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# Hydrogen isotope retention in B<sub>4</sub>C coating on RGT graphite under high heat fluxes of DIII-D divertor plasma.

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**Abstract** - The results of the investigation of retention and thermal desorption of hydrogen isotopes of B<sub>4</sub>C coated RGT ( a recrystallized graphite with high thermal conductivity, 600 W/mK) after the exposure to high heat flux in the divertor strike point region of DIII-D using the DiMES sample exchange system are reported. It is shown that the material is very promising for plasma facing elements of tokamaks.

## 2. EXPERIMENTAL PROCEDURE.

### 2.1 Plasma exposure.

B<sub>4</sub>C coated RGT sample (an SEM picture of the coating surface is shown in fig. 1) was inserted into the divertor floor of DIII-D using the DiMES sample exchange system [5].

The sample had a plasma facing surface of 5 cm diameter and during exposure is located at a major radius of 148.5 cm. During the exposure it was apparent from the observation of the sample surface with a CCD video camera that the insertion was not perfect.

## 1. INTRODUCTION.

Boron carbide coatings are promising to improve the performance of graphite as a plasma facing material in the divertor because these coatings provide reduced physical and chemical sputtering and reduced recycling of oxygen. Previous studies of the performance of B<sub>4</sub>C coatings produced by high temperature CVD process on different kind of graphites, like MPG-8, ATG, RGT ( a recrystallized graphite with high thermal conductivity, 600 W/mK) in plasma disruption simulation experiments under electron, ion, plasma fluxes [1,2] and in real tokamak conditions ( on MPG-8 and ATG) in tokamaks T-10 and DIII-D [2,3,4] have been reported. Under high power e-beam testing coatings on MPG-8 graphite survived at heat fluxes up to about 10 MW/m<sup>2</sup>, whereas coatings on RGT were undamaged at the highest tested power flux of about 13 MW/m<sup>2</sup>. Similar improved performance was seen in plasma gun tests. Tests of B<sub>4</sub>C coated MPG-8 in real tokamak conditions on T-10 and DIII-D showed very good overall performance. However, the data on the interaction of a CVD B<sub>4</sub>C coating with hydrogen isotopes in tokamak conditions, that is so important due to hydrogen isotopes recycling and fuel substance losses, were not obtained. The goal of this study was to investigate: 1) the hydrogen isotope retention of CVD boron carbide coating on RGT graphite after the exposure to DIII-D divertor plasma and 2) thermal desorption of the captured hydrogen isotopes. The exposure reported here was characterized by high incident heat fluxes resulting from unexpected sample misalignment. Thus, this exposure provided an evaluation of the B<sub>4</sub>C coated RGT in a divertor at heat fluxes exceeding 12 MW/m<sup>2</sup>, comparable to ITER conditions. Various plasma diagnostics provided the information on plasma conditions at the strike point. After removal from the vessel the sample surface was analyzed using SEM ( surface morphology), X-ray crystallography ( surface crystalline structure), Rutherford Backscattering (surface elemental composition), Nuclear Reaction Analysis (deuterium retention) and thermal desorption analysis (TD).

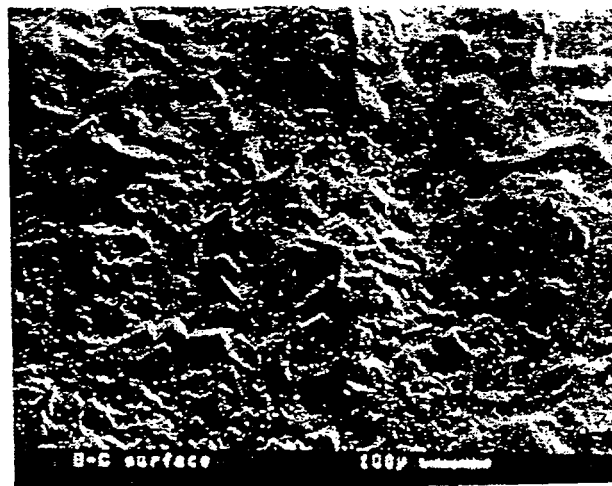


Fig.1 The surface of the B<sub>4</sub>C coating on RGT graphite.

The sample was tilted with respect to the surrounding divertor tile surfaces, with the result that some small region of the surface received about 4 times the average strike point flux. The sample was exposed to 14 deuterium ELMy H-mode discharges, with a total exposure time of 11 seconds. During the early phases of each discharge, the sample resided in private flux region, where it received negligible plasma flux. Near the end of each discharge the plasma conditions were set to the exposure requirements, and the divertor strike point ( region of peak flux) was moved onto the sample surface. During the set of 14 discharges plasma current was varied between 1.4 and 1.0 MA, the toroidal field was 2.1T, the injected deuterium beam power was typically 7 MW, with total radiated power of about 2.0 to 2.5 MW. Divertor plasma conditions were measured by a variety of diagnostics as follows: analysis of Langmuir probe traces in

the divertor floor yield  $T_e = 40-80$  eV and  $n_e = 4 \times 10^{19} \text{ m}^{-2}$ , divertor Thomson scattering yields similar results; analysis of IR TV data observing the strike point at a toroidally displaced position from DiMES gave a peak heat flux at the strike point of  $3.0 \text{ MW/m}^2$ . Simple geometric considerations indicate that the peak heat flux on the sample was about  $12 \text{ MW/m}^2$ . The magnetic field pitch angle at the nominal strike point location was  $1.5^\circ$ , however the apparent tilting of the sample surface increased the pitch angle to the  $\text{B}_4\text{C}$  surface significantly (in the neighborhood of  $6^\circ$ ).

## 2.2 Methods of post-exposure sample analysis.

After removal the sample surface was analyzed by Scanning Electron Microscopy (SEM) and X-ray crystallography (RSA) to analyze morphological and phase changes in the coating.

Rutherford Backscattering (RBS) was used to evaluate the changes in chemical composition of the surface after exposure and finally, the retention of deuterium in the coating was measured by counting protons from the  $\text{d}(\text{He}^3, \text{p})\alpha$  nuclear reaction with 700 KeV  $\text{He}^3$  beam (NRA). This analysis give the areal density of deuterium within  $1 \mu\text{m}$  of the surface. The beam spot size for RBS and NRA was  $1 \times 1 \text{ mm}^2$ .

To carry out TD analysis samples from undamaged and damaged areas were ohmically heated from 300K to 1400 K at about 6 K/s linear ramp rate. The temperature was measured with an optical pyrometer. The pressure during the TD measurement was  $1 \times 10^{-5} \text{ Pa}$ . Desorbed  $\text{D}_2$  and HD molecules were analyzed by a calibrated quadrupole mass spectrometer (QMS). The  $\text{CD}_4$  signal was negligible.

## 3. EXPERIMENTAL RESULTS.

SEM, RSA and RBS showed that everywhere even in the most loaded region, the  $\text{B}_4\text{C}$  coating is preserved without changing its chemical and crystalline structure. SEM showed regions of resolidification formed due to some melting during plasma exposure. More detail of these results were reported at the 12 PSI conference [6]. The initial thickness of the coating was  $175 \mu\text{m}$  and about  $45 \mu\text{m}$  was eroded in the most damaged spot. The composition everywhere is consistent with  $\text{B}_4\text{C}$ , even in the damaged area. The concentration of oxygen is seen to be almost doubled in the damaged region ( $4.5 \times 10^{17} \text{ O/cm}^2$ ) compared to the undamaged surface ( $2 \times 10^{17} \text{ O/cm}^2$ ). The increase in O concentration could be initiated by boron sputtering and activation of boron atoms in the near surface region by the incident heat and particle flux, resulting in higher B reactivity with O followed by formation of oxides.

The retention of deuterium was measured using NRA along lines both parallel and perpendicular to the magnet field (fig. 2). The measured areal concentration is about  $(2-3) \times 10^{17} / \text{cm}^2$  in the undamaged area, similar to previous measured retention for graphite in DIII-D under similar plasma conditions. However, in the damaged zone, the retention is much higher, about  $12 \times 10^{17} \text{ D/cm}^2$ . This increase may be due to the increase in particle flux and the thickness of deposited layer as well due to the unexpected tilting of the surface. The development of the coating surface and the increase of the density of radiation defects as well as radiation stimulated

diffusion of D in  $\text{B}_4\text{C}$  may also contribute. The relatively high electron temperature, 80 eV, of the plasma would imply a  $\text{D}^+$  incident kinetic energy of about 300 eV, with the tail of the distribution extending higher.

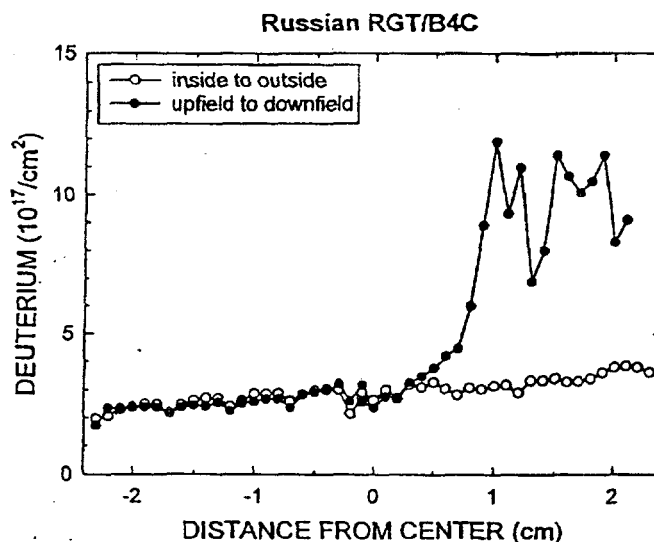


Fig.2 Deuterium retention, NRA data.

TD spectra of  $\text{D}_2$  and HD molecules released from undamaged and damaged areas of CVD  $\text{B}_4\text{C}$  coating on RGT graphite are presented in Fig. 3.

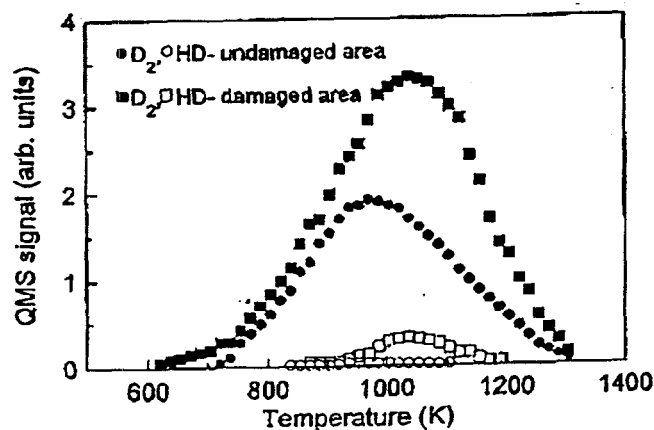


Fig.3 Thermal desorption of  $\text{D}_2$  and HD molecules from undamaged and damaged areas of CVD  $\text{B}_4\text{C}$  coating on RGT graphite/ Heating rate was about 6K/s.

The original QMS signals are shown as a function of temperature. The temperature range of deuterium release is the same for the undamaged and damaged areas, however the peaks of D release for these regions are different - for the damaged spot the peak was observed at 1040K and for the undamaged spot - at 960K. The  $\text{D}_2$  TD spectrum of the damaged area, compared to that of undamaged area, showed the following features: (1) the  $\text{D}_2$  peak position is 80 K higher, (2) the integrated peak intensity is a factor 2.5 higher. The shift of the

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peak maximum can be explained by the higher temperature of the damaged area as well as appearance of a graphite phase during irradiation. It is known that graphite has desorption temperatures higher [8]. The additional retention could be connected with the increasing surface area of CVD B<sub>4</sub>C resulting from the erosion at the plasma exposure and diffusion of deuterium beyond the kinetic ion range. It is important to note that ion fluxes onto different regions of the sample surface varied during the exposure period resulting in different heating of the sample surface. It is obvious that at temperatures higher than 1300 K, CVD B<sub>4</sub>C has no D uptake, so the character of the obtained spectra demonstrates that in the case, D was taken at comparatively low temperatures about 600-700K, in normal regime discharges.

We would like to mention that the second peak of the D desorption for RGT graphite is observed at 1160K [9], least 100° more then we have seen for our CVD B<sub>4</sub>C coating. This result means that the coating is more preferable from the point of H isotope recycling.

#### 4. CONCLUSIONS.

A B<sub>4</sub>C coated RGT sample was exposed in the DIII-D divertor using the DiMES sample exchange system. The sample received an estimated heat flux of 12 MW/m<sup>2</sup> for a total of 11 seconds. The coating remained intact everywhere, and maintained its chemical and phase composition. The retention of deuterium was seen to increase in the high heat flux region, approximately proportional to the increased particle flux.

The measured deuterium areal concentration is about  $(2-3) \times 10^{17}/\text{cm}^2$ . In the damaged zone, the retention is much

higher, about  $12 \times 10^{17} \text{ D}/\text{cm}^2$ . The temperature ranges of deuterium releasing are the same for the undamaged and damaged areas, however the peaks of D release for these regions are different - for the damaged spot the peak was observed at 1040K and for undamaged spot at 960K.

The results of this exposure under rather extreme conditions, along with previous data in tokamaks and in e-beam and plasma-beam simulation devices, indicate that thick CVD B<sub>4</sub>C coating could provide a long lasting "boronized" surface in high heat flux tokamak regions and compared to other carbon based materials has some advantages from the point of view of hydrogen recycling.

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