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# Hydrogen retention in divertor tiles used in JT-60 for hydrogen discharge period

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## Abstract

Hydrogen retention characteristics of graphite divertor tiles used in JT-60 for hydrogen discharge period were investigated by the thermal desorption spectroscopy. Most of the tiles were covered with re-deposited layers of the maximum thickness of 70 μm. Total amount of hydrogen desorbed from each tiles was proportional to the thickness of the re-deposited layer on the tiles. Hydrogen desorption from substrate determined after the removal of the re-deposited layer was quite small compared to that from the re-deposited layers. Assuming homogeneous hydrogen distribution in the re-deposited layers, H/C ratio in the re-deposited layer was approximately 0.015. This is much smaller than those observed in JET and other devices. The re-deposited layers are very likely subjected to high temperatures because of their poor thermal contact to the substrate owing to their porous structure.

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## 1. Introduction

The accumulation of tritium in in-vessel components of thermonuclear fusion devices is a major safety concern. Therefore it is a very important issue to evaluate retention characteristics of hydrogen isotopes (T, D, H) in in-vessel components of current plasma devices [1–6]. We have been studying the retention of hydrogen isotopes in plasma facing graphite tiles exposed to both deuterium and hydrogen discharges in JT-60U by using various methods under the joint research between Japanese universities and JAERI [7–10]. We have found that most of tritium produced by the D–D reactions was implanted into depth of micrometer range, and tritium areal distribution was independent of carbon deposition profiles, while H and D seem to be co-deposited with carbon. We have tried to evaluate the hydrogen isotope

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retention in the divertor tiles quantitatively by the thermal desorption spectroscopy (TDS). However, for those tiles exposed to both H and D discharges, different depth profiles of H and D and isotope mixing made the evaluation very difficult [11].

In this study, therefore, we have focused on the tiles used as open divertor tiles in JT-60, which were exposed only to H discharges and made TDS to examine characteristics of hydrogen retention. The depth profiles of hydrogen retained in the graphite tiles was also investigated by secondary ion mass spectroscopy (SIMS) and elastic recoil detection (ERDA). Crystal structure of the re-deposited layers was also analyzed by Raman spectroscopy.

## 2. Experimental

Fig. 1 shows the cross-sectional view of the vacuum vessel of JT-60 with an open divertor structure and lower X-point configuration (a), and sampling positions in three divertor tiles (1Af14D-1, 1Af14D-2, 1Ag14D-1) (b). Thickness profiles of the re-deposited layers on the tiles measured [12] as well as surface temperatures of the tiles for one particular divertor discharge [13] are plotted against the poloidal distance from the inboard end of the tile in Fig. 1(c). All tiles were exposed to total of 1800 hydrogen discharges with the limiter configuration including 300 divertor discharges during January 1988 to October 1988. Operational temperature of the vacuum vessel and the base temperature of the tiles were kept at 573 K. Most of the tiles was covered with re-deposited layers of the maximum thickness of 70 μm as shown in Fig. 1(c). These divertor tiles were made by isotropic graphite (ETP-10, Ibiden). For TDS analysis, eleven samples (8 mm × 8 mm 1 mm) were cut from the tiles to include the surface area; five from the inner strike region (Sample 1–5), three from the private flux region (Sample 6–8), three from the outer strike region (Sample 9–11) (seen Fig. 1(b)). One sample from which re-deposited layers were completely removed (referred as a removed sample hereafter) was also analyzed by TDS to compare hydrogen retention in the re-deposited layers and that in the substrate.

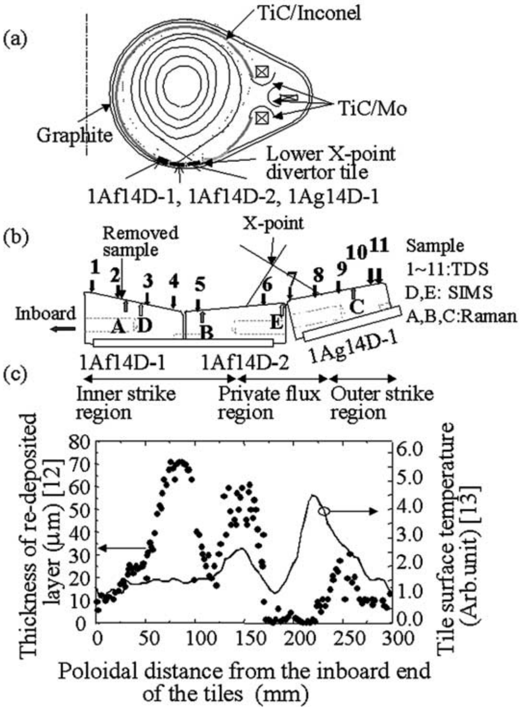

Fig. 1. Cross-sectional view of the vacuum vessel of JT-60 with an open divertor structure and lower X-point configuration (a), sampling positions in divertor tiles (b), and profiles of thickness of the re-deposited layer [12] and surface temperature for one particular divertor discharge [13] as a function of the poloidal distance from the inboard end of the tile (c).

For TDS measurements, the sample was put on a sample holder and heated from room temperature to 1273 K with a constant ramp rate of 0.42 K/s, and then was held at 1273 K for 10 min. The dominant mass numbers observed by QMS during the heating were 2($H_2$), 15($CH_4$), 26($C_2H_4$), 30($C_2H_6$) and 39($C_3H_8$).

Hydrogen depth profile in the re-deposited layer of a tile located in the private flux region during different hydrogen discharge campaign was also determined by ERDA, in which helium ions with energy of 1.7 MeV were used as a primary beam. The depth profiles of hydrogen in D and E samples (see in Fig. 1(b)) were also analyzed by SIMS using 5 keV Cesium ions ($Cs^+$) as a primary beam. In SIMS, the negative secondary ion intensity of hydrogen was normalized by that of $^{12}C$ for comparison. Laser Raman Spectrometry was applied to examine microstructure of the samples A, B and C indicated in Fig. 1(b).

## 3. Results

The most dominant species desorbed from the samples was $H_2$ molecule. Hydrocarbons such as $CH_4$, $C_2H_4$, $C_2H_6$ and $C_3H_8$ were also desorbed as minor desorption species. Fractions of $H_2$ gas to the total amount of hydrogen atoms retained in the samples are approximately 80–70%. Fig. 2 shows thermal desorption spectra of $H_2$ gas (a) and $CH_4$ gas (b) for the samples covered by the re-deposited layers with different thicknesses, and for the removed sample. In these TDS spectra, desorption peaks of $CH_4$ and $H_2$ appeared around 750 K and 973 K, respectively. One can see that desorption rate of $CH_4$ and $H_2$ increased with the thickness of

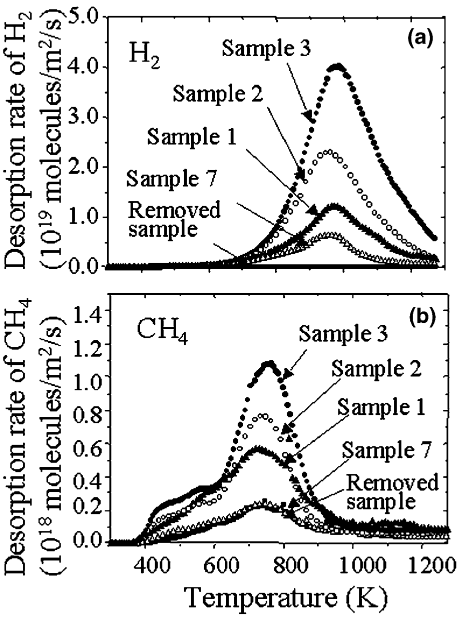

Fig. 2. Thermal desorption spectra of $H_2$ gas (a) and $CH_4$ gas (b) for the sample covered by the re-deposited layers with different thicknesses, and for the removed sample (the sample on which re-deposited layers were completely removed). Thickness of the re-deposited layer of the sample 1, 2, 3, 7 are 10, 25, 50, 2 μm, respectively.

the re-deposited layers. Fig. 3 shows the total amounts of hydrogen desorbed from the sample as a function of the thickness of the re-deposited layers. The total amount of the retained hydrogen in the samples were 0.7–7.5 × $10^{22}$ H/m$^2$. The amount of hydrogen desorbed from the removed sample was also plotted in the figure. Hydrogen desorption from the removed sample was quite small compared to those from the samples with the re-deposited layers, indicating that hydrogen was mainly retained in the re-deposited layer. In addition, one can be note that the total amount of the retained hydrogen in the samples shows very good linear relationship with the thickness of the re-deposited layers except two samples. This indicates hydrogen was retained in the re-deposited layers uniformly with constant hydrogen concentration irrespective of their thickness or poloidal position. From the slope of the linear relationship (straight line) we can determine hydrogen concentration in the re-deposited layers as 1.32 × $10^{21}$ H/m$^2$/1 μm, assuming carbon density of the re-deposited layers is the same as that of the substrate graphite, 1.75 g/cm$^3$. Accordingly, H/C ratio in the re-deposited layers is ~0.015, which is much smaller than those observed in JET [2,4,6] and other low temperature operating machines [6,14,15] where H/C is 0.1–0.4.

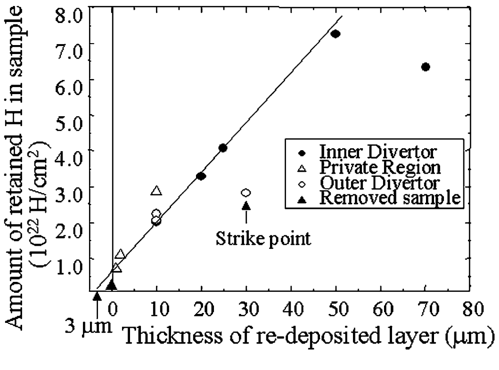

Fig. 3. Total amounts of hydrogen desorbed from the samples as a function of the thickness of the re-deposited layers.

It is also important to note that as shown in Fig. 3, when the linear relationship is extrapolated to zero concentration, the straight line crosses at ~3 μm in the thickness of the re-deposited layer. This clearly indicates that the substrate also retained hydrogen. Interestingly, this extrapolated retention is much larger than the total retention in the removed sample. This means that hydrogen retention in the substrate just beneath the re-deposited layers was not small but likely decreased exponentially in the scale of μm. Consequently the hydrogen retention in the removed sample was much smaller because certain thickness of the substrate was also lost during the removing the re-deposited layers.

In Fig. 3, there are two exceptional data points showing much less hydrogen retention in the samples. One is assigned as "strike point". This corresponds the tile at the strike point in the outer strike region where was covered by the thick re-deposited layer (~30 μm). Nevertheless the H/C ratio was very small. Because the "strike point" is the position where the strike point hit most frequently during hydrogen discharges and surface temperature must be the highest among all measured samples as shown in Fig. 1(c). Thus the re-deposited layers on the strike point must be subjected to higher temperatures to release once retained hydrogen.

Fig. 4 shows depth profiles of SIMS represented by signal intensity ratios of H/$^{12}$C for the inner divertor tile (sample D) and the private flux region tile (sample E) within the depth of 1.7 μm. Depth profiles of ERDA represented by H/C ratio of the private flux region tile were also shown for comparison. The thicknesses of the re-deposited layers of D and E samples were 40 and 1 μm, respectively. The surface concentration was higher for the sample E, but it decreased exponentially with the depth, while the concentration of the sample D having the thick re-deposited layers kept rather constant with the depth. This again confirms the above discussion that the hydrogen concentration in the re-deposited layers is constant. As shown in Fig. 4, in the tile located in the private flux region, where was covered only thin re-deposited layers, H/C ratio within the depth of 300 nm was observed to be ~0.05 by ERDA, while H/C ratio

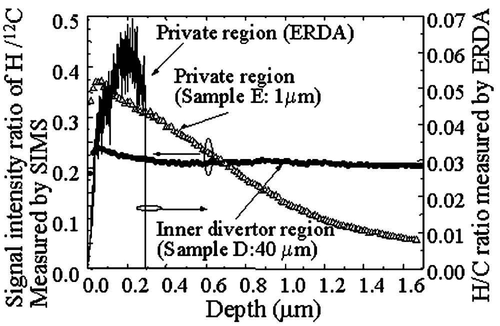

Fig. 4. Depth profiles of SIMS represented by signal intensity ratio of H/$^{12}$C for inner divertor tile (sample D) and the private flux region tile (sample E), and that of ERDA represented by H/C ratio of the private region tile.

measured by TDS was ~0.015. Thus that the surface hydrogen retention for the tiles which was not covered by the thick re-deposited layers, was rather high but hydrogen did not diffuse into deep and decreased rapidly with the depth. On the other hand, the hydrogen concentration in the thick co-deposited layers except those subjected to higher temperatures is nearly constant throughout the layers. Still H/C for the re-deposited layers is much lower than the hydrogen saturated amorphous hydrogenated films. This indicates that hydrogen was mostly retained near surface regions and did not diffuse in the deeper region during hydrogen discharges and re-deposited layers grew shot by shot with nearly constant hydrogen concentration. The very small but constant H/C in the thick re-deposited layers also suggests that hydrogen once retained in the re-deposited layers might be released by temperature increase due to their poor thermal contact with the substrate. This motivates us to observe Raman spectra of the re-deposited layers.

The Raman spectra of sample A, B and C are shown in Fig. 5. That of the substrate (ETP-10) is also shown in the figure for comparison. The Raman spectra showed two peak structure at around 1580 cm$^{-1}$ (G-band) and 1360 cm$^{-1}$ (D-band), respectively assigned crystalline like structure and defective structure of graphite. In the spectra, however no clear peak appeared at around 1520 cm$^{-1}$, which is attributed to hydrogenated amorphous carbon [16]. This is another confirmation that H/C in the re-deposited layers was very small. Both ratios of the peak intensity ($I_{\text{D-band}}/I_{\text{G-band}}$) and a full width at half maximum (FWHM) of G-band peak of the re-deposited layer for three samples, which were 0.66–0.74, 84–100 cm$^{-1}$, respectively, are much larger than those of the substrate ($I_{\text{D-band}}/I_{\text{G-band}} = 0.27$, FWHM = 21 cm$^{-1}$), indicating the re-deposited layers was not crystallized. Still both ratios were much larger than fully amorphized carbon [17], and the re-deposited layers were slightly graphitized [12].

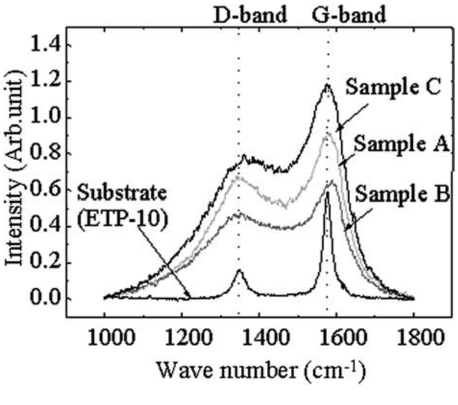

Fig. 5. Raman spectra of sample A, B, C and the substrate (ETP-10).

Actually, Gotoh et al. [12] have found graphitic layers parallel to the substrate on the top of the re-deposited layers, and in between the graphitic layers and the substrate, porous deposited layers remained. Since such graphitic layers could be grown at very high temperatures, we can concluded that poor thermal contact of the re-deposited layer to the substrate resulted the temperature elevation of the re-deposited layers under high plasma heat load. Consequently hydrogen once retained in the deposited layers was mostly released.

## 4. Summaries and conclusion

Hydrogen retention in the graphite tile used as open divertor tiles in JT-60 for hydrogen discharge period was investigated. Most of hydrogen was retained in the re-deposited layers for both inner and outer divertor tiles and the hydrogen concentration in the re-deposited layers, which is very likely uniform throughout their thickness, is as low as ~0.015. This is much smaller than the saturated hydrogen concentration in ion implanted carbon and hydrogenated amorphous carbon. Such low retention is most likely owing to the temperature increase above the base temperature of 573 K during the discharges. The temperature increase could be enhanced by the poor thermal contact of the re-deposited layers to the substrate.

Just beneath the re-deposited layers, appreciable amount of hydrogen was retained but the concentration decreased exponentially with the depth, which also supports the temperature increase of the re-deposited layers. Accordingly the total retention in the substrate was much less than that in the re-deposited layers, which was confirmed by the very small retention in the sample of which re-deposited layers were completely removed.

The present results indicates that tritium retention in the re-deposited carbon layers in the ITER first wall could be reduced significantly comparing the current estimation based on the JET results, if the surface temperature could be substantially higher than 573 K.

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