



Deuterium retention of DIII-D DiMES sample

Y. Yamauchi ^{a,*}, Y. Hirohata ^a, T. Hino ^a, K. Masaki ^b, M. Saidoh ^b, T. Ando ^c,
D.G. Whyte ^d, C. Wong ^e

^a Department of Nuclear Engineering, Hokkaido University, Kita-13, Nishi-8, Kita-ku, Sapporo 060, Japan

^b Naka Fusion Research Establishment, JAERI, Naka-machi, Naka-gun, Ibaraki 311-01, Japan

^c ITER-JCT, Garching Joint Work Site, Boltzmannstraße 2, 85748 Garching, Germany

^d University of California – San Diego, 9500 Gilman Drive, La Jolla, CA 92093-0417, USA

^e General Atomics, P.O. Box 85608, San Diego, CA 92186-9784, USA

Abstract

The deuterium retention property of B₄C converted graphite and isotropic graphite exposed to DIII-D deuterium plasma was examined by using a technique of thermal desorption spectroscopy. Major outgassing species were HD, D₂ and CD₄ in both the graphite and the B₄C. In the case of the graphite, the ratios of deuterium desorbed in the forms of HD, D₂ and CD₄ to the total desorption amount of deuterium were 40%, 27% and 33%, respectively. In the case of the B₄C, which was covered by carbon due to redeposition, these ratios were similar to those of the graphite. In a thermal desorption spectrum of deuterium, three desorption peaks appeared in both the graphite and the B₄C covered by the redeposition layer. At low temperature region, the desorption rate of deuterium for the B₄C covered by the redeposition layer was larger than that of the graphite. From two dimensional distribution of deuterium retention, it was seen that the retained amount at the electron drift side was quite large. The amount at the ion drift side and the edge of inward major radius was also observed to be large. The average retained amount of the graphite was almost the same as that of the B₄C covered by the redeposition layer. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Boron carbide; Deuterium inventory; DiMES; Thermal desorption; Spectroscopy

1. Introduction

Graphite materials have been widely used as plasma facing materials (PFMs), because of several advantages, such as high thermal conductivity and high thermal shock resistance [1–3]. However, the graphite was chemically eroded due to hydrogen isotope particles and oxygen impurities [4–6]. Recently, surface modifications of plasma facing wall, such as boronization, have been carried out in order to getter the oxygen impurity and suppress the chemical erosion, resulting the improved plasma confinement [7–11].

The hydrogen isotope retention in the plasma facing material affects on the enhancement of the hydrogen recycling and the tritium inventory. If the amount of

retained hydrogen in the plasma facing wall becomes large, the hydrogen re-emits into the plasma during the discharge, and then the hydrogen recycling will be enhanced. Therefore, it is necessary to evaluate the hydrogen retention properties of PFMs, and to develop suitable conditioning methods in order to reduce the hydrogen retention in PFMs.

In the present study, B₄C converted graphite and isotropic graphite were exposed to DIII-D deuterium ohmic plasmas, by using a DiMES probe. For this DiMES sample, the deuterium retention properties, namely two dimensional distribution of deuterium retention and the thermal desorption properties of deuterium, were evaluated.

2. Experiment

The DiMES sample was made by ATJ graphite. The diameter of this sample was 47.75 mm. The half of the

* Corresponding author. Tel.: +81 11 706 7194; fax: +81 11 709 6413; e-mail: yamauchi@apollo.hune.hokudai.ac.jp

surface area exposed to deuterium plasmas was B_4C converted graphite made by a chemical vapor reaction, $2B_2O_3 + 7C \rightarrow B_4C + 6CO$. This treatment was carried out at Hitachi Chemical. The other half area was the graphite (ATJ graphite). The thickness of B_4C layer was about $340 \mu m$. The concentration of boron was estimated as approximately 70–75% before the plasma irradiation. Before and after the B_4C conversion, the sample was polished by a diamond paste. Before the installation into the DIII-D divertor region, the DiMES sample was degassed for few hours at 673 K in the vacuum. After that, the sample was placed at near the outer divertor trace of DIII-D. Fig. 1 shows the location of the DiMES sample and the direction of incoming plasma. The DiMES sample was exposed to the deuterium ohmic plasma with a total discharge time of about 15 s. An average heat flux was 500 kW/m^2 , an electron temperature was 30–35 eV and a plasma density was $1.5 \times 10^{19} \text{ m}^{-3}$. During the discharge, a maximum temperature of the DiMES sample was approximately 373 K.

After the plasma irradiation, the surface of the DiMES sample was cut into 52 pieces with a size of $5 \times 5 \times 3 \text{ mm}^3$ for the measurement of thermal desorption spectroscopy (TDS). The sample piece was introduced into TDS apparatus shown in Fig. 2. After the evacuation for 2.5 h, the sample piece was heated linearly from RT to 1273 K with a ramp rate of 0.5 K/s by an infrared furnace. The sample temperature was monitored by a thermocouple spot-welded at sample holder

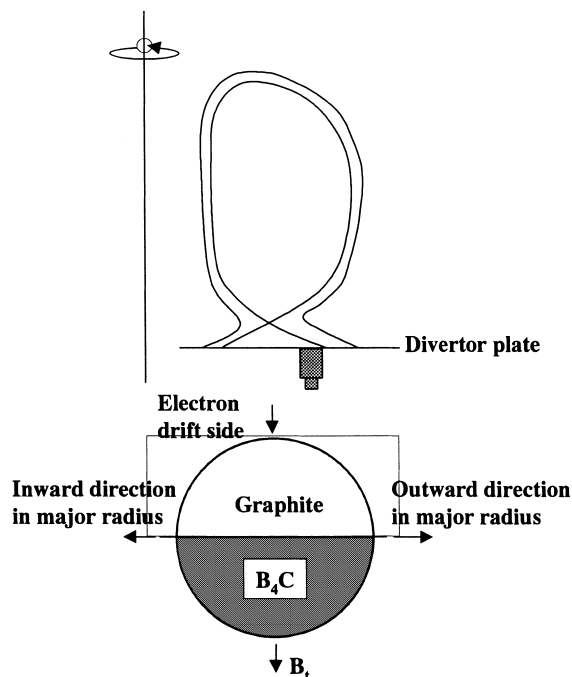


Fig. 1. DiMES probe sample.

made by tantalum. During the heating, the desorption rates of gas species including deuterium atoms, e.g., HD, D_2 and CD_4 , were measured by both partial pressure and effective pumping speed [12]. The background pressure during the TDS measurement was about $1 \times 10^{-6} \text{ Pa}$. The amount of retained deuterium was obtained by time-integration of the desorption rate of the deuterium species. In addition, the depth profiles of atomic composition of B_4C converted graphite surfaces were analyzed by Auger electron spectroscopy with 3 keV Ar^+ ion etching.

3. Results

Major outgassing species of the surface irradiated by the deuterium plasma were HD, D_2 and CD_4 in both the graphite and the B_4C surfaces. No desorption of boron hydride was observed. In addition, little other hydrocarbon such as C_2H_6 was emitted from the sample. This desorption property was similar to other work [13,14]. The ratios of deuterium desorbed in forms of HD, D_2 and CD_4 to the total desorption amount of deuterium in the case of the graphite was 40%, 27% and 33%, respectively. In the case of the B_4C surface, the fractions of desorption amount desorbed in forms of HD, D_2 and CD_4 were 40%, 21% and 39%, which was similar to those for the graphite. However, the fraction of CD_4

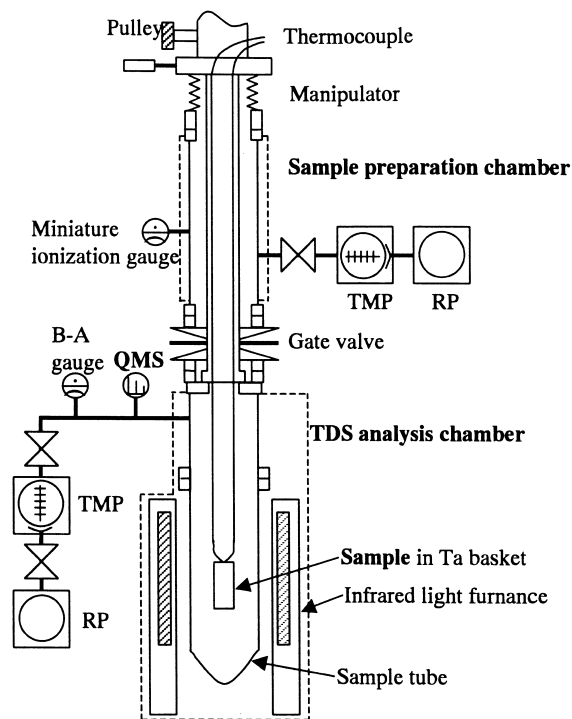


Fig. 2. Apparatus of thermal desorption spectroscopy (TDS).

desorption was observed to be large, that was different from the results of other experiments for B₄C [15–17]. The reason was considered that the surface of B₄C was covered by the carbon due to the redeposition from the first wall during the discharge (Fig. 6). In addition, the redeposited carbon may be amorphous. This redeposition was observed in other experiment of DIII-D [13,18].

Fig. 3(a) shows thermal desorption spectra of HD and D₂ for the graphite surface. In the HD desorption spectrum, major peak appeared at about 1100 K with a small shoulder at about 900 K. D₂ desorption behavior was similar to that of HD. The peak temperatures of these hydrogen isotopes were similar to those of isotropic graphite irradiated by hydrogen ion (5 keV H₃⁺) [15]. Fig. 3(b) shows thermal desorption spectra of HD and D₂ for the B₄C surface covered by the redeposition layer. In the desorption spectrum of HD, three peaks appeared. Compared with the case of the graphite, the low temperature peak at 800 K was higher. In the desorption spectrum of D₂, the broad peak was observed at the temperature region from 800 to 1100 K. Fig. 4 shows the desorption spectra of CD₄ for the graphite

and the B₄C surfaces. In both the cases, two major desorption peaks appeared at 300 and 700–900 K. In the case of B₄C, the desorption at the low temperature region was dominant, compared with that of the graphite, similarly to those of the desorptions of HD and D₂. In our previous hydrogen ion irradiation experiment for B₄C, the low temperature peaks in the hydrogen and methane spectra became larger, compared with the case of graphite [15,19]. So, it can be assumed that the desorptions of HD, D₂ and CD₄ at the lower temperature region are due to the deuterium trapped in the boron content.

Fig. 5 shows two dimensional distribution of deuterium retention. The amount of retained deuterium was considerably large at the edge of the electron drift side. In addition, the amount was large at the ion drift side and the edge in inward region of major radius.

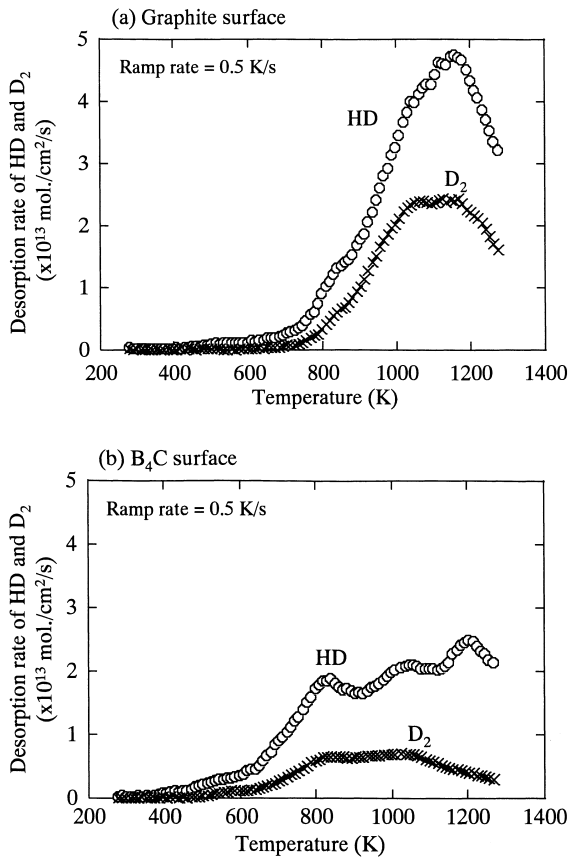


Fig. 3. Thermal desorption spectra of HD and D₂ for graphite surface (a) and B₄C surface covered by redeposition layer (b).

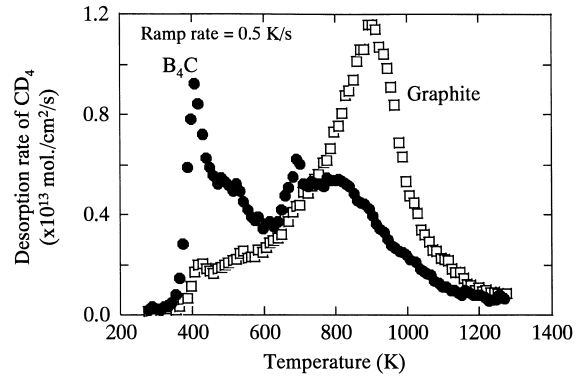


Fig. 4. Thermal desorption spectra of CD₄ for graphite and B₄C surfaces covered by redeposition layer.

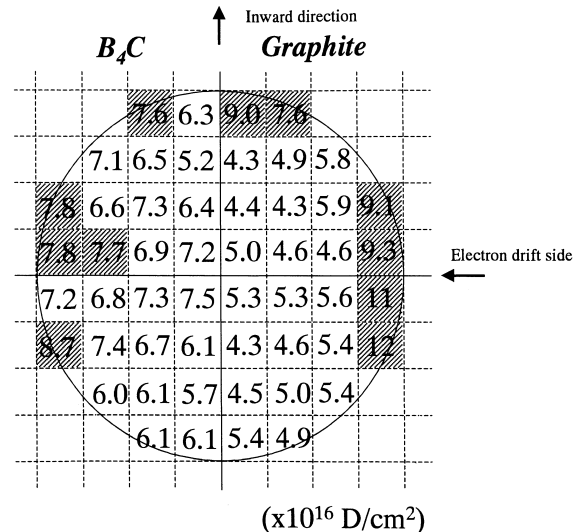


Fig. 5. Two dimensional distribution of deuterium retention.

retention may be due to the large particle flux. The average amount of retained deuterium for the B₄C surfaces covered by the redeposition layer was 6.9×10^{16} D/cm², which was a little larger than that of the graphite surfaces (6.1×10^{16} D/cm²). Except the value at the edges of sample, the average amount of retained deuterium for the graphite surfaces was 4.9×10^{16} D/cm². On the other hand, the average amount of the B₄C surfaces covered by the redeposition layer was about 1.5 times larger than that of graphite (7.0×10^{16} D/cm²). This result was similar to that obtained in our previous hydrogen ion irradiation experiment [15].

Fig. 6 shows depth profile of atomic composition for the B₄C surface after the TDS measurement. At the top surface, the carbon concentration was approximately 90 at.%. The boron concentration at the top surface was only 5 at.%. In the bulk region, boron concentration was about 70 at.%. From this result, it is seen that the boron concentration at the top surface was much reduced, compared with the case before the irradiation. The reduction of the boron concentration may be due to the redeposition of carbon from the first wall. This assumption is also supported by that the CD₄ desorption for the B₄C covered by the redeposition layer was observed to be large.

4. Conclusion

The deuterium retention properties of the DiMES probe sample were examined by using a technique of thermal desorption spectroscopy. Major outgassing species from both the graphite and the B₄C covered by the redeposition layer exposed to DIII-D deuterium plasma, were HD, D₂ and CD₄. The ratios of deuterium desorbed in forms of HD, D₂ and CD₄ to the total desorption amount of deuterium, in a case of the B₄C

covered by the redeposition layer, were similar to those in a case of the graphite. The increase of the CD₄ desorption for the B₄C is assumed to be due to the redeposition of carbon from the first wall. In the thermal desorption spectrum of deuterium, three desorption peaks appeared in both the graphite and the B₄C covered by the redeposition layer. The low temperature peak in the B₄C covered by the redeposition layer was large, compared with the case of the graphite. It is presumed that the desorption at this low temperature region is due to the detrapping from the B–D bond.

From two dimensional distribution of deuterium retention, the retained amount at the electron drift side was very large, compared with other positions. The retained amounts at the ion drift side and the edge of inward major radius were also large. The average amount of retained deuterium for the B₄C covered by the redeposition layer was 1.5 times larger than that for the graphite.

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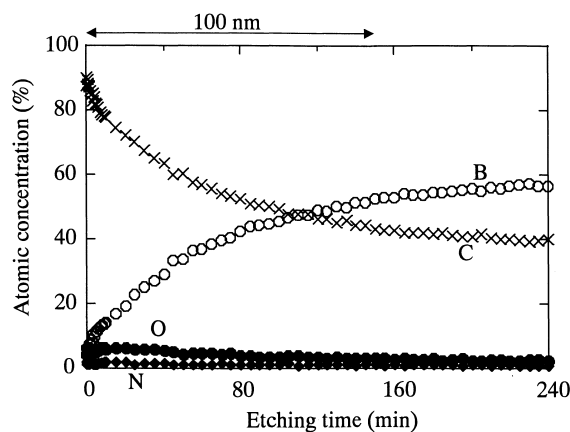


Fig. 6. Depth profile of atomic composition in B₄C surface after the TDS measurement.