Temperature dependence of erosion of graphite under high-temperature ion irradiation

L B Begrambekov, A V Grinin, G D Dolganov, N A Puntakov and K M Titkova
National Research Nuclear University «MEPhI», 115409, Kashirskoe sh. 31, Moscow, Russia
np9293.2009@my.bristol.ac.uk

Abstract. In this paper, a dependence of the erosion of graphite under a high energy ion flux from the ion dose is investigated at temperatures of 750, 1700 and 2050 °C. High doses of irradiation stimulate diffusion processes that lead to the transport of carbon atoms from the bulk of the sample towards the surface, leading to the formation of a porous layer which thickness increases both with the increase of sample’s temperature, as well as the dose of irradiation.

1. Introduction
Graphite is being used for plasma-facing elements in a number of fusion and plasma technology facilities. The erosion of graphite irradiated by ion flows with moderate ion flux at high temperatures has been relatively well-researched [1-4]. However, the behavior of graphite under irradiation by high intensity ion flux at high temperatures, that merits significant practical interest, has been researched poorly. The first results of our investigation this problem are presented in work [5]. This paper presents new experimental results and discussion of these results.

2. Experimental
Irradiation of MPG-8 grade small-grain graphite was conducted on the “Stand for Coating Deposition and Material Testing” (CODMATT) (Figure 1), designed to investigate interaction of ions and electrons with energies of up to 25 keV and power density of up to 240 MW/m² with the surface of materials. The schematic of the modification of the stand for irradiating the samples is shown on Figure 1. Graphite samples were sequentially polished with sandpaper ranging in grain size from 40 to 3 µm, then washed in an ultrasonic bath with ethanol and annealed in plasma. Ions of plasma initiated in the plasma chamber between the cathode and the anode are accelerated and focused on the sample negatively biased relative to the plasma chamber anode. Main elements of the sputtering mode of the stand are shown on Figure 1.

The experimental parameters were the following: residual gas: <2×10⁻⁶ torr; energy of deuterium ions irradiating the sample – 7.8 keV/at.; ion flux density – 2.9×10²² ion/s×m² ± 0.5×10²² ion/s×m². Dose dependence on the interaction of ions with the surface of graphite was investigated in the range of doses from 1.7×10²⁵ to 2.1×10²⁶ at./m², with temperatures ranging from 750 to 2050 °C.

Temperature of the samples during the experiments was determined by power of ion flux and thermal radiation of the sample. To reduce the surface of the sample and, as a result, increase its temperature in the range of 750-2350 °C, thickness of the sample was altered between 5 and 0.5 mm.
Figure 1. Schematic of the stand:
1 – vacuum chamber;
2 – plasma chamber;
3 – glow cathode;
4 – anode;
5 – inlet;
6, 7 – gate chamber;
8 – valve;
9 – gasket;
10, 14 – electric inlet;
11 – sample;
12 – cooling system;
13 – sample inlet;
15 – antidynatron electrode;
16 – ceramic insulator.

The amount of mass sputtered from the sample at each stage of irradiation was determined by the difference between the levels of irradiated surface before and after the experiment, as well as weighting the sample (respectively, \( L_{\text{surf}} \), \( L_{\text{mass}} \), \( M_{\text{surf}} \) and \( M_{\text{mass}} \)). The dependence of thickness of sputtered layer on irradiation dose at different temperatures (respectively, \( \Sigma L_{\text{surf}} \) and \( \Sigma L_{\text{mass}} \)) measured by these two ways, is shown on Figure 2a and 2b.

![Figure 2a](image1.png)  ![Figure 2b](image2.png)

Figure 2. Dose dependence of the thickness of sputtered layers (\( \Sigma L_{\text{surf}} \) and \( \Sigma L_{\text{mass}} \)): (a) T = 2050 °C; (b) T = 2050, 1700 and 750 °C

3. Results and discussion
The existence of a discrepancy between the weighting measurement and the level measurement (Figure 2) allows concluding that irradiation by ions with a flux density of \( 2.9 \times 10^{22} \) ion/s×m² stimulates development of a porous layer in the entire range of ion energies and doses used in the experiment. Figures 3 and 4, showing the porous surface of the samples irradiated by different doses, as well as the cross-section and the reverse side of the sample irradiated at higher temperatures.
Figure 3. Surface of samples irradiated at $T = 2050\, ^\circ\mathrm{C}$: (a) after irradiation by dose of $2.6\times10^{25}$ at./m$^2$, scale x500; (b) after irradiation by dose $1.6\times10^{26}$ at./m$^2$, scale x1000.

The thickness of a porous layer could not be determined in this cycle of experiments. However, it’s obvious that the increase in thickness of a porous layer should be higher than $\Delta L = L_{\text{mass}} - L_{\text{surf}}$ during each stage of sputtering. Judging by the data from Figure 2, it can be stated that the increase of the porous layer in thickness accelerated both with the increase of temperature of graphite and irradiation dose. At the same time, a tendency for the decrease of the erosion rate of the surface of graphite with the dose and, consequentially, with the increase of the thickness of a porous layer can be observed.

Figure 4. (a) Cross-section of a sample irradiated by a dose of $3.4\times10^{25}$ at./m$^2$, $T = 2350\, ^\circ\mathrm{C}$, Scale x1500; (b) Reverse side of a sample irradiated by a dose $5.2\times10^{25}$ at./m$^2$, $T = 2350\, ^\circ\mathrm{C}$, scale x1000.

Some idea on the size of the porous layer can be deduced from Figure 4b. It can be seen that the reverse side of a 0.5 mm thick sample irradiated by a dose of $5.2\times10^{25}$ at./m$^2$ at $T = 2350\, ^\circ\mathrm{C}$ was already in the range of a porous layer. In this particular case, the value of $M_{\text{mass}}$ was 475 µm.

The reason behind the appearance of pores could be the difference in the rate of sputtering between different areas of polycrystalline graphite: differently oriented crystal grains, grain boundaries, poorly crystallized areas etc.
The increase in thickness of a porous layer with the irradiation dose could possibly be supported by a flow of carbon atoms from below the irradiation spot. Atoms going to the surface partially compensate the sputtering effect of ion irradiation. This flow could be formed due to structurisation of graphite occurring at high temperatures along with destructurisation caused by ion bombardment, which lead to the development of porosity and the diffusion of vacations into the bulk of the sample.

Comparison of surface morphologies after irradiation by a dose of $2.6 \times 10^{25}$ at./m$^2$ and $1.6 \times 10^{26}$ at./m$^2$ (Figure 3 (a) and (b), respectively) shows that the surface porosity is virtually unchanged after a certain dose of irradiation despite the increase of thickness of the porous layer with the duration of sputtering. As such, one can propose that the transport of vacancies into the bulk of graphite generated a flow of atoms towards the surface not only due to the increase of a porous layer in thickness, but also due to the increase of porosity in each newly created layer for continuous irradiation.

Development of pores and formation of an atom flow towards the surface probably occurred along the grains with basis planes having large angles with the surface plane. As seen on Figure 3(a), grains with basis planes at small angles with the surface plane were eroded at a lower rate for the first stages of irradiation. Pores on the surface were not significantly developed due to large distances between basis planes. No grains oriented in such way could be observed on the surface of the samples after large doses of irradiation. Their disappearance could be caused by diffusion of vacancies along the basis planes leading to the drift of atoms from these grains onto neighboring pores or grains with larger angles towards the surface plane, and further transport of these atoms towards the surface.

4. Conclusion
Some aspects of erosion and modification of graphite at high temperature irradiation by euterium ions with energy of 7.8 keV/at. and ion flux density $2.9 \times 10^{22}$ ion/s×m$^2$ at temperatures of 750, 1700 and 2050 °C.

Ion irradiation stimulates development of porosity on the surface layer of the sample at all temperatures and irradiation doses used in the experiment. The thickness of a porous layer after a certain irradiation dose is large than the thickness of a layer sputtered according to the mass loss of a sample, as well as the measurement of the sputtered layer. Thickness of the porous layer increases with the increase of irradiation temperature as well as the dose of irradiation. At the same time, the rate of erosion of graphite decreases with temperature and, as a consequence, the increase of the porous layer. The flow of carbon atoms from the porous layer towards the surface partially compensates the sputtering effect of the ion flux. This atom flow formed due to structurisation of graphite along with the crystal disorientation due to ion bombardment, which lead to development of porosity and diffusion of vacations into the bulk of the samples.

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