Properties of graphite to be used as a material for the T-15MD tokamak plasma-facing elements

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Abstract. The results of the measurement of physical properties, sputtering yield during irradiation by hydrogen ions and a study of surface morphology modification of graphite to be used for the plasma-facing materials of the T-15MD tokamak are presented in this work.

1. Introduction

A number of fusion facilities implement graphite for plasma-facing components. Among them there are J-TEXT [1] and KTM [2] tokamaks, as well as a Wendelstein 7-X stellarator [3]. Graphite is also going to be a material for divertor and first wall tiles in the T-15MD tokamak [4]. Since graphites of different grades can possess different properties, a research on the characteristics of the R6510P100D01 graphite chosen for usage in the T-15MD tokamak was required.

2. Results

2.1. Density of the R6510P100D01 grade graphite

To measure the density of the R6510P100D01 grade graphite, four samples were used. Density values of the samples were found as 1.77±0.05; 1.81±0.05; 1.82±0.05; 1.84±0.02 g/cm³. It is worth noting that the density of this particular graphite is in the range of densities of graphites of the MPG series, measured by various authors (MPG-6 grade graphite – 1.65-1.9 g/cm³ [5], MPG-7 grade graphite – 1.7-1.9 g/cm³ [5], MPG-8 grade graphite – 1.75-1.85 g/cm³ [6]).

2.2. Impurity content and structure of graphite

Energy dispersion spectroscopy (EDS) analysis showed an absence of any impurities within the 0.05 wt.% margin of error. A structure of R6510P100D01 was analysed and compared to the structure of MPG-8. The average size of crystal grains for R6510P100D01 was approximately 60 µm (up to 100 µm for MPG-8). Analysis of the cross-section of both graphites showed the existence of three-dimensional pores. The average size of pores was approximately 10 µm for both, and the maximum size was 20 µm for R6510P100D01 and 30 µm for MPG-8. Pores of varying sizes were evenly distributed on the cross-section of both graphites, with 10-15 pores for R6510P100D01 (15-20 pores for MPG-8) on average in a 50×50 µm² area.
2.3. Thermal diffusivity and thermal conductivity of the R6510P100D01 grade graphite

Thermal diffusivity was measured using the laser flash method on the Netzsch Laser Flash Apparatus LFA-427. The measurements were made at room temperature on samples cut in mutually perpendicular planes. The measurements showed the same thermal diffusivity of all samples (90 ± 3 mm²/s, the difference between the samples is below 0.9%), which indicates the isotropy of graphite.

To determine the temperature dependence of thermal conductivity, we measured the thermal diffusivity of the sample (with a density of 1.77 ± 0.05 g/cm³) in the temperature range of 300-1675 K (Fig. 1). The calculation based on the measurement results showed that the thermal diffusivity of R6510P100D01 is described by the approximation formula

$$\alpha = 331 \times e^{\frac{T}{162.4}} + 42.4 \times e^{\frac{T}{766}} + 9.17 \text{ mm}^2/\text{s}, \quad T \text{ is K, with an accuracy of 0.5\% and is close to the thermal diffusivity of MPG-6 with a density of 1.776 and 1.783 g/cm}^3 [7].$$

To calculate the temperature dependence of thermal conductivity, temperature dependences of both specific heat capacity and density are required. It is known that specific heat capacity of isotropic graphites has a weak dependence on its grade [8]. This dependence could be calculated using an approximation formula

$$C_p(t) = 0.636 + 3.09 \times 10^{-3} t - 3.724 \times 10^{-6} t^2 + 2.609 \times 10^{-9} t^3 + 6.92 \times 10^{-13} t^4,$$

where $t$ is °C, $C_p$ is J/(g K) with 0.7% margin of error [7]. Dependence of density on temperature is deduced using linear expansion coefficient. The difference in the linear expansion coefficients for different grades of graphite is insignificant [9] and taking into account the effect of this difference on a change in the density of graphite with temperature contributes less to the error in determining the density than the error in measuring density at room temperature. As such, a dependence of MPG-6 on room temperature value equal to $6.67 \times 10^{-6}$ K⁻¹ was used for calculating the dependence of density on temperature. Based on this data, a temperature dependence of thermal conductivity was calculated (Fig. 1). The data was approximated ($T - K$, $\lambda - W/m\cdot K$, deviation 2%): $\lambda(T) = 1.0525 \times T^{-0.00346 + 5.067 \times 10^{-5} T^2 - 3.835 \times 10^{-8} T^3 + 1.4636 \times 10^{-12} T^4 - 2.23 \times 10^{-16} T^6}.$

A temperature gradient for a thermal conductivity for MPG-6 of varying densities could be calculated by a formula

$$\lambda(t) = \lambda_{20} \times (0.351 + 0.675 \times e^{0.397 t}),$$

where $t$ is °C, $\lambda_{20}$ - a thermal conductivity at 20°C [7]. It is seen (Fig. 1) that in a range of 400-800°C a complete match is obtained of the dependence of R6510P100D01 and the dependence calculated using the formula. A difference at 100-200°C is explained by the fact that this formula was deduced in the experiments that didn’t include any measurements in the range of 20-200°C. The difference at the temperatures higher than 800°C can be explained by the error of either the measurements or the approximation.

2.4. Sputtering yield of the R6510P100D01 grade graphite

The experiments show that sputtering yields of R6510P100D01 irradiated by ions of hydrogen plasma with energies 180 eV/at. and 350 eV/at. at a flux density of $2.6 \times 10^{20}$ ion/m²s at a temperature of 330 K...
are close to the sputtering yields of MPG-8 irradiated under same conditions and are 0.050±0.006 for the energy of 180 eV/atom and 0.097±0.011 for the energy of 350 eV/atom. With the increase in the ion energy, the sputtering yield lowered. An increase in flux density to 3.8×10^{20} ions/m^2 s at the ion energy of 350 eV/atom, and the temperature increase to 750 K at the ion energy of 180 eV/atom, lead to the increase of sputtering yields to 0.17±0.01 and 0.15±0.01, respectively. Increase in sputtering at higher temperature is explained by chemical sputtering, and the increase in sputtering at a higher flux density - by the increase in temperature to 370 K and by the higher density of sputtering cascades.

2.5. Pore formation on the R6510P100D01 grade graphite under irradiation by moderate ion flux. When R6510P100D01 was irradiated by hydrogen ions with energy of 350 eV/atom and a dose of 9.4×10^{23} ions/m^2, the number of pores on the surface increased by less than 10% compared to an unirradiated sample, with the increase being due to the appearance of small (less than 10 μm in diameter) pores. Intense pore formation was observed, when the ion energy approached approx. 1200 eV/atom. At an ion energy of 2500 eV/atom, the number of medium-sized (10-30 μm) pores increased by approx. 50%, and pores larger than 30 μm appeared. In general, the nature of the development of pores on the surface of both R6510P100D01 and MPG-8 grade graphites under ion irradiation is similar. However, the pores larger than 10 μm appeared on an MPG-8 grade graphite when it was irradiated by ion with energy as low as 350 eV/atom.

2.6. Modification of the R6510P100D01 grade graphite under high temperature irradiation by intense ion flux. The experiments were conducted on a “COating Deposition and MATerial Testing” (CODMATT) stand [10]. Flux density of H_2^+ ions with energy of 6 keV/atom was (5.6±0.9)×10^{21} ions/m^2 s. The sample was subjected to 10 irradiation pulses 60 seconds each. Temperature of the sample was increased to 2400 K during 30 sec at an initial period of irradiation and was stayed unchanged during the remainder of the pulse. After each pulse, the sample was cooled for 60 seconds, and the temperature during cooling was reduced to 1500 K. The total irradiation dose was ≈ 3.8×10^{24} ions/m^2. The thickness of the samples was 0.5; 1; 1.5 and 2 mm. The irradiated surface, as well as the reverse surface of the samples, were studied using a Tescan Vega 3 scanning electron microscope, giving the data on the modification of the surface and the near-surface layers of graphite during high intensity pulse loads.

After irradiation, significant surface destruction was observed. With the decrease of the sample’s thickness, the surface destruction was more apparent. For example, before irradiation, there are about 5 pores on a 50×50 μm^2 area for a R6510P100D01 graphite, with the total area covered by pores being approximately 3-5%; after irradiation, the area covered by the pores on a 2 mm thick sample is increased to about 20%, with 20 pores on a 50×50 μm^2 area. In both cases, most of the pores are less than 10 μm in size (Fig. 3a). For a 0.5 mm thick sample (Fig. 3b), there are almost no pores less than 10 μm in size. The total area covered by pores exists 50%, and some pores reach 50 μm or even more. Irradiation did not lead to modification of the reverse side of the 2 mm thick sample. There is some recrystallization on a 1.5 mm thick sample (Fig. 3c). Graphene planes of most surface crystallites (2-5 μm in size) are oriented parallel to the surface. The relative porosity of the surface is the same as that of an unirradiated sample. On the reverse side of the 1 mm thick sample, the size of the surface crystallites reaches 10-15 μm. Some of them are oriented perpendicular to the surface. Most pores are smaller than 5 μm in size, but some of them reach 20 μm. The relative porosity of the surface approaches 7%. The reverse side of a 0.5 mm thick sample is significantly destroyed (Fig. 3d). The size of the crystallites is 10-20 μm, with a large amount of flakes that are 1-5 μm long. Most crystals are perpendicular to the surface. A large number of pores greater than 20 μm are located between the crystals. In general, relative porosity of the reverse surface approaches that of the irradiated surface.

The experiment results show that, for an ion penetration depth of 120 nm, relative porosity of the 1 mm thick near-surface layer increased by 20-30% compared to the non-irradiated graphite. It’s also worth noting that, for 2 mm thick graphite samples, the development of pores in near-surface...
layers wasn’t as intense as that for 1- and 0.5 mm thick samples. However, the thickness of the porous layer is apparently larger. This is explained by the fact that the vacancy diffusion to the surface in a thicker graphite sample will initiate transport of carbon atoms to the surface from deeper layers than for thinner samples. The rate of erosion during ion irradiation increases sharply [11] due to the development of radiation-accelerated sublimation of graphite under these conditions [12]. As such, plasma-facing elements composed of graphite need protection from plasma-surface interactions resulting in their rapid deterioration. The role of a protective in situ renewable coating, [13], can be played by a coating of crystalline boron carbide.

![Figure 3. Samples of the R6510P100D01 grade graphite, irradiated by H\textsuperscript{+} ions with energy of 6 keV/at.; flux density - (5.6±0.9)×10\textsuperscript{21} at./m\textsuperscript{2}s; dose – 3.8×10\textsuperscript{24} ion/m\textsuperscript{2}: a) irradiated surface, 2 mm; b) irradiated surface, 0.5 mm. c) reverse side, 1.5 mm; d) reverse side, 0.5 mm.](image)

3. Conclusion.

Density and porosity of the R6510P100D01 grade graphite, chosen for usage in a T-15MD tokamak, were measured, and an impurity content was evaluated. Temperature dependence of thermal conductivity, as well as a dependence of sputtering yield on temperature and ion energy, were measures. The measurements show that the properties of R6510P100D01 are not significantly different from the properties of the MPG-8 grade graphite.

The features of graphite modification upon irradiation with moderate and high intensity ion fluxes are investigated. A modification of the graphite structure and the development of bulk porosity to significant depths under irradiation with long pulses of intense ion irradiation are noted. In order to avoid relatively fast erosion, it may be necessary to protect graphite from radiation when operating in such modes with in situ renewable coating, [13] coating of crystalline boron carbide.

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