Dose dependence of the erosion of graphite under high temperature ion irradiation

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Abstract. In this paper, a dependence of the erosion of graphite under a high energy ion flux at temperature of 2050 °C on the ion dose is investigated. It is shown that high doses of irradiation stimulate diffusion processes that lead to the removal of carbon atoms from the bulk of the sample, significantly altering morphology of graphite at depths exceeding penetration depth of irradiating ions for large doses.

1. Introduction
Graphite is used as a plasma-facing element in a number of thermonuclear and plasma technology facilities [1–4]. However, the behavior of graphite under high energy ion beam at high temperatures has not been sufficiently studied. In this work, a research on the behavior of graphite under intensive deuterium ion flow irradiation at high temperatures is conducted.

2. Experimental setup
Investigation was conducted on a “COating Deposition and MATerial Testing” Stand (CODMATT) (figure 1) [5]. Experimental parameters were as follows: residual gas pressure <2·10⁻⁶ torr; ion flux 2–2.3·10¹⁷ ion/s. Average penetration depth of deuterium ions under these parameters is 120 nm.

Figure 1. Stand for Coating Deposition and MATerial Testing (CODMATT).
Small-grain graphite was used as a material under investigation. 14×16×2 mm³ samples were polished using a sequence of sandpapers with grain sizes of 40 to 3 µm, then cleaned in an ultrasonic bath with ethanol and annealed in plasma. A Central Sector (CS) on the surface of the target was analysed, where the average power density was 36 MW/m², and the average ion flux density was 1.7·10²² ion/s×m², with the variation of ±16 % on the area of 1.3 mm in radius. The samples were irradiated by doses from 5.25·10²⁰ to 6.3·10²¹ ion/cm² at a temperature of 2050 °C.

3. Results and discussion
Photographs of surfaces of graphite samples after varying doses of irradiation are shown in figure 2. As can be seen, with the increase of dose, surface porosity increased, as well. Some areas that significantly differed in size, concentration and depths of pores were distinguishable. Electron microscope analysis has shown that pore formation was hindered on the surface of graphite grains with graphene layers oriented at small angles relative to irradiated surface.

CS mass loss and, consequentially, sputtering coefficient, were measured by weighing the samples before and after irradiation, and also by measuring the depth of graphite layer removed from CS relative to unsputtered surface after each dose of irradiation.

![Figure 2. CS surface morphology: (a) – before irradiation; (b) – irradiated by 1.05·10²¹ ion/cm²; (c) – irradiated by 4.73·10²¹ ion/cm².](image)

The analysis of the measurement results has shown that the quantity of removed material and, as a consequence, sputtering coefficient, decreases steadily with the increases doses of ions. The ratio of loss of graphite material in CS measured through weighing to the loss measured through profilometry started at approximately 3 for a dose of 5.25·10²⁰ ion/cm² and ended at 3.5 at 6.3·10²¹ ion/cm². For example, at a dose of 6.3·10²¹ ion/cm², the mass loss obtained via weighing and the mass loss calculated through depth were 4.72 mg and 1.33 mg, respectively. This fact leads to the assumption that in the entire range of irradiation doses used in this research, irregularities in the bulk of the material are developed that lead to the formation of a porous layer.

Such development of porous layers could be possibly attributed to two radiation-stimulated effects acting simultaneously: development of porosity into the bulk of the sample and carbon atom transport to the surface. Both these processes could be carried out due to the evolution of material structure disordering approaching the surface of the sample. This leads to stimulation of the development of the porous layer into the bulk of the material and to diffusion of carbon atoms to the surface.

Sputtering depths obtained via weighing and measurement of the CS relative to the non-sputtered areas of the sample (figure 3) increase with the irradiation dose. At the same time, the “mass” depth grows faster with the increased irradiation dose then the “profile” depth. It is also worth noting that, as can be seen in figure 2, the porosity of the surface layer also increases at higher irradiation doses.

If, starting with a certain stage of sputtering, thickness of the porous layer and its porosity were constant, sputtering depth and mass of sputtered material measured by different methods would have
been the same and the related sputtering coefficients have been similar. However, as mentioned above, the depth and mass of sputtered material measured by weighing grow faster than the respective values measured by profilometry. This means that: 1) sputtering of material from the surface of the porous layer is partially compensated by a diffusion flux of carbon atoms to the surface and 2) thickness of the porous layer increases with irradiation doses.

After the initial irradiation period \((5.25 \times 10^{20} \text{ ion/cm}^2)\), the ratio of “mass” depth to profile depth is kept constant, meaning that the rate of growth of porous layer was constant for consecutive doses, and the density of all newly formed layers and related sputtering coefficients were the same (figure 4).

This conclusion allows determining the rate of growth for porous layers depending on the irradiation dose, and the density of graphite in such layers.

If the increase of thickness of a porous layer at the certain stage of irradiation is \(\Delta L_p\), \(S\) is the surface area of CS and the mass of the removed material measured by weighing and profilometry is, \(M_m\) and \(M_p\), respectively, density of the part of porous layer added at this stage of irradiation is determined as:
Density of a porous layer addition after irradiating the sample with a dose between 5.25 \cdot 10^{20} and 1.05 \cdot 10^{21} ion/cm^2 calculated using expression (1) appears to be 1.49 g/cm^3, and after irradiation with a dose between 4.73 \cdot 10^{21} ion/cm^2 and 6.3 \cdot 10^{21} ion/cm^2 – 1.30 g/cm^3. These results show that density of a porous layer is much smaller than that of the non-irradiated graphite, which is near 1.8 g/cm^3, and decreases with the irradiation dose.

Assuming that the sputtering rate of bulk porous layer areas irradiated by the ion flow is significantly lower than the sputtering rate of the surface porous layer areas, sputtering coefficient of the latter on each stage of sputtering can be determined as:

$$ Y_{pore} = Y_s \times \left( \frac{C_{graf}}{C_{pore}} \right)^\frac{1}{3} = \frac{N_c}{N_i} \times \left( \frac{C_{graf}}{C_{pore}} \right)^\frac{1}{3}. $$

Here, $Y_{pore}$ and $Y_s$ are, respectively, sputtering coefficients of the pores surface and of the non-irradiated surface, $C_{graf}$ and $C_{pore}$ are, respectively, density of graphite and density of a porous layer, and $N_c$ and $N_i$ are, respectively, the amount of irradiating ions and atoms sputtered from the irradiated surface on the current stage of irradiation.

The calculations show that the sputtering coefficient of CS measured both by mass and by depth decrease at higher irradiation doses, but there is a tendency of flattening out at approx. 0.16 at./ion and 0.05 at./ion for coefficients measured through weighing and profilometry, respectively, after an irradiation dose of 3.15 \cdot 10^{11} ion/cm^2.

4. Conclusion
Erosion and modification of small-grain graphite’s surface layer under irradiation at temperature of 2050 °C by deuterium ions with the energy of 7.5 keV and ion flow power density of 30 MW/m^2 for doses of up to 6.3 \cdot 10^{21} ion/cm^2 has been investigated.

It was found that a porous layer is formed on the irradiated surface with a thickness of 2000 µm after being irradiated by a dose of 6.3 \cdot 10^{21} ion/cm^2, while the level of irradiated surface retreats to the depth of 165 µm relative to the non-irradiated surface, and the average penetration depth of ions into graphite is approx. 120 nm.

Sputtering coefficient that starts at 0.38 on the first stage of irradiation, decreases at higher doses and virtually stabilizes at 0.17 for doses of 3.15 \cdot 10^{21} ion/cm^2 and higher.

A proposition is made that graphite irradiation at the temperature of about 2000 °C stimulates diffusion of carbon atoms from the bulk of graphite, leading to the growth of the porous layer at higher doses and, consequentially, deceleration of growth of the “crater”.

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