



Experimental modelling of plasma–graphite surface interaction in ITER

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Abstract

The investigation of graphite erosion under normal operation ITER regime and disruption was performed by means of exposure of RGT graphite samples in a stationary deuterium plasma to a dose of 10^{22} cm⁻² and subsequent irradiation by power (250 MW/cm²) pulse deuterium plasma flow imitating disruption. The stationary plasma exposure was carried out in the installation LENTA with the energy of deuterium ions being 200 eV at target temperatures of 770°C and 1150°C. The preliminary exposure in stationary plasma at temperature of physical sputtering does not essentially change the erosion due to a disruption, whereas exposure at the temperature of radiation enhanced sublimation dramatically increases the erosion due to disruption. In the latter case, the depth of erosion due to a disruption is determined by the depth of a layer with decreased strength. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

For imitation the expected ITER-operating conditions – combination of normal operation with plasma disruptions – the samples of graphite, firstly, were exposed to high doses (up to 10^{22} cm⁻²) in a stationary plasma, then they were irradiated by the pulsed high power deuterium plasma.

2. Experimental technique

Samples of RGT-type graphite characterized by a high heat conduction, due to the presence of Ti at about 7 wt% in the form of TiC [1], were used in the experiments. The samples of graphite were 3 cm in diameter and were fastened to the target holders with a metallic pin screwed into the rear-side. Six samples of RGT graphite were exposed at once in a deuterium plasma in

the LENTA facility with a beam plasma discharge at irradiation temperatures of 770°C (sample 1) and 1100°C (sample 2) to a dose of 10^{22} cm⁻². The density of current to the target was 5×10^{17} cm⁻² s⁻¹. The energy of deuterium ions was 200 eV.

Later other samples were irradiated in the MKT-electrodynamical plasma accelerator at a deuterium plasma density of about 10^{15} cm⁻³ and at the maximal ion energy of 1–2 keV. The energy flux density was 0.25 MJ m⁻². The pulse duration was 60 μs and the number of pulses was equal to six; i.e. the total energy flux density to the target was 1.5 MJ m⁻².

The erosion and sublimation products were collected upon basalt filter fibres located in the shadow of the plasma flux around the exposed target. The disposition of a graphite target and that of the erosion product collectors is shown in Fig. 1. The numbers 1 and 2 designate those areas of the basalt cloth, where the probes were taken.

After exposure the targets in a stationary plasma, as well as after an additional irradiation by the pulsed plasma flux, the studies of the graphite surface were done with SEM. Transmission electron microscopy and

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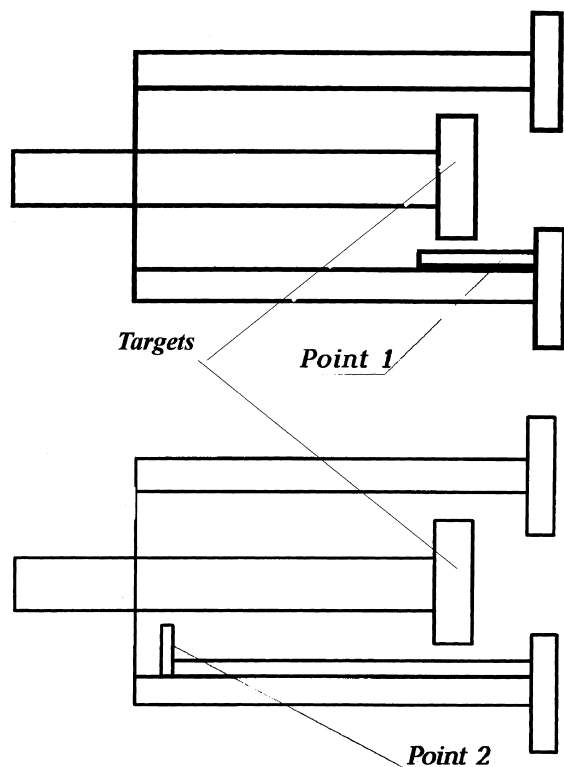


Fig. 1. The graphite target and the erosion product basalt collectors (1), (2).

scanning microscopy techniques were used, as well as observations with an optical microscope, for studying the graphite erosion products and sublimation. Some layers, including 2–3 layings of fibres with the graphite particles deposited on them, were separated and studied with the transmission electron microscope. The chemical composition of the target surfaces was analyzed by the Auger electron analysis with the successive etching of the layer by Ar^+ -ions and by the Rutherford backscattering. Elastic recoil detection was used for determining the deuterium distribution profiles in the irradiated graphite targets after exposure in the stationary and pulsed deuterium plasmas.

3. Experimental results

3.1. RGT graphite surface microstructure after a plasma effect

Fig. 2 illustrates a characteristic structure of the RGT graphite surface after its bombardment by 200 eV D^+ -ions at 770°C. Upon the graphite surface sputtered by a high deuterium ion doses, a relief characteristic for the growth of whiskers was developed. As is known [2], the condition for the emergence of whiskers is the

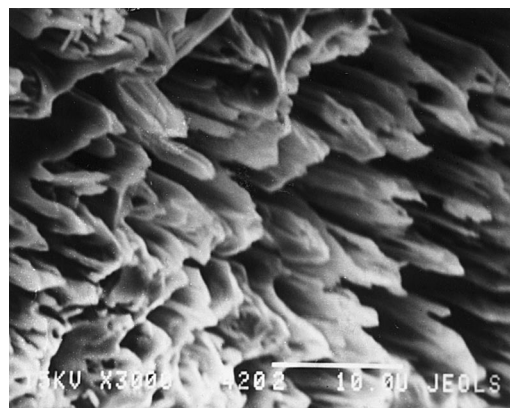


Fig. 2. Electron microphotographs of the RGT graphite (sample 1) after irradiation at the temperature 770°C by 200 eV D^+ -ions up to a dose of 10^{22} cm^{-2} . The scale length corresponds to 10 μm .

presence of an impurity in the material under sputtering. In this case, it is related with different sputtering yields for graphite together with the particles of TiC present in it. The size of the produced whiskers is about 10 μm . Slits are seen between whiskers. The formation of such a structure upon the graphite surface is a result of interaction among the processes of sputtering, redeposition, and surface diffusion. Relief of the surface is brush-like.

The subsequent effect of six high power, deuterium plasma pulses results in an inessential change in the surface structure (Fig. 3). Upon the surface, the configurations known as “balls with the legs” [3] are produced. From the comparison of the surface structures, which have developed under the influence of stationary (Fig. 2) and successive stationary and pulsed plasma (Fig. 3), we can conclude that the surface formed by deuterium ions

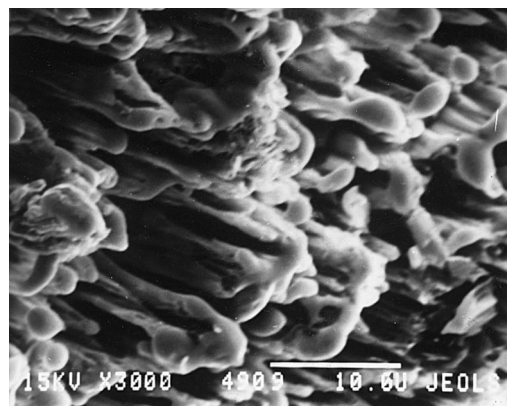


Fig. 3. Electron microphotographs of the RGT graphite (sample 1) after irradiation by the stationary and pulsed (0.25 MJ m^{-2} , 60 μs , 6 pulses) plasmas. The scale length corresponds to 10 μm .

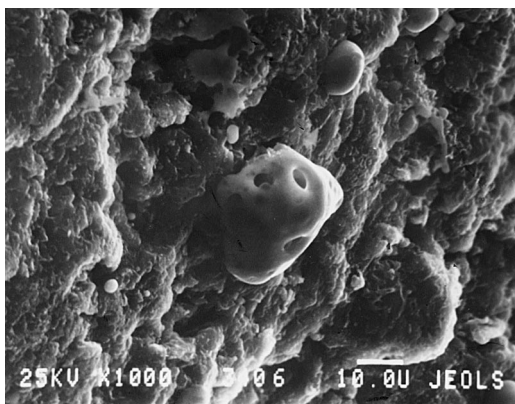


Fig. 4. Electron microphotographs of the RGT graphite (sample 1) after irradiation only by pulsed (0.5 MJ m^{-2} , $60 \mu\text{s}$, 10 pulses) plasma. The scale length corresponds to $10 \mu\text{m}$.

with high dose sputtering is thermally stable. The developed structure is considerably distinguished from that formed after irradiation only by the pulsed plasma flux (Fig. 4) [4]. Fig. 5 illustrates the surface structures of sample 2 irradiated at the temperature 1150°C and other condition identical to those for sample 1 (Fig. 2). The surface microstructure differs from that formed on the surface of the same graphite exposed in the LENTA deuterium plasma at the temperature of 770°C , all other conditions being equal. Such an effect was provided by different erosion processes at different temperatures of a target: physical sputtering takes place at 770°C , and the radiation-enhanced sublimation of graphite takes place at 1150°C . As a result of an effect by a high deuterium ion fluence on the graphite surface, the periodic structure with a honey-comb morphology of a terrace-type (from 0.5 to $2.5 \mu\text{m}$) was produced. They were previously observed on a number of solids after the ion bombardment [5].

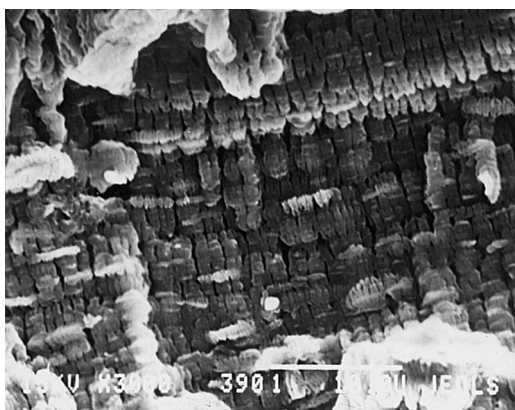


Fig. 5. Microphotographs of the RGT graphite (sample 2) after irradiation at the temperature 1150°C by 200 eV D^+ -ions up to a dose of 10^{22} cm^{-2} . The scale length corresponds to $10 \mu\text{m}$.

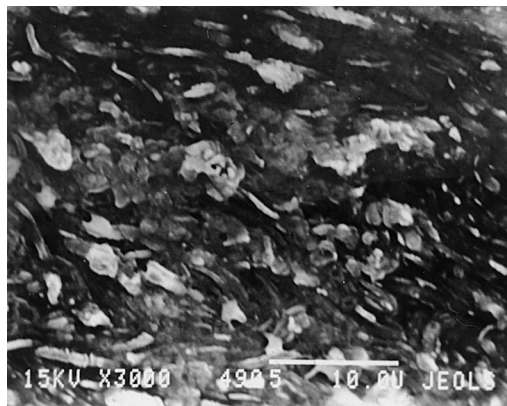


Fig. 6. Microstructure of the graphite (sample 2) after stationary and pulsed plasma irradiations. The scale length corresponds to $10 \mu\text{m}$.

Along with the formation of a recurring structure, somewhere on the surface one can see separate pieces of a “husk” which were probably produced as a result of the sublimed graphite back-diffusion and of its condensation upon the target.

After the joint effect of stationary and pulsed plasmas the surface topography is essentially changed (Fig. 6). The periodic relief produced by the stationary plasma is destroyed. One can see the traces of chipping and brittle destruction. The results of studying the erosion products with an electron microscope confirm this conclusion.

3.2. Graphite erosion products

Study of the samples with electron and optical microscopes has allowed us to determine the particle size. The particle distribution with respect to their sizes for two graphite samples exposed in the stationary plasma at 770°C (sample 1) and at 1150°C (sample 2) are represented as histograms in Figs. 7 and 8. Both distributions have two maxima located in the ranges 0.2 – $0.4 \mu\text{m}$ and 1 – $2 \mu\text{m}$, respectively. The erosion products from sample 1 after exposure to stationary and pulsed plasmas are mainly plates of irregular configuration measuring 1 – $3 \mu\text{m}$ (Fig. 9); one can also see condensed round drops, 1.0 – $3.5 \mu\text{m}$ in diameter, which coincides with the size of balls with the legs (see Fig. 10). Large particles, up to $40 \mu\text{m}$ in size, were detected in sample 2. One should note that the majority of the particles from sample 2 (Fig. 11) captured upon the basalt have a lentil-like configuration and dimensions characteristic of the honey-combs on the graphite surface after a large dose of irradiation in the stationary deuterium plasma (Fig. 5). Large particles have an arbitrary configuration.

The comparison between the graphite erosion product distribution histograms with respect to size, produced as a result of the joint stationary and pulsed

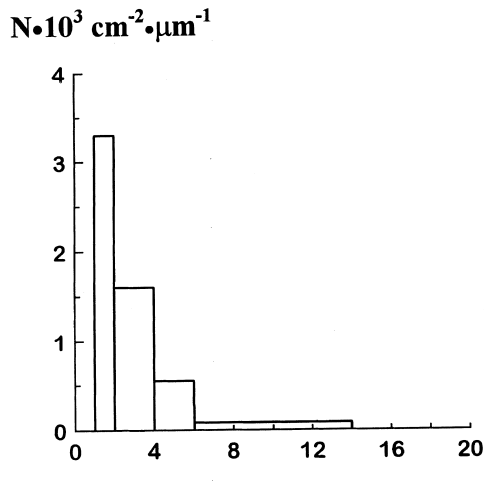
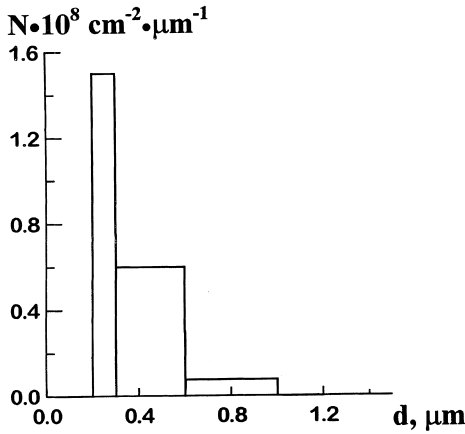


Fig. 7. Particle distributions 0.02–1.00 μm (a) and 1–40 μm (b) in size for deposited RGT graphite (sample 1) after stationary and pulsed plasma irradiations.

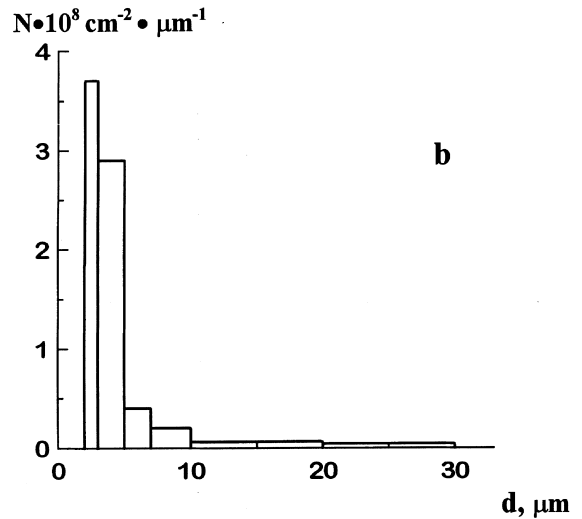
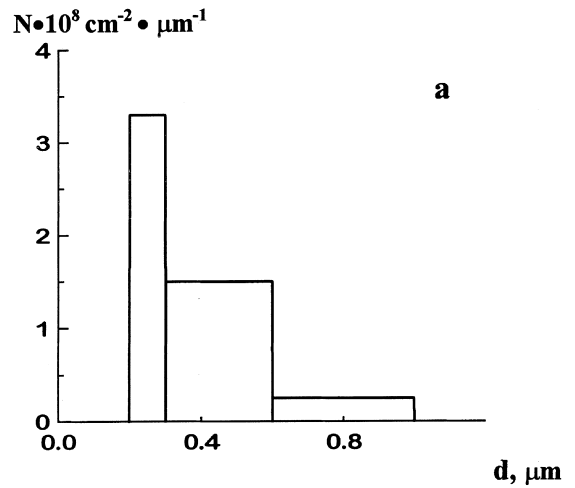


Fig. 8. Particle distributions 0.02–1.00 μm (a) and 1–40 μm (b) in size for deposited RGT graphite (sample 2) after stationary and pulsed plasmas irradiation.

irradiation, and similar data for the same graphite under only the pulsed high power plasma flux confirms the emergence of large particles, 15–40 μm in size in the distribution tail for sample 2 irradiated at 1150°C.

The results confirm the brittle destruction of the RGT graphite under combined long irradiation by a stationary deuterium plasma and by the pulsed high power plasma flux.

3.3. Plasma irradiation effect on the chemical composition of a surface layer

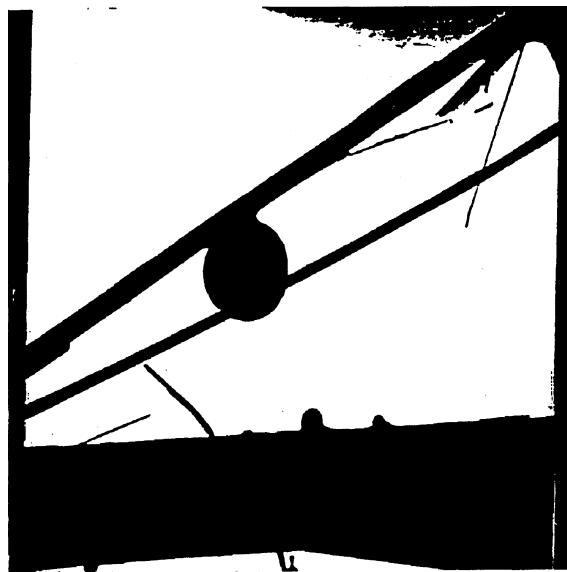
The titanium depth distributions for sample 2 after the exposure to stationary and pulsed plasmas from irradiated (a) and back (b) sides are compared in Fig. 12. As a result of deuterium ion bombardment, the graphite surface is enriched with titanium up to 6.5 at.%, and the titanium concentration at the distance of 1 μm from the

surface is still 5.5 at.%. On the non-irradiated side of the target, the titanium concentration on the surface is smaller than that in depth. That means that the radiation-enhanced segregation of titanium atoms takes place under deuterium bombardment.

One should note that the studies of composition and distribution profiles have a rather approximate nature since the target surface has a high degree of roughness.

3.4. Deuterium accumulation

D⁺-ion distribution profiles in the RGT graphite (sample 1) after bombardment by 200 eV D⁺-ions to the dose 10²² cm⁻² (curve 1) and after an additional irradiation in the pulsed plasma flux (curve 1') are given in



5 μm



5 μm

Fig. 9. Electron microphotographs of the filter with erosion products of the graphite (sample 1) after stationary ($D=10^{22}$ cm^{-2} , $T_{\text{irr}}=770^\circ\text{C}$) and pulsed (0.25 MJ m^{-2} , $60 \mu\text{s}$, 6 pulses) plasma irradiations.

Fig. 10. Electron microphotographs of the filter with deposited erosion products of the RGT graphite (sample 2) after stationary ($D=10^{22}$ cm^{-2} , $T_{\text{irr}}=1150^\circ\text{C}$) and pulsed (0.25 MJ m^{-2} , $60 \mu\text{s}$, 6 pulses) plasma irradiations (point 1 on Fig. 1).

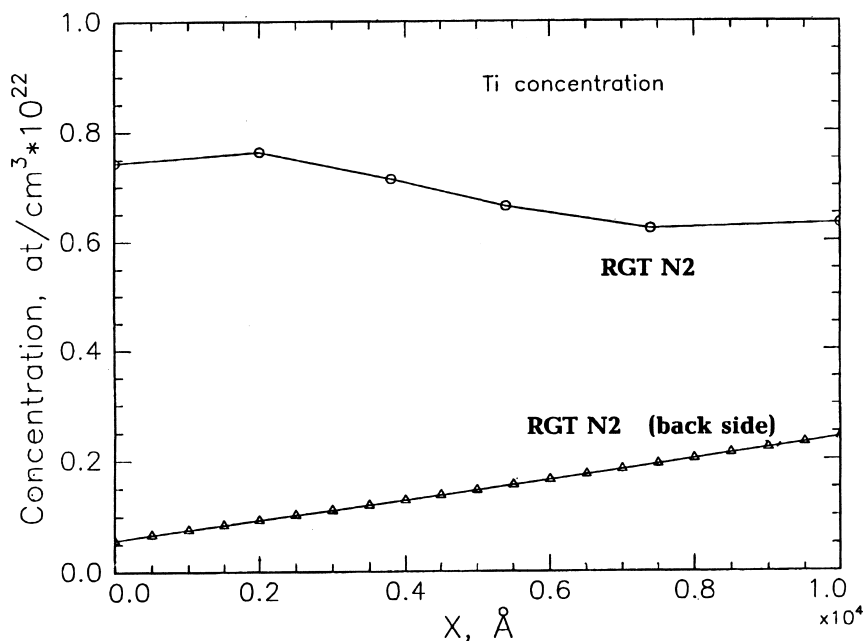


Fig. 11. The titanium depth distribution for sample 2 after stationary and pulsed plasmas irradiation (a) and back side (b).

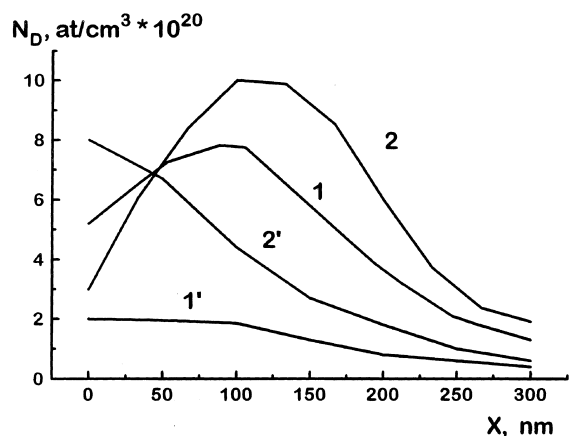


Fig. 12. Depth distribution of D in RGT graphite. (1) – after stationary plasma irradiation at 770°C, (1') – after stationary plasma irradiation at 770°C + pulsed plasma flux, (2) – after stationary plasma irradiation at 1150°C, (2') – after stationary plasma irradiation at 1150°C + pulsed plasma flux.

Fig. 12. An analysis of the given distribution confirms the diffusion of deuterium deeply into the target at 770°C. The diffusion yield calculated from curve 1 is $D = 5 \times 10^{15} \text{ cm}^{-2} \text{ s}^{-1}$. The integral amount of accumulated deuterium in the layer under analysis is $1.9 \times 10^{16} \text{ atoms cm}^{-2}$. As a result of the subsequent bombardment of the pulsed plasma simulating a plasma disruption, the integral deuterium concentration in graphite is reduced by about 5.5 times. In this case, the deuterium distribution is gradually reduced in depth from $2 \times 10^{20} \text{ cm}^{-3}$ on the surface to $4 \times 10^{19} \text{ cm}^{-3}$ at a point 3000 Å deep.

An essentially greater deuterium concentration after the combined treatment in LENTA and MTK-facilities (Fig. 12, curves 2 and 2'), remained in target 2 treated in the plasma at 1150°C. It can be associated with greater deuterium transgranular diffusion at 1150°C so that a greater amount of deuterium is retained after the surface layer destruction under the pulsed plasma effect than in sample 1. The integral deuterium concentration in it is 10^{16} cm^{-2} .

3.5. Discussion

The increase of graphite erosion due to plasma disruption after preliminary irradiation in stationary deuterium plasma is caused by a number of effects. The main ones are deuterium accumulation in graphite and intergranular bond destruction.

Deuterium implanted into graphite occupies cavities existing before irradiation and bubbles arising during the irradiation. During a high power plasma pulse, deuterium is heated, and its pressure is a failure factor [6]. At the temperature $T < 1000 \text{ K}$ only surface po-

rosity diffusion is possible [7], which transports deuterium to great depth (deeper than ion range) through open channels. Therefore, one cannot expect high pressure of deuterium in the deep cavities during stationary plasma irradiation at target temperature below 1000 K. However, at $T > 1000 \text{ K}$, transgranular diffusion can transport deuterium to deep closed cavities or other traps where bubbles are formed [7]. Therefore, one can expect a high pressure in such cavities during high power plasma pulses.

At $T > 1300 \text{ K}$, the radiation enhanced sublimation of graphite is observed, which is a result of interstitial atoms diffusing towards the surface [8]. The activation energy of this process is $\sim 0.78 \text{ eV}$. The interstitial atoms can diffuse not only to the surface but also to the inner boundaries of the grains. It is of importance that each interstitial atom transports potential energy equal to its energy of formation. At a grain boundary this energy can be released and excite the atoms of the boundary [9]. It results in radiation stimulated reconstruction of the boundary and decrease of intergranular bonds. Thus the grain boundaries at the depths much greater than the ion range can lose their strength. The surface structure after stationary plasma irradiation at $T = 1150^\circ\text{C}$ shows scale-like grains which are isolated from each other. This isolation is a result of surface diffusion of interstitial atoms along the intergranular spaces. Such isolated grains can be easily removed under a rather low thermal shock. In this case, the erosion depth during a disruption will be determined by the depth of such a loose layer and will be independent of energy absorbed by the surface.

4. Conclusion

- Graphite erosion during a disruption is determined by the temperature of preceding stationary plasma irradiation. At the temperature where the physical sputtering takes place, a brush-like structure is developed on the surface. This structure is stable under thermal shock. At temperatures $T > 1000^\circ\text{C}$ when the radiation enhanced sublimation takes place, the periodic terrace-type structure with lentil-like grains appears after stationary plasma exposure. Most of the lentil-like grains are weakly bound one with another and can be easily removed during a disruption.
- The graphite erosion product size distributions have two maxima in the ranges 0.2–0.4 μm and 1–2 μm independent of irradiation temperature. However, the density of particles and their maximal dimensions are larger in the case of stationary plasma exposure at the temperature corresponding to radiation enhanced-sublimation: the greatest particle size was up to 40 μm in the case of 1150°C stationary plasma irradiation, whereas in the case of 770°C it was 14 μm .

3. As a result of plasma exposure the RGT graphite surface is enriched with titanium.
4. The integral deuterium retention decreases after disruption from $1.9 \times 10^{16} \text{ cm}^2$ to $3.4 \times 10^{15} \text{ cm}^2$ in the case of 770°C stationary plasma irradiation, whereas in the case of 1150°C stationary plasma irradiation the final retention is 10^{16} cm^2 .
5. The effect of stationary plasma action is explained by deuterium accumulation in graphite and intergranular bond destruction due to radiation-stimulated defect migration.
6. In the case of radiation enhanced sublimation the erosion depth during a disruption is determined by the depth of the loose layer formed by prior stationary plasma irradiation. This is expected to be valid for any kind of graphite.

Acknowledgements

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