Plasma-Wall Interaction in the Open Divertor of Globus-M Spherical Tokamak

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Abstract. Composition, morphology and structure of the deposited (mixed) surface layers after preliminary carboboronization procedure (B/C:H layers deposition) and subsequent plasma-wall interaction in the different regions of Globus-M spherical tokamak have been analyzed. Globus-M to the best of our knowledge is unique in that its first wall is almost completely covered by RGTi protection tiles: currently about 90% of its inner vessel area facing to plasma, is covered by RGTi graphite tiles doped with 2 at.% of Ti and 0.3-0.7 at.% of Si. The mixed layer properties were examined by different diagnostic techniques (electron probe microanalysis, scanning electron microscope, X-ray diffraction, Rutherford backscattering, nuclear resonance reactions, thermal desorption spectroscopy, infrared Raman scattering, and secondary ion mass spectroscopy) after sample exposure to plasma during 3000-10000 tokamak pulses. It was found that mixed protective layers and/or deposits existed even in high flux regions (separatrix strike points).
The initial composition of the protective layers formed during boronization is dramatically modified during long-term plasma-wall interaction and resulted in significant intermixing of its components with inner vessel materials. Properties of the mixed layers deposited onto the surfaces intensively interacting with plasma, as well as the composition of the mixed layers deposited in the "shadowed" zones are discussed. Deuterium retention in Globus-M tokamak was estimated relying upon the data on deuterium concentrations in the mixed layers that were measured by different methods. It is revealed that deuterium was absorbed only in the mixed layers and its concentration vanished in the bulk of the tiles. Conditions of deuterium desorption are analyzed and merits of Ti doped graphite are discussed.

1. Introduction

Widely used carboboronization and boronization procedures are identified as the standard methods of impurity minimization in tokamaks. This is due to impurity and hydrogen isotopes are absorbed by deposited films providing plasma discharge stabilization [1, 2]. Properties of mixed layers deposited onto Globus-M divertor plates during plasma-wall interaction as well as studying of initial protection films features are described in [3-5]. Globus-M is the first Russian spherical tokamak built at A.F.Ioffe Institute in 1999 [6]. Currently about 90% of its inner vessel area facing to plasma is covered by RGTi graphite tiles (5-10 mm thick). To minimize chemical sputtering and improve a thermal shock resistance the graphite during manufacturing process was doped by metals, Ti in particular [7]. Although Ti doping process of carbon was proposed relatively long ago, Globus-M is the first spherical tokamak, and to the best of our knowledge is unique in that its first wall is almost completely covered by graphite protection tiles doped by Ti.

Paper is devoted to chemical composition and structural-phase analysis of deposits (mixed layers) obtained during primary boronization procedure performed numerously and following deposits modification during plasma-wall interaction in divertor and other areas of Globus-M tokamak. Deuterium retention and light element (C, B) contents in the mixed layers formed onto tile surfaces contacting with plasma and in plasma "shadow" are under scrupulous attention. Deposit property evolution during exposition to plasma in the tokamak is discussed. First data on investigation of dust originated during plasma-wall interaction are presented.

2. Samples obtained during tokamak experiments

First wall protection tiles of Globus-M tokamak (614 tiles totally) were manufactured from RGTi type of graphite which was doped with 2 at.% of Ti and 0.3-0.7 at.% of Si [7, 8]. The first wall area protected by tiles constantly grew up during tokamak operation which resulted in plasma parameters improvement. Carbon-boron layers were deposited several times a year [1] onto the plasma facing surface of vacuum vessel (including tile surface) (Fig. 1). During whole tokamak operation about 30 boronization procedures were made. Tokamak typically operates in relatively high density regime ($n \sim (3–10)\times10^{19}$ m^{-3}) with plasma current of 0.2-0.25 MA and toroidal magnetic field strength ~ 0.4 T on the vessel axis. Basic working gas is deuterium. Neutral beam injection of deuterium atoms (E = 22–29 keV, P = 0.4–0.6 MW) and ion cyclotron heating on fundamental harmonics of hydrogen minority (f = 7–9 MHz, P = 0.2 MW) are used as methods of auxiliary plasma heating. A high value of specific power deposition is achieved during auxiliary heating up to 1-2 MW/m^3. A high power density flux to the first wall is also realized. This is due to the close wall to plasma spacing in Globus-M, as well as to a small ratio of plasma wall area to plasma volume in the "spherical" geometry of plasma column. Power flux "focusing" effect along the separatrix footprint onto divertor plates, when plasma column is limited by magnetic field, increases the power density flux.

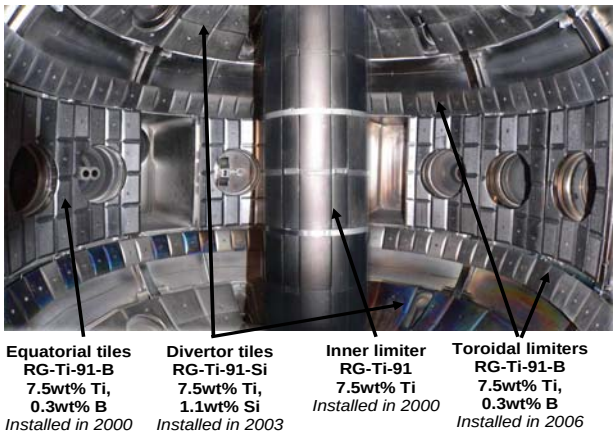

Fig. 1. Plasma facing surface (first wall) of the Globus-M vacuum vessel.

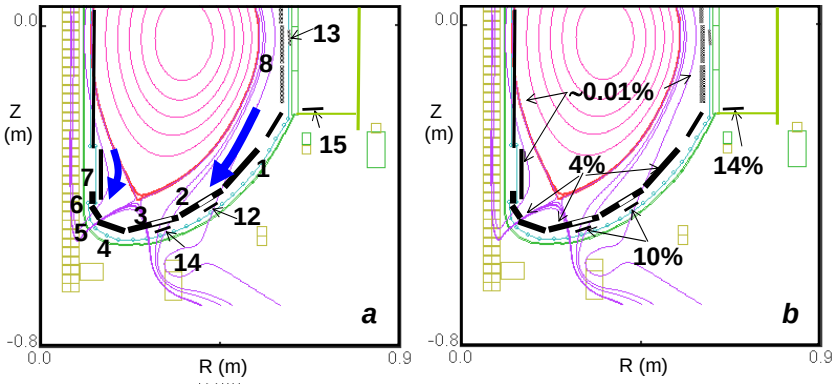

FIG. 2. a) lower part of Globus-M. Numbered are divertor RGTi tiles and samples exposed to plasma. b) deuterium retention coefficient in different areas of Globus-M first wall.

Lower part of Globus-M poloidal cross-section with one of possible magnetic plasma configuration enclosed is shown in Fig. 2a. As it was briefly mentioned, the divertor area of Globus-M vacuum vessel is protected by RGTi graphite tiles. Tiles 1-7 are placed side by side along the loop of poloidal vessel circumference. To detect the zones with maximal power deposition recently 10 Langmuir probes were installed onto the lower divertor plates. Ion

saturation current density distribution along the lower divertor plate circumference is shown in Fig. 3. The data are averaged over 100 tokamak pulses (~ 10 s). Also micrographs of corresponding divertor tile surfaces are depicted. One could see that maximal fluxes are recorded approximately in the regions of separatrix footprints. Supposing that about ¼ of the power deposited into tokamak discharge is directed to the lower divertor tiles with the area of ~0.6 m^2 in double X-point magnetic configuration one could estimate average power density deposition. In high auxiliary heating power discharges with (P_{OH}+P_{AH}) ~1.3 MW, average power density is about 0.5 MW/m^2. Maximal power flux density could reach several MW/m^2 in a narrow zone of scrape-off layer footprint onto divertor tile surface.

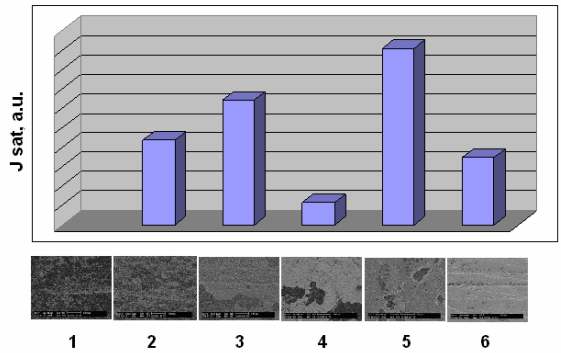

Fig. 3. Ion saturation current density (particle flux) distribution along the lower divertor plates (top). SEM micrographs of divertor tiles [3]. The sizes of the every micrograph are 500×600 μm. The numbering is in accordance with Fig. 2.

Part of the lower divertor tiles (1-6 see Fig. 2a and two more) were extracted from the vessel for the analysis after interaction with plasma during ~ 10000 discharges (exposition time ~ 100 s). Besides thin stainless steel probes (samples 12-15) manufactured from austenitic steel (12Cr18Ni10Ti, another definition is 304 stainless steel) were extracted from "shadowed" zones in which they were exposed to 2800 plasma discharges (exposition time ~300 s). Eight graphite tiles and five stainless steel probes were extracted from the vessel in total. The samples for the analysis of the deposits (mixed layers) formed in the zone of intensive plasma-wall interaction as well as in the "shadowed" zones were produced from extracted materials.

3. Methods of analysis

Composition, morphology and structure of the sample surface layers deposited onto divertor tiles as well as in the "shadowed" zones were analyzed by electron probe microanalysis (EPMA) [9], scanning electron microscope (SEM) and X-ray diffraction (XRD), respectively. Deuterium depth profiles in the layers deposited onto the divertor tiles were determined by the D(^{3}He,p)^{4}He nuclear reaction in a resonance-like technique [10]. To determine the D depth distribution, an analyzing beam of ^{3}He ions with energies varied from 0.69 to 3.2 MeV was used. D concentration profile in deeper layers on the divertor tiles was obtained with the computer program SIMNRA [11] by deconvoluting the proton yields measured at different ^{3}He ion energies.

In parallel the chemical composition of the mixed layers in the "shadowed" zone was analyzed by Rutherford backscattering spectrometry (RBS) and nuclear reaction analysis (NRA) with an electrostatic accelerator of deuterons in B.P. Konstantinov Institute. The boron/carbon ratio in the layer was determined by NRA comparing alpha and proton particle yields in ^{10}B(d,α)^{8}Be and ^{12}C(d,p)^{13}C reactions. Alpha-particles and protons originated in the reactions of 1 MeV deuterons with nuclei of boron and carbon isotopes. Deuterium upper content limit was estimated from the reaction of D(d,p)^{3}H [12 - 14].

Deuterium release from samples located in the "shadowed" zones (samples 12-15) was studied by thermal desorption spectroscopy (TDS) in the temperature range 300-1100 K [15]. Contents of boron, carbon, silicon and titanium in the mixed layers before and after TDS were

measured by EPMA. The retained contents of boron and carbon were determined also by EPMA after mechanical removal of surface films from the part of the samples.

4. Results

In the zone exposed to direct plasma fluxes (direct contact with plasma flowing along the field lines) the analyzed divertor tiles (2-4) did not suffer from destruction. At least inside 10 μm surface layer depth after 3-year of tokamak operation no changes were recorded by XRD structural analysis. RGTi material remained in well graphitized and textured mode with a basic plane faced to plasma (inter plane spacing 3.36 Å). Graphite matrix contained TiC inclusions which were visualized by relatively intensive Bragg reflections (Fig. 4).

All the samples prepared from divertor tiles were covered by mixed layers. The layers deposited onto surfaces experienced direct plasma fluxes and contained, besides carbon and boron, practically all the elements of vessel construction units and oxygen. A diffraction spectrum (Fig. 4) also demonstrates weak reflections from a boron carbide layer which was deposited onto the sample surface during boronization procedure. Some increase of line width with interplanar spacing 2.03 Å is connected with possible existence of iron and iron oxide particles on the tile surface. This was confirmed by XRD and SEM [3], as well as XRD analysis of dust collected from the lower divertor plate surfaces. Analysis of boron and carbon characteristic radiation lines testified that boron concentration decreased in mixed layers during plasma-wall interaction. Average thickness of mixed layers deposited onto the divertor tile surfaces was 2-3 μm.

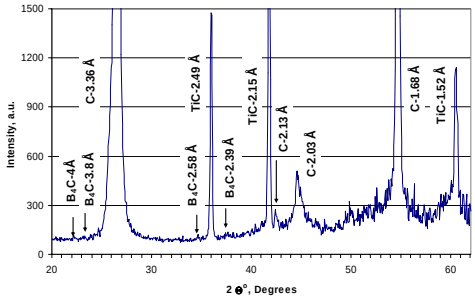

Fig. 4. X-ray diffraction spectrum of the surface layer (tile 3) recorded at the wavelength 0.154 nm (CuK_α radiation)

Deuterium retention in the layers deposited onto divertor plate surfaces (zone of direct contact with plasma) was estimated from NRA and TDS data. Intensive deuterium desorption took place in the temperature range 600-900 K (Fig. 5a) which was significantly less than an initial temperature of deuterium release from RGTi graphite exposed to deuterium plasma [15]. This confirmed that deuterium was basically captured by the mixed layers. Deuterium concentration in the divertor tile deposits was not more than 7-8 at.% (i.e., ~10^{18} D/cm^2) after 10000 tokamak pulses [3]. Minimal deuterium concentration corresponded to the maximal power flux zone (tiles 1 and 2). Deuterium concentration in the bulk of RGTi tiles did not exceed 2x10^{-3} at.%. This information was taken from tile 6 containing the maximal deuterium concentration after removing of 100 μm surface layer exposed to plasma.

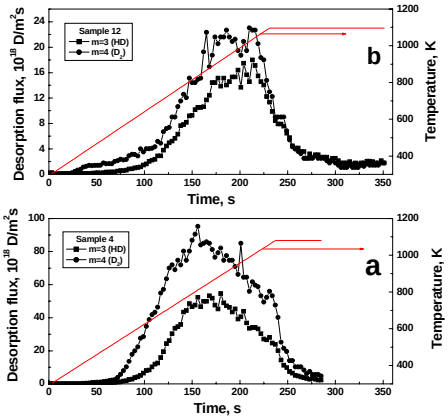

Fig. 5. a) – deuterium thermodesorption spectrum for the tile 4. b) - deuterium thermodesorption spectrum for the layer on sample 12. Linear heating rate is 3.2 K/s. The highest D_2 release is at 800 - 900 K

In the "shadowed" zones, which were screened from direct plasma flows along the field lines, the mixed layers were deposited onto

the stainless steel samples. The deposits were characterized by well developed surface (Fig. 6).

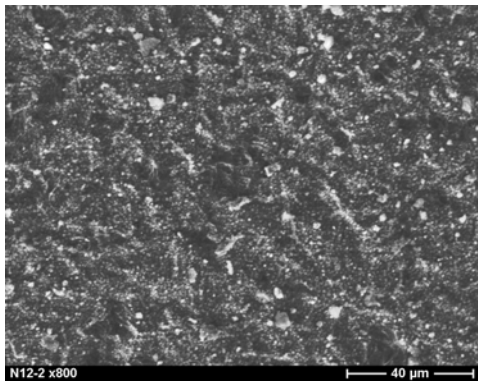

Fig. 6. SEM micrograph of the deposit on the sample 12. The deposit surface is quite rough.

The structure of the deposited layer (sample 12) was analyzed by IR Raman scattering method. A Raman spectrum of the film is shown in Fig. 7. A wide asymmetric band with two peaks at 1500-1600 and 1330-1350 cm^{-1} (G and D peaks of disordered carbon with sp^2 - bonds) is typical for Raman spectrum of amorphous hydrogenated carbon. Such data together with XRD data confirmed that the deposited mixed layers contained basically amorphous carbon phase. Also those contained particles of well graphitized carbon, titanium carbide (TiC) and iron oxide (Fe_2O_3). NRA and EPMA performed in parallel showed than boron concentration was lower than carbon one (Table 1). The layers also contained main stainless steel components (Fe, Cr, Ni). Comparative analysis of EPMA spectra taken before and after mechanical removal of the layers showed that boron-carbon layers contained O, Si and Ti too.

TABLE 1. BORON AND CARBON CONTENTS (SAMPLES 12-15, EXPOSURE TO PLASMA FOR 2800 DISCHARGES)

Sample	C, 10^{16} at/cm^2		B, 10^{16} at/cm^2	
	NRA	EPMA	NRA	EPMA
12	370	165	25	62
14	310	86	8.4	23
15	115	36	9.0	23

A discrepancy in carbon and boron contents measured by NRA and EPMA may be partially connected with difference in analyzing area. In NRA the information was obtained from ~ 7 mm^2 area, while EPMA utilized ~ 50×50 µm^2 beam, that resulted in much smaller area under analysis. So, dust particles contribution might be different.

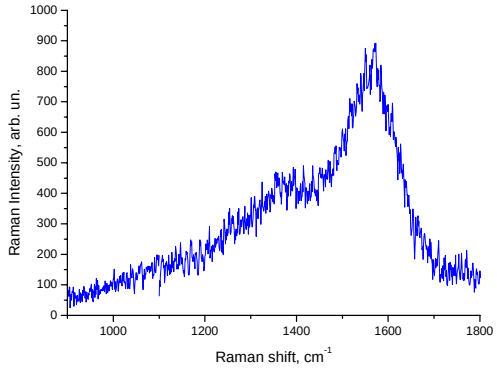

Fig. 7. Raman spectrum of the deposit from sample 12.

NRA and TDS data shown that the samples located in "shadowed" areas accumulated a significant amount of deuterium (Table 2). It should be noted that during thermal desorption masses 3 and 4 (HD and D_2 molecules, respectively) were measured only. The signal of mass 3 was delayed with respect to the signal of mass 4, which testified about H-D isotope exchange on a surface of TDS equipment, but not in the deposit. Thus, the protected B/C films contain mainly deuterium after exposure in tokamak deuterium discharges. Intensive deuterium release started at 600-700 K for all the investigated samples (Fig. 5b). At heating up to 1100 K, about 23, 45 and 35% of trapped

deuterium was released in form of D_2 molecules from 12, 14 and 15 samples, respectively.

TABLE 2. NRA AND TDS DATA ON DEUTERIUM CONTENTS IN THE MIXED LAYERS DEPOSITED ON THE SAMPLES 12, 14, 15. THE C AND B CONTENTS OBTAINED BY EPMA AFTER SAMPLES ANNEALING AT 1100 K

Sample	Deuterium content, 10^{16} at/cm^2		C, 10^{16} at/cm^2	B, 10^{16} at/cm^2
	NRA	TDS	EPMA	
12	<200	46	80	31
14	<125	56	25	17
15	<43	15	10	16

TDS analysis showed a lower deuterium release than could be expected from NRA measurements. EPMA performed after TDS measurement (which results in deposits annealing) indicated that the deposits sublimated. It is clear, that part of deuterium was released from deposits as its volatile compounds with boron and carbon.

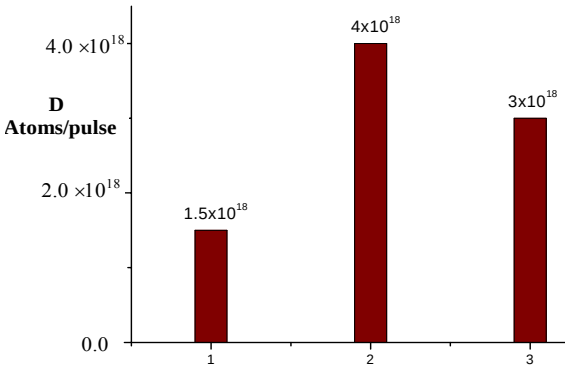

Fig. 8. The distribution of captured deuterium in the tokamak pulse across different vacuum vessel zones. 1 - divertor tiles surface. 2 - open part of vacuum vessel surface (shadow of divertor and limiter). 3 - inner surface of vessel ports.

The data obtained by different techniques provided deuterium retention estimate (upper limit). The distribution of captured deuterium in a tokamak pulse across different vacuum vessel zones is given in Fig. 8 as a histogram. Deuterium is mainly accumulated in the "shadowed" zones deposits. Taking for 100% the deuterium amount captured in the tokamak pulse, one can see that in the "shadowed" zone deposits (openings in the divertor plates for access to ports and toroidal belts of the vessel not covered by RGTi tiles) about 50% of deuterium is captured. A significant deuterium amount is captured inside vessel ports ~35%. And only about 15% of deuterium is captured by the deposits in the zone exposed to direct plasma fluxes.

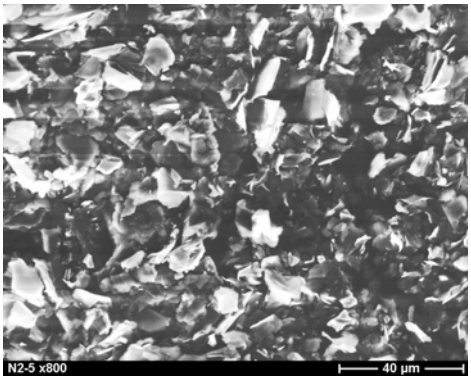

Fig. 9. SEM micrograph of dust particles collected from surface of the tile 2.

It was shown, after Globus-M vessel opening to the air, that on tile surfaces, mainly on the tiles 2 and 3 dust particles were accumulated with sizes up to 40 μm which was looking like broken stone (Fig. 9). XRD analysis showed that broken stone pieces contained particles of well graphitized carbon, titanium carbide (TiC) and iron oxide (Fe_2O_3). Amorphous carbon was present too. EPMA showed that the dust contained the following elements: C, O, Al, Si, Ti, Cr, Fe. A role of the dust in total deuterium retention is not clear yet, but seems not to be significant.

5. Discussion

The initial carbon-boron protection layers deposited onto the first wall surface of the Globus-M vessel during decomposition of dodecarboran powder ($B_{10}C_2H_{12}$) in the DC glow discharge

contained B/C:H with B/C ratio ≈ 1 [4]. An initial hydrogen concentration was up to 40-50 at.%. After 50 – 100 plasma discharges hydrogen concentration decreases significantly [5]. The layer thickness was less 3 μm. During Globus-M plasma discharges a significant change of a deposited layer content took place due to interaction of the layer with discharge products. Implantation of atoms C, Si, Cr, Fe, Ni, Cu, and W, which are sputtered from in-vessel components occurred as well as deuterium capture and accumulation in the surface layer.

Boron and carbon are dominating elements in the mixed layers deposited on the divertor tiles (zone exposed to direct plasma fluxes), as well as in the mixed layers formed in the "shadowed" zones screened from direct plasma fluxes. A boron/carbon concentration ratio in the deposited layers is changed in the favor of carbon during plasma-wall interaction. Additional analysis of a depth distribution of elements was performed by secondary ion mass spectroscopy (SIMS) profiling. The data were taken from sample 12 with secondary-ion microanalyser CAMECA IMS7f using primary O_2^+ and Cs^+ ions (Fig. 10). One can see that a carbon-boron layer is saturated by oxygen, mainly due to oxidation at the air before analysis. A carbon/boron concentration ratio differs from the initial one C/B ≈ 1 [4, 5]. During plasma - wall interaction the boron concentration in the layer decreases, this is in agreement with NRA and EPMA data. The boron concentration decreases with the layer depth. This may be connected with radiation-stimulated boron diffusion towards the surface with its subsequent sputtering during surface-plasma interaction.

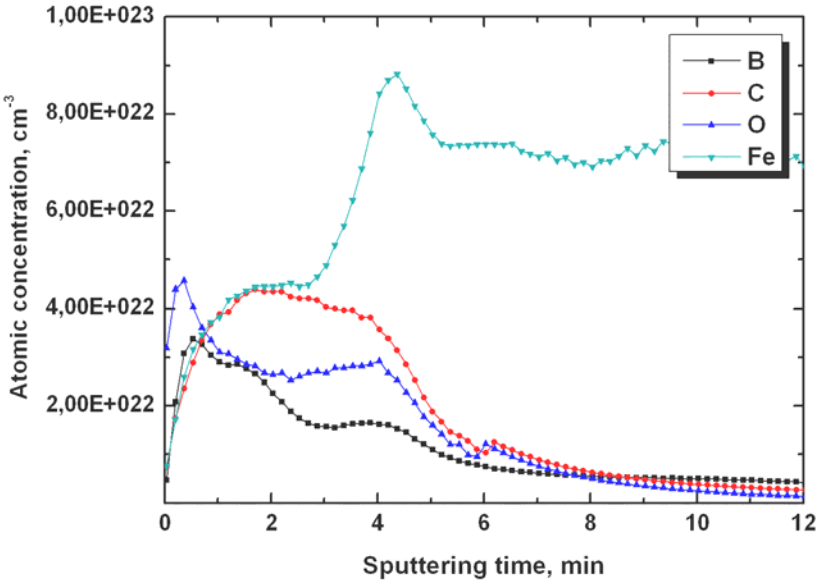

Fig. 10. SIMS depth profile of sample 12

Deuterium is captured by the mixed layers. Taking data from Fig. 9 and data on deuterium injection during the tokamak discharge by a gas puffing system we can derive an estimate of a retention coefficient. The upper limit of the retention coefficient in the Globus-M is ~28%. This does not contradict the data obtained from other tokamaks 3-30% [18]. The deuterium retention for different in-vessel zones is the following: the surface layers deposited in the zones exposed to direct plasma fluxes - about 4%, the "shadowed" zone deposits - about 14%, inside vessel port deposits - about 10% of the totally injected deuterium during each tokamak discharge. The estimated deuterium retention distribution is shown in Fig. 2 b.

6. Summary

Summarizing, we briefly describe main processes of protective carbon-boron film modification inside Globus-M tokamak equipped for the first time by the wall tiles manufactured from RGTi graphite. After boronization the protective layer is amorphous boron-carbon one saturated with hydrogen. Shortly after initial plasma-wall interaction the

hydrogen leaves the protective layer. Implantation of the sputtered elements of in-vessel components into the film occurs and transformation of the film into a mixed layer with a complex structure take place. The layer remains amorphous. Its thickness and element contents are dependent on plasma-layer interaction intensity (the minimum thickness is recorded in the zone of maximal loads). The mixed layer is a trap for hydrogen isotopes. The amount of captured hydrogen isotopes is also dependent on plasma-layer interaction intensity. The main hydrogen isotopes retention is observed in the "shadowed" zones. During heating up to 700-800 K the intensive release of hydrogen isotopes starts from the mixed layer, part of them releases together with layer components (due to "volatility" of layer). Important is "low" temperature of deuterium release from amorphous layers compared with significantly higher temperature specific for crystalline layers [19]. Hydrogen isotopes are not accumulated in the bulk of the divertor tiles. Quite positive experience of the RGTi graphite utilization in the Globus-M spherical tokamak is connected with its high density (2.2 g/cm^3), low erosion coefficient [7] and poor deuterium sorption ability (hence tritium in reactor). This may also explain a renovating of interest to Ti doped graphite as a promising armor material for plasma-facing components [20].

Acknowledgements. The work is supported by Russian academy of science, Rosnauka RF and RFBR grants No 06-02-16709, 06-02-08186, 06-03-32854, 06-08-00878, 07-02-13557, 08-02-13537ofi. SIMS analysis is made by the staff of the North Western Regional Center of Multi-User Equipment "Material Science and Diagnostics in Advanced Technologies".

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