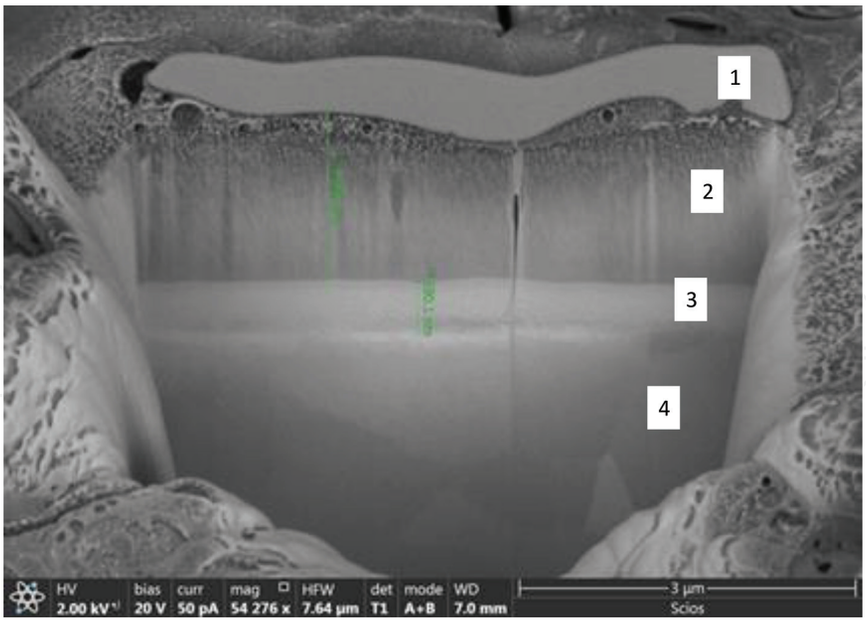

Figure 5. Cross section of a globule-free sample area. (1) Pt technical layer, (2) coating, (3) intermediate layer, and (4) substrate.

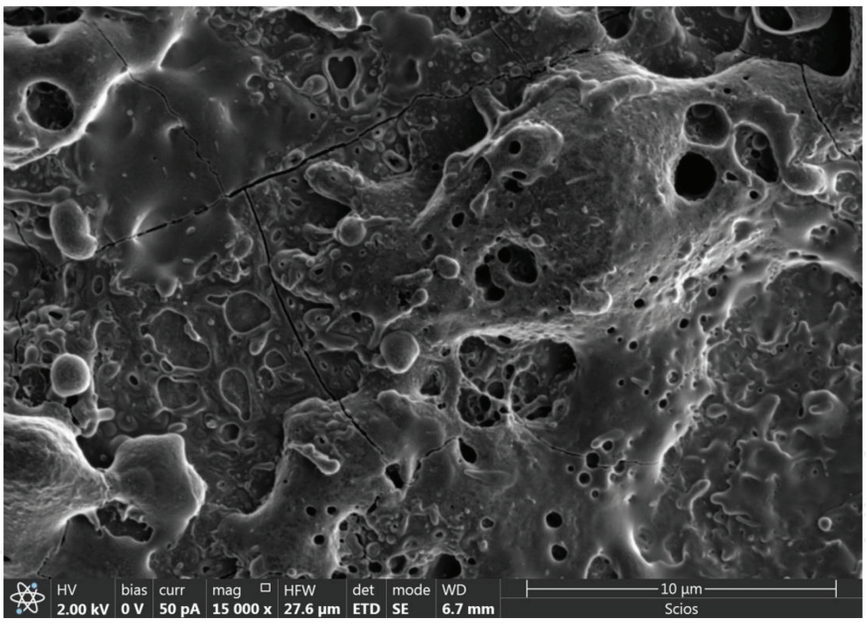

Figure 6. Area with a crack on the remaining coating.

molten coating accumulation, with the latter not collecting into globules, but rather collecting on the peripherals of molten areas. The size of molten parts ranges from 5 to 200 µm. The ratios of boron to carbon in these areas were 2.3:1, 2.6:1 and 3.1:1, respectively. Thus, it can be stated that the content of the coating on this area of the sample was much closer to the original stoichiometry than on the previous one.

The last characteristic area on a sample is shown in **Figure 8**.

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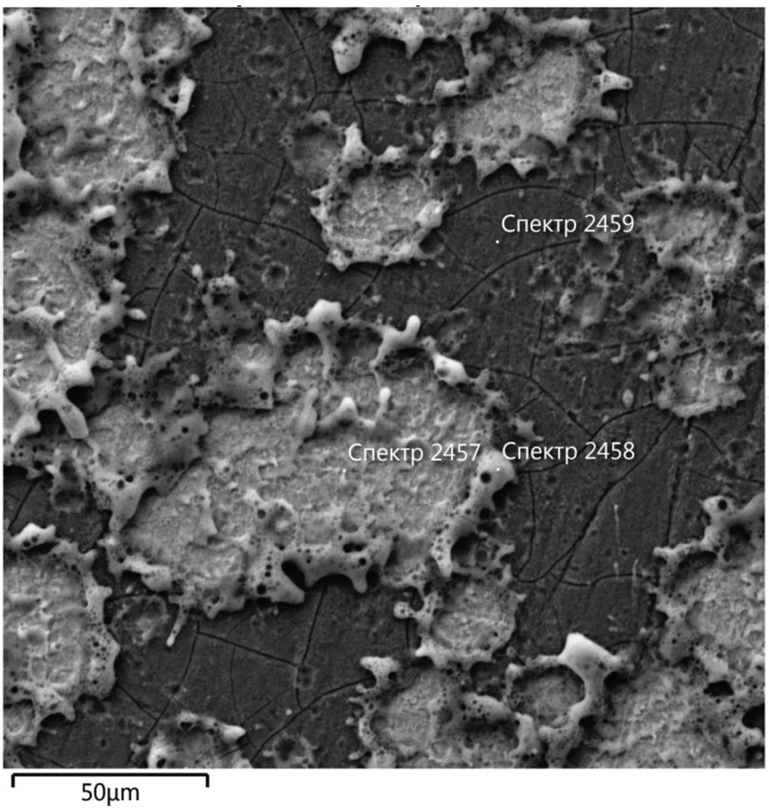

Figure 7. Area of a partially molten coating.

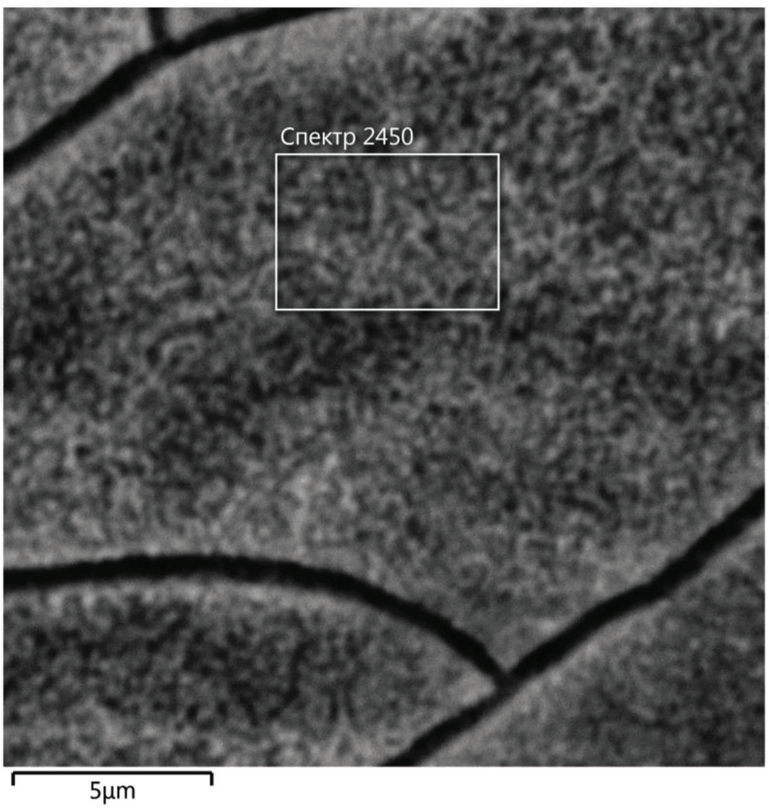

Figure 8. Area of nonirradiated coating with cracks.

There was no trace of coating melting due to plasma interaction. There was also no indication of change in the coating thickness, and EDS analysis did not show any change in element content. The appearance of cracks shows that destruction of tungsten occurred even on this part of substrate, which was positioned next to a holder and heated less than the rest of the sample.

4. Measurement of gas trapped in the sample

Analysis of ion irradiation parameters during plasma disruption has shown that the coating was irradiated by ions with an average energy of approx. 100 eV with a mean dose of approx. 1×10^{18} at/cm^2. Measurement of gas content trapped in the coating during plasma irradiation was done using a method of thermal desorption spectroscopy (TDS). The analysis was performed for two parts of the samples: a partially molten area (**Figure 7**) and an intact area (**Figure 8**). TDS analysis of both these parts is shown in **Figure 9**. The amount of trapped deuterium is 9×10^{14} at./cm^2 for the area with a partially molten coating and 2×10^{14} at./cm^2 for the area with cracking.

The temperature of the maxims and high-temperature edge of the spectrum of deuterium thermal desorption from the coating irradiated in a tokamak suggests that plasma ions introduced into the coating during disruption, when the coating temperature was much higher, could not stay in the coating. Deuterium was trapped in the coating that was already cooled and crystallized, at a relatively low temperature—lower than thermal desorption maxima temperature. Moreover, based on deuterium trapping occurring at a significantly higher

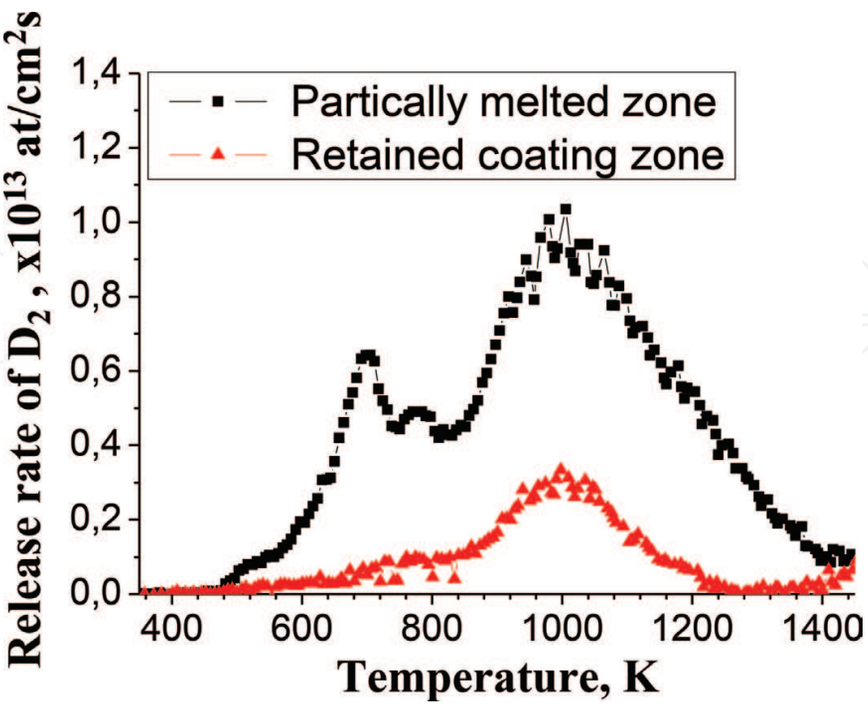

Figure 9. TDS spectra of deuterium desorbed from the sample irradiated in T-10 tokamak.

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rate in an area with a partially molten coating than in an area with cracked coating, one can assume that the amount of high-temperature traps in boron carbide increased with the increase of coating's heating temperature and the loss of some boron in a film.

5. Dependence of coating morphology change on temperature and power density of irradiation

The results obtained in the experiment allowed determining the heat flux distribution of plasma irradiation on the surface of the coating to heat part I of the sample to the temperature of ≥3600 K, causing melting of tungsten, and heat part II of the sample to T ≥ 2800 K, at which the coating melts. The duration of plasma interaction with the sample was taken as 70 ms based on the measurements of the dependence of ion flux on time. Temperature calculation was done using a COMSOL Multiphysics package for two cases: irradiation from the coating side and irradiation from the substrate side. The calculations have shown that temperature difference does not exceed 100 K for both cases.

Temperature distribution on a sample surface, as well as heat loads in corresponding areas, is shown in **Figure 10**.

A graph of temperature change for coating areas 1 s after disruption based on their distance from the molten part is shown in **Figure 11**. As can be seen, maximum temperature eats the distance of approx. 5 mm away from molten tungsten which is achieved during plasma

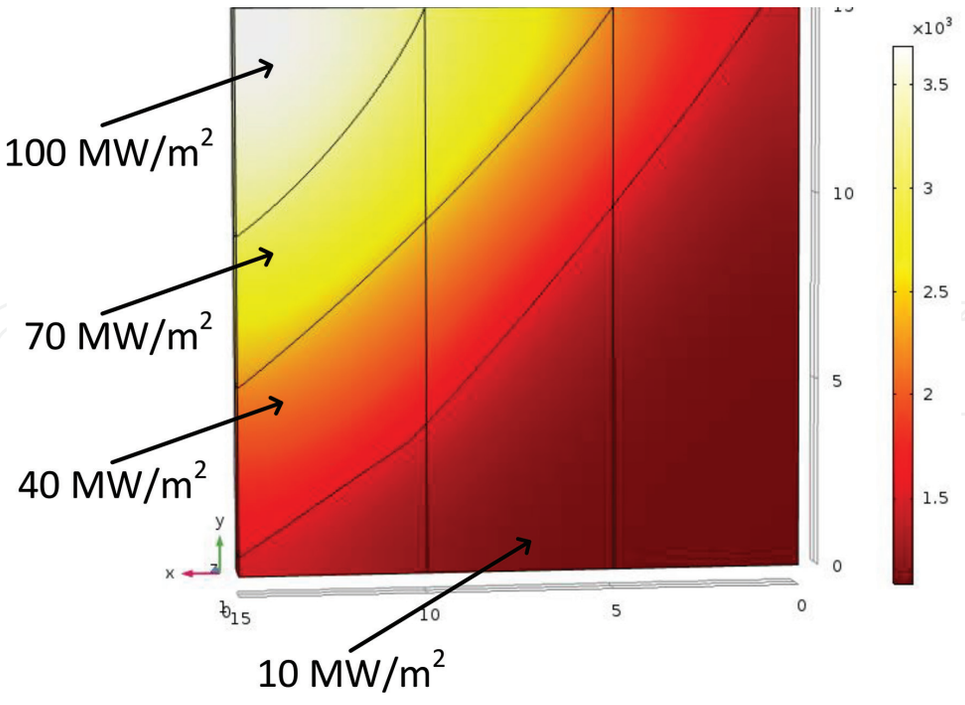

Figure 10. Maximum temperature distribution in each area and heat loads in corresponding parts of the sample (calculations). Thin lines show least distance from solidified molten tungsten for which temperature changes 1 s after irradiation are shown in Figure 11.

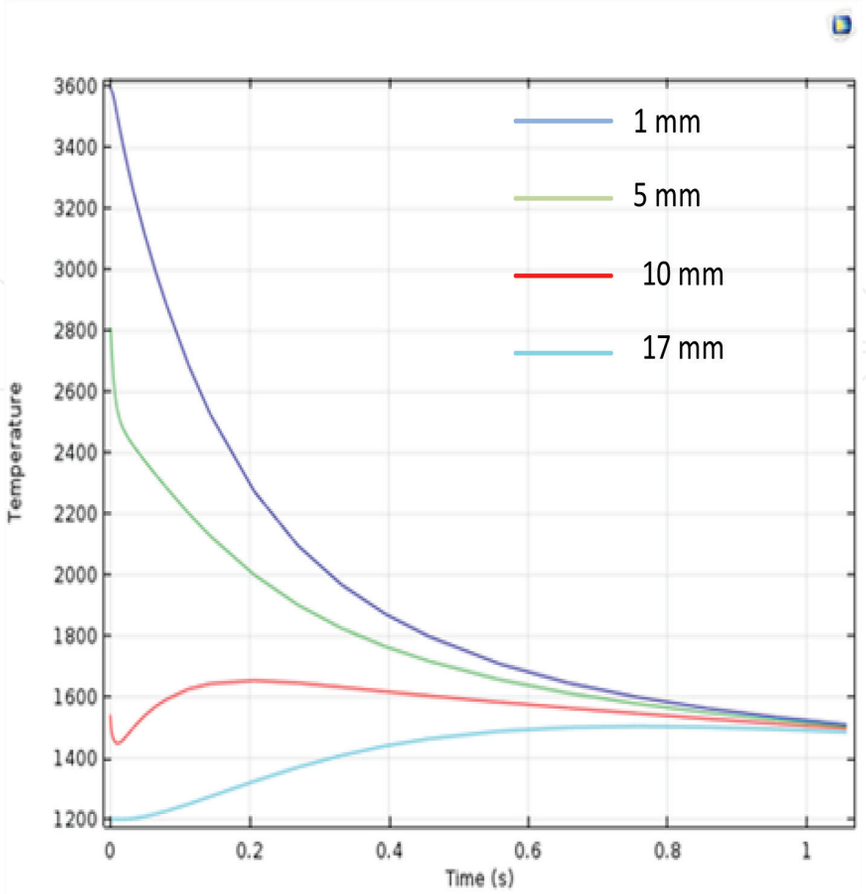

Figure 11. Temperature change dynamics of sample areas depending on their distance from molten tungsten.

irradiation. At the same time, during cooling (with radiation as a main mechanism), some heat stored in the sample spreads along its volume, leading to further increase of temperature on parts of the sample further away from the molten area 1 s after the end of irradiation.

Comparison of data allows determining approximate temperature maxima on the boundaries of each area and, as such, links the change of coating structure with temperature and power density of plasma irradiation.

Intact coating area. $T < 2000$ K.

Partially molten coating area. Temperature range 2000–2500 K.

Molten coating area. Temperature range 2500–3695 K.

Molten tungsten area. $T > 3695$ K.

6. Conclusion

The boron carbide coating deposition and testing were performed on the CODMATT facility. 5-μm-thick B_4C coating tungsten samples for experiments in T-10 tokamak were formed by atoms of boron and carbon sputtered by plasma ions from corresponding targets. The

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adhesion of the boron carbide coating to tungsten was tested by cycles of high-temperature hydrogen ion irradiation. During the last cycle, flux power density was elevated up to 5 MW/m^2 (E_i = 15 kV/at, W = 5.0 MW/m^2, τ = 0.6 s, t = 30 s, T = 700–1200°C). Ion irradiation did not lead to coating exfoliation, the appearance of caverns, cracks, and changes in coating composition.

The boron carbide coating on tungsten sample was subjected to intense plasma irradiation during a plasma disruption in the T-10 tokamak. Power density of an ion flow on the sample reached 100 MW/m^2, with the irradiation time being 70 ms.

The results of plasma irradiation in a tokamak can be summed up as follows:

- In the area with a temperature ranging from 2500 to 3695 K, most of the coating melts and forms globules. The structure of globules and the remaining coating layer approx. 1.8 μm thick retains boron carbide crystal lattice, but the concentration of boron is reduced to B:C = 1:1. No cracking or exfoliations are observed on this or any other areas. Part of the coating left on the surface is uniform, retaining adhesion to tungsten, which allows protection of tungsten from plasma irradiation.
- On the areas with a temperature range 2000–2500 K, partial melting is observed. The nature of melting suggests heating of individual coating areas up to high temperatures during plasma irradiation. Coating element ratio on this area approximate stoichiometric.
- At temperatures less than 2000 K, the coating is left intact on the surface of tungsten. Coating element ratio is close to stoichiometric.

The performed experiments allow making the following conclusions:

- Boron carbide coating withstands the thermal cycling and high-power density irradiation by plasma ions.
- Boron carbide coating retains uniformity and adhesion to tungsten and protects it from direct plasma interaction for temperatures up to melting point of tungsten.
- The retaining of uniform coating in contact with tungsten substrate allows renewing the coating on its surface even after high-energy plasma loads.

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References

[1] Krieger K, Geier A, Gong X, Maier H, Neu R, Rohde V. ASDEX upgrade team, erosion and migration of tungsten employed at the main chamber first wall of ASDEX upgrade. Journal of Nuclear Materials. 2003;**313-316**:327-332

[2] Buzi L, de Temmerman G, Unterberg B, Reinhart M, Litnovsky A, Philipps V, et al. Influence of particle flux density and temperature on surface modifications of tungsten and deuterium retention. Journal of Nuclear Materials. 2014;**455**:316-319

[3] Krasheninnikov SI, Smirnov RD, Rudakov DL. Dust in magnetic fusion devices. Plasma Physics and Controlled Fusion. 2011;**53**(8):083001

[4] Begrambekov L, Buzhinsky O, Gordeev A, Miljaeva E, Leikin P, Shigin P. TDS investigation of hydrogen retention in graphites and carbon based materials. Physica Scripta. 2004;**T108**:72-76

[5] Maier H, Greuner H, Balden M, Böswirth B, Lindig S, Linsmeier C. Erosion behavior of actively cooled tungsten under H/He high heat flux load. Journal of Nuclear Materials. 2013;**438**:S921-S924

[6] Begrambekov LB, Buzhinskiy OI. Boron carbide properties and its use as protective coating for plasma facing components. The questions of atomic science and technique. 2016;**4**:14. [in Russian]

[7] Ayrapetov AA, Begrambekov LB, Dyachenko MY, Evsin AE, Grunin AV, Kalachev AM, et al. Stand for coating deposition and coating/materials testing. Journal of Physics: Conference Series. 2016;**700**(1):012041