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Mass Spectrometric control of the process of cleaning and degassing of protective graphite tiles of the Globus-M2 tokamak

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Using a specially designed time-of-flight mass reflectron with a heating element inside the device, which makes it possible to heat solid samples and analyze the gases released from them, protective graphite tiles covering almost the entire plasma-facing surface of the Globus-M2 tokamak vacuum chamber were studied. Mass spectrometric analysis made it possible to determine the composition of the released gases to estimate their quantities, and thus to quantitatively characterize the quality of cleaning and degassing of tiles. It is shown that as a result of the applied method for processing tiles, the content of the light hydrogen isotope - protium, water, carbon monoxide and dioxide decreased several times, and the content of deuterium and helium isotopes decreased almost to zero.

Keywords: protective graphite layer, time-of-flight mass reflectron, mass spectrum, tokamak.

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The Globus-M2 spherical tokamak [1,2] is a compact next-generation unit (major radius, 0.36 m; minor radius, 0.24 m; toroidal magnetic field $\leqslant 0.9$ T) that is designed to study physical processes in plasma (with a divertor configuration) and optimize engineering solutions for spherical tokamaks of the megampere range. Approximately 98% of the inner plasma-facing surface of the vacuum chamber of Globus-M2 are covered with protective graphite tiles with a thickness of 5–10 mm and an area of 100–200 cm^2 (614 tiles in total; see Fig. 1). These tiles are fabricated from graphite RGTi doped with 2 wt.% of titanium, 0.3−0.7 wt.% of silicon, and 0.25 wt.% of boron [3,4].

The composition and amount of sorbed gases in protective graphite tiles exposed to a great number of deuterium plasma discharges ($\sim 20\,400$) were examined with a specially designed mass reflectron with a linear trajectory of ions [5]. The principal difference between this instrument and similar existing designs consists in the presence of a heating element with a thermocouple in the vacuum chamber of the mass analyzer. A quartz ampoule containing the studied sample may be positioned within this heating element. The inner diameter of the ampoule is ~ 2 mm, and its height is 10−12 mm. The open ampoule end is mounted in immediate proximity to the lower edge of the ionization zone of the ion source of the mass reflectron (so that gases released from the sample during heating would enter the ionization zone directly). Samples for analysis were collected by removing a thin upper layer from the surface of graphite tiles, which interacted with plasma, mechanically with a sharp scalpel. The obtained graphite powders were collected, marked and stored in separate containers. The mass of a graphite sample for analysis introduced into the quartz ampoule was ~ 1 mg (samples were weighed using an analytical balance with an error of 0.1 mg). A tungsten filament 0.3 mm in diameter wound around a quartz tube was used as a heater. This structure provided an opportunity to heat a sample from room temperature to 700°C in steps of, e.g., 100°C.

In accordance with the conventional procedure, a blank measurement of the background signal was performed (i.e., the spectrum of residual gases in the instrument was recorded at different temperatures with no sample present in the quartz ampoule) when a vacuum level of $\sim 1 \cdot 10^{-7}$ Torr in the mass analyzer chamber was reached. In subsequent processing of the obtained data, this spectrum is subtracted from the spectra of studied samples recorded at the corresponding temperatures. Figure 2 shows the „blank“ background spectrum of the empty quartz ampoule at $T = 500°$C. This is a typical mass spectrum of residual vacuum of a metallic vacuum chamber. It features atomic and molecular hydrogen H^+, H_2^+ (peaks at 1 and 2 a.m.u.); atomic carbon $^{12}C^+$; methane CH_4^+; hydroxyl group and water OH^+, H_2O^+ (16−17−18 a.m.u.); carbon monoxide CO^+; nitrogen N_2^+; ethylene-like organic radicals $C_2H_4^+$ (unresolved peak at 28 a.m.u.); and carbon dioxide CO_2^+ (peak at 44 a.m.u.).

At the next stage, the ampoule with a graphite sample was positioned within this heating element of the instrument and a series of measurements of mass spectra of gases released in the process of heating of the sample to different temperatures (in 100°C steps) was carried out. It should be noted that averaging over 500 points is performed in the process of measurement of spectra; i.e., the instrument outputs a single averaged spectrum instead of 500 recorded ones.

The obtained measurement data revealed that all spectra of samples, which were collected from different protective graphite tiles, recorded at different temperatures feature the

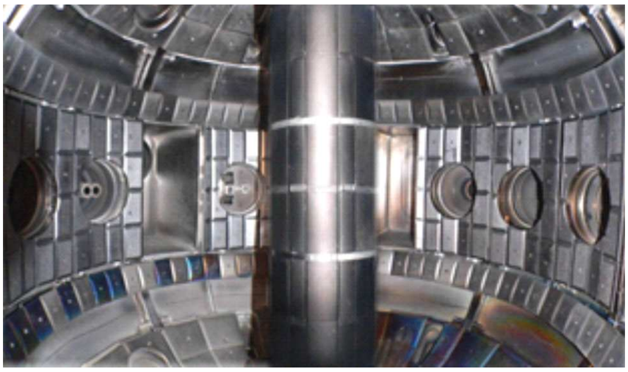

Figure 1. Interior view of the vacuum chamber of the Globus-M2 tokamak that is lined with graphite tiles. Non-covered surfaces are stainless steel.

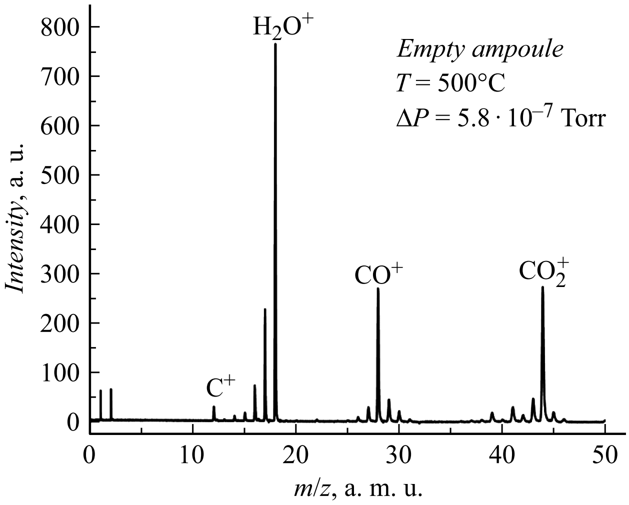

Figure 2. Mass spectrum of the empty ampoule at a temperature of $T = 500°$C.

same mass peaks. These peaks differ only in amplitude; the amplitudes of background lines are 2–3 times lower, and the background spectra contain two peaks corresponding to 3 and 4 a.m.u. In our view, these are the unresolved HHH^+–HD^+–$^3He^+$ triplet and $^4He^+$–$^2D^+$ doublet peaks, since a light hydrogen isotope (protium; H^+, H_2^+) is always present in any metallic vacuum chamber, deuterium (D^+, D_2^+) is the working gas for plasma experiments, light isotope $^3He^+$ is one of the products of thermonuclear synthesis involving deuterium, and heavy helium isotope $^4He^+$ is used to clean the vacuum chamber of the Globus-M2 tokamak with a glow discharge (therefore, trace amounts of it may remain inside the instrument).

Thermal desorption curves $I^+_{ion} = f(T[°\mathrm{C}])$ for almost all gases indicated above were plotted after the measurement of mass spectra of graphite samples at different temperatures. Having analyzed these curves, we found that ions corresponding to peaks with masses of 3 and 4 a.m.u. are not positioned on the solid surface of graphite samples; instead, they are „driven“ by plasma into the bulk of graphite. Notably, the intensity of these peaks drops to zero at a temperature on the order of 600–700°C. Water ions apparently have two fractions located on the sample surface and deep within the bulk of the material. Carbon monoxide and dioxide ions are likely to be located deep within the bulk of protective graphite tiles, since the release of these components is maximized in the high-temperature region of all desorption curves. A more detailed description of these findings can be found in [6].

A more thorough analysis of a large number (approximately 150) of mass spectra of graphite samples corresponding to different temperatures and thermal desorption curves, which were plotted based on these spectra, allowed us to conclude that curves $I^+_{ion} = f(T[°\mathrm{C}])$ are not needed in certain cases. The fact is that the overall shape of mass spectra, the ratio of amplitudes of components (e.g., hydrogen and helium isotopes), and the amplitudes of peaks at high heating temperatures provide an opportunity to estimate qualitatively the amounts of these components and the possibility of cleaning and degassing of protective graphite tiles. This was verified in subsequent studies.

Let us list the key stages of cleaning and degassing of graphite tiles:

— smoothing of the surface of tiles under running water by an abrasive paper with a stepwise reduction of the grain size from 50 to 7 μm (silicon carbide, SiC);

— preliminary cleaning of tiles with distilled water at a temperature of 80°C for 2 h;

— thorough drying of tiles under atmospheric pressure;

— cleaning of tiles in an ultrasonic bath in an intermediate alkaline medium (Kavisan-Multi U) at 50°C for 1 h;

— cleaning of tiles in a bath with bubbling (in distilled water) for 1 h;

— drying of tiles with hot air for 1.5 h;

— high-temperature annealing of tiles in an electric vacuum furnace for 5 h at 900°C and a residual pressure of $\leqslant 10^{-7}$ Torr.

When this fairly complex and labor-intensive cleaning procedure was complete, protective graphite tiles were again examined for the presence of sorbed gases. Figure 3, *a* presents a comparison between the typical mass spectra of graphite samples from the surface layer of tiles subjected to long-term plasma exposure in the Globus-M2 tokamak (blue curve; a color version of the figure is provided in the online version of the paper) and tiles that were processed in accordance with the above cleaning and degassing procedure (red curve). The amplitudes of peaks of the above-mentioned key gas components in the spectrum of uncleaned graphite heated to 500°C are more than two times greater than the amplitudes of peaks in the „blank“ spectrum in Fig. 2. An enlarged 0–5 a.m.u. region of the mass spectrum corresponding to the same temperature is shown in Fig. 3, *b*. An increased amplitude of the peak with a mass of 2 a.m.u. here may be attributed to the release of deuterium D_2^+, which was introduced repeatedly into the tokamak chamber. Peaks with

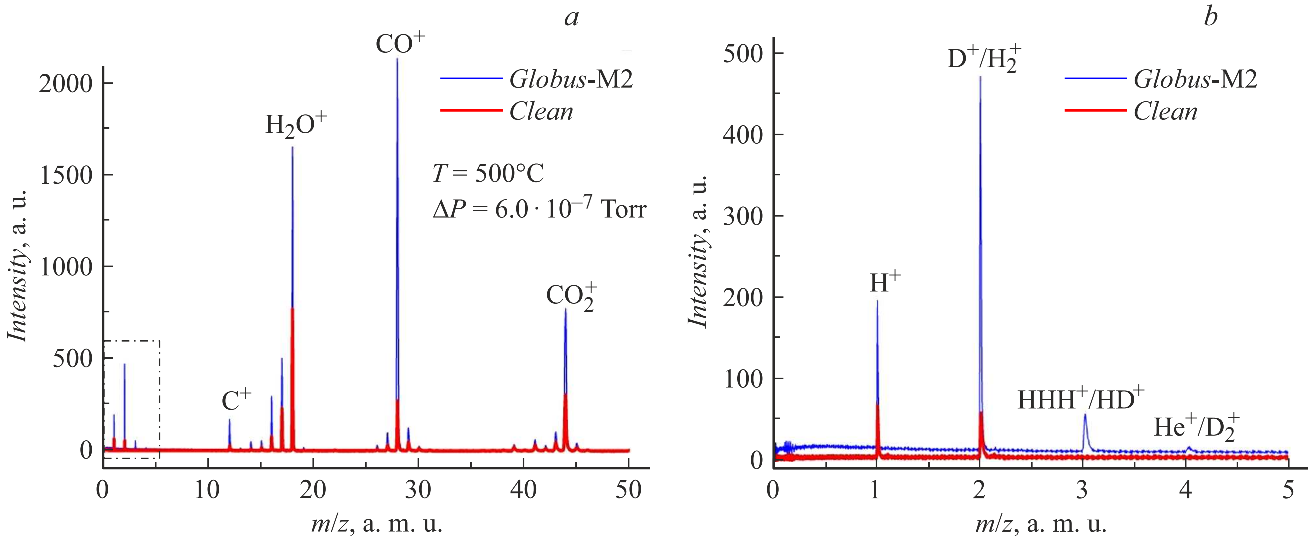

Figure 3. *a* — Mass spectrum of a graphite tile before (blue curve) and after cleaning (red curve). *b* — Enlarged 0–5 a.m.u. region (bounded by a dash-and-dot line in the left panel) of the mass spectrum of a graphite sample. A color version of the figure is provided in the online version of the paper.

masses of 3 and 4 a.m.u. are likely to be an unresolved $HHH^+-HD^+-{}^3He^+$ triplet and an unresolved ${}^4He^+-D_2^+$ doublet, respectively (blue curve). The spectrum of a cleaned graphite sample has no peaks at 3 and 4 a.m.u., since deuterium and helium were desorbed from graphite in the course of high-temperature annealing of tiles in vacuum.

A complex method for cleaning and degassing of graphite tiles, which served as a protective material of the first wall of the tokamak, was used in the study. The efficiency of this method was verified in mass-spectrometric analysis of gases released in the process of heating of graphite samples collected from protective tiles.

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Conflict of interest

The authors declare that they have no conflict of interest.

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