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Ion beam analysis of H and D retention in the near surface layers of JT-60U plasma facing wall tiles

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Abstract

H and D retention characteristics in the near top surface of the JT-60U plasma facing carbon tiles were investigated by means of elastic recoil detection analysis. From the result, H was dominantly retained within 1 μm depth because of the H discharges for the wall cleaning carried out before the removal of sample tiles. (H + D)/C ratios were small: it was ~0.07 at a maximum in the divertor samples except for the deposition on the dome outer wing tile which was ~0.13, and it was ~0.15 at a maximum in the first wall samples. The hydrogen concentration in the redeposited carbon layers is strongly influenced by the temperature. Consequently, (H + D)/C ratios at the observed low level are a result of the operation of JT-60U at wall temperatures of 300 °C or higher.

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Keywords: Hydrogen inventory; JT-60U; Divertor; First wall; Erosion and deposition; Carbon-based materials

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1. Introduction

The hydrogen retention characteristic in plasma facing materials (PFM) is a critical issue for the accumulation and removal of in-vessel tritium in a next step fusion reactor. If carbon-based materials are used as PFM, a major concern is tritium incorporation in the redeposited carbon layers [1,2]. In the present design of ITER, carbon fibre composite (CFC) will be used at the divertor target tiles [3] where the highest heat load is expected, because of its advantages as a high heat flux component [4]. Thus, the erosion of CFC and subsequent re- and co-depositions with tritium are critical issues to estimate the tritium inventory build-up in ITER.

Major tokamaks using carbon-based PFM such as JET [5], TFTR [6] and Tore-Supra [7] have shown the formation of the co-deposition of hydrogen with carbon having a H/C ratio ranging from 0.4 to 1.0. Those results led to a pessimistic estimation of tritium-uptake e.g. ITER in-vessel tritium

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inventory could reach 350 g, which is the in-vessel limit imposed by radiation safety, within several tens of discharges. On the other hand, JT-60U, which has used carbon-based materials as primary PFM, shows less carbon deposition and hydrogen retention. A result obtained from thermal desorption spectroscopy indicated that hydrogen retention in the redeposited layer for the inner divertor was less than 0.05 [8]. From this point of view, the hydrogen retention characteristic in PFM has to be investigated carefully to get more reliable extrapolation for the in-vessel tritium inventory in ITER.

JT-60U was usually operated with deuterium (DD discharges) and occasionally employed hydrogen discharges (HH discharges) before the vacuum vessel opening for removal of tritium. Thus, studies of both H and D retention properties in JT-60U will give important information to understand the fuel retention mechanism in DT discharge machines. Depth profiling of H and D has been done by secondary ion mass spectroscopy (SIMS) [9]. However, SIMS hardly gives quantitative results. As far as D retention, depth profiling by Nuclear Reaction Analysis (NRA) was successfully done for investigating D retention [10,11], but not yet for H. In this study, hydrogen (H) and deuterium (D) absolute depth profiling in the near surface of JT-60U tiles was measured by means of elastic recoil detection (ERD) analysis in order to understand the hydrogen retention characteristics in JT-60U plasma facing wall.

2. Experimental procedure

Fig. 1 shows a schematic cross-sectional view of the JT-60U vacuum vessel and the W-shaped divertor with the inner pumping geometry together with sample locations. The divertor samples were CFC (PCC-2S for dome top and CX-2002U for divertor target) tiles used from June 1997 to October 1998. During this period, about 3600 discharges (D plasma with D neutral beam injection (NBI)) were performed. The first wall samples were graphite (IG430U) tiles used from June 1991 to October 1999. More than 15000 shots of DD discharges were performed in this period. In both cases, HH discharge operation for the reduction of accumulated tritium in the vessel was done before the tile removal: about 300 discharges in October 1998 and about 350 discharges in October 1999. Small samples (about $10 \times 10 \times 2$ mm) were cut from the tiles at Japan atomic energy agency (JAEA) and transported to the Max-Planck Institut für Plasmaphysik (IPP) to be analyzed.

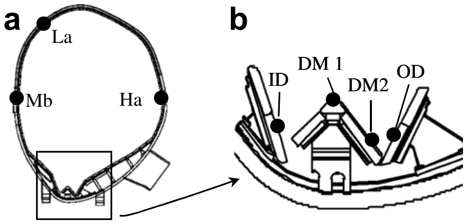

Fig. 1. Cross-sectional view of: (a) the JT-60U vacuum vessel and (b) the W-shaped divertor together with sample locations.

Quantitative depth profiles of H and D were obtained by means of ERD analysis at IPP. 3.0 MeV ^{4}He ions obtained from a 3 MV tandem accelerator with an incidence angle of 75° from normal incidence were used. Some of ^{4}He ions penetrate into subsurface layers and make elastic collisions with H or D atoms in the sample. The recoiled ions escaping from the sample surface are detected. The energy of the recoiled ions depends on the target mass (H or D) and its initial depth in the sample. In this experiment, backscattered He ions were stopped in a 5 μm Ni foil. Therefore, it is possible to detect recoiled H and D separately and estimate each depth profile by measuring energy of respective recoiled ions. Fig. 2 shows an example of recoil energy spectrum obtained from one of the samples, which shows both H and D peaks clearly. The quantitative depth profiles of H and D were calculated from the ERD spectra by using the SIMNRA program [12]. Although the projected range of 3 MeV ^{4}He ions in graphite with an incidence angle of 75° is ∼3 μm, taking the energy of recoiled ions

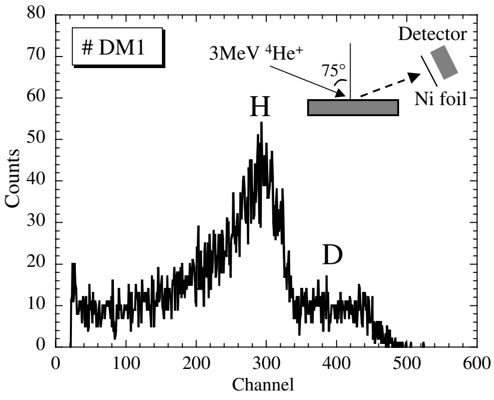

Fig. 2. An example of a recoil energy spectrum (obtained from DM1 sample). The spectrum consists of H and D peaks.

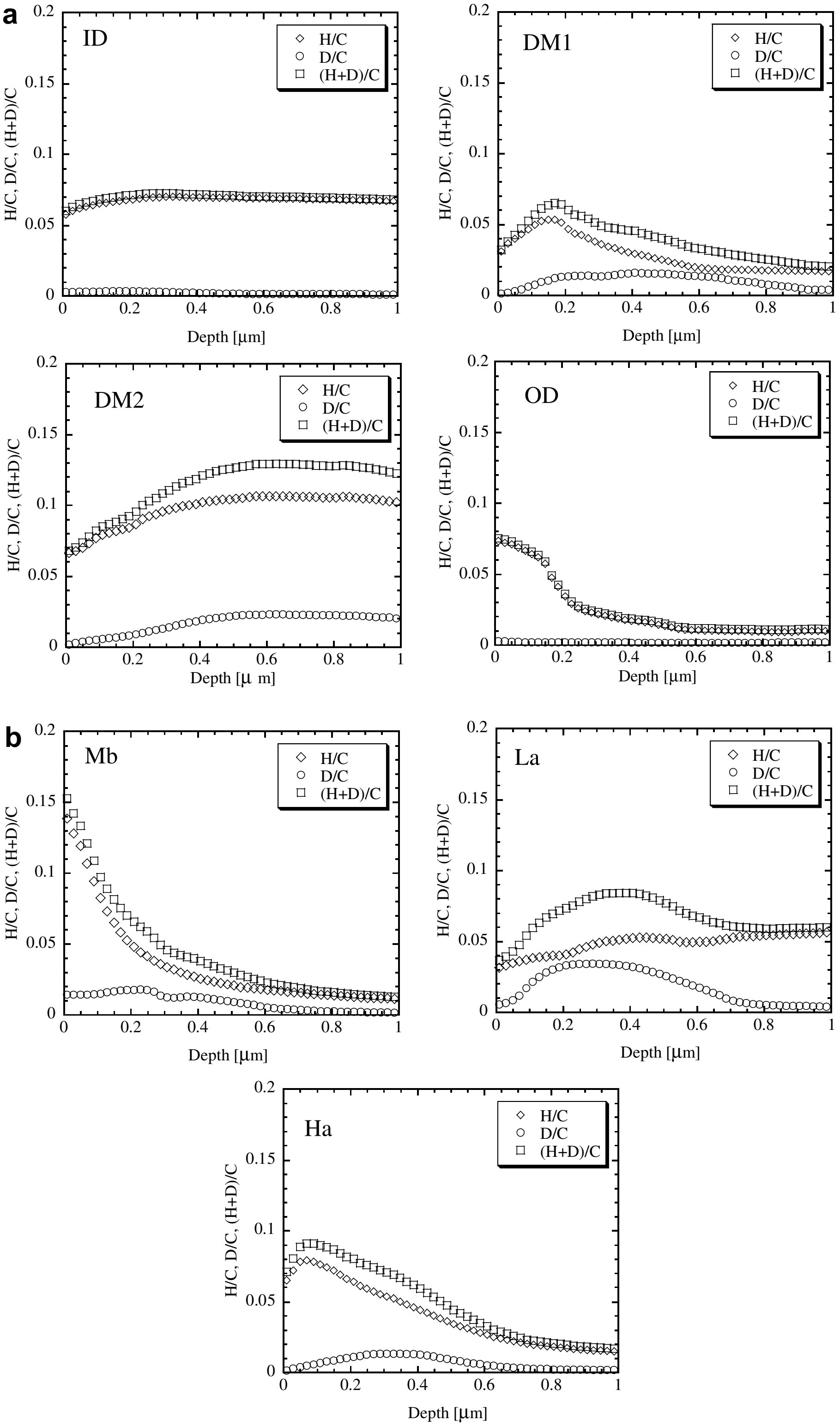

Fig. 3. H/C, D/C and (H + D)/C depth profiles obtained from: (a) divertor samples and (b) first wall samples. In divertor tiles, ID and DM2 was covered by the a redeposited layers.

into account, the depth information obtained by this technique is within approximately 1.0 μm. In the calculation, the density of carbon tiles was assumed to be 1.8 g/cm^3, and the surface roughness was not taken into account. From the surface observation by SEM, the redeposited layers in JT-60U were not flaky but showed columnar/lamellar structures with small roughness [13].

3. Results

Fig. 3(a) shows the H and D depth profiles obtained from the divertor samples. The samples: DM1 (from the dome top tile) and OD (from the outer target tile) had almost no deposition on their surfaces, while thick redeposited layers appeared on ID (from the inner target tile) and DM2 (from the dome outer wing tile). As seen in the figure, within 1.0 μm from the surface, H is dominantly retained and D retention is small. Especially, more or less no D is retained in the OD sample.

The profile for the samples from areas without deposition or erosion dominated areas show that the $(H + D)/C$ ratio was the highest at the top surface and decreased with depth. On the other hand, the deposited samples show a nearly constant $(H + D)/C$ ratio over the analyzed depth. These depth profiles are almost consistent with the results obtained by SIMS [9,14,15]. $(H + D)/C$ ratios remained below 0.07 for all samples except for the DM2 of which $(H + D)/C$ ratio reaches 0.12.

Fig. 3(b) shows results for the first wall samples. The $H + D$ contents in these samples are a little higher than those of the divertor samples. The maximum $(H + D)/C$ ratio in the first wall tiles was ranging from 0.08 to 0.16. The samples: Mb and Ha show similar profiles with those for the non-deposited divertor samples, i.e. $H + D$ content was the highest at the top surface and decreased with depth. La shows a different profile of which peak appeared at 0.4 μm in depth and remained at 0.06 in $(H + D)/C$ ratio even in deeper region.

4. Discussion

4.1. Hydrogen uptake from the moisture in the atmosphere

Although the tiles were stored in evacuated plastic bags, they were certainly exposed to air. Hence, the tile surface could uptake hydrogen from the moisture in the atmosphere. The ERD analysis is generally the most sensitive for detecting such surface hydrogen. Nevertheless, $(H + D)/C$ remained small at the surface and rather constant in depth. In addition, SIMS measurements showed that the oxygen content was also small. Furthermore, due to smaller hydrogen retention and subjection of the tiles to higher temperatures, the structure of the redeposited layers on the tiles was somewhat graphitized. Accordingly they were hard to oxidize, which was confirmed by combustion made for the determination of the tritium content [16]. Hence, we can conclude that the present depth profiles of hydrogen were not influenced by hydrogen uptake from the moisture in the atmosphere.

4.2. H, D retention characteristics in eroded samples

Since the diffusion of H or D in the graphite during a single discharge is not important in this temperature region (<1000 K), the initial depth profile should be determined by the range of incident hydrogen ions composed of those from boundary plasma and charge exchange neutrals. In addition, some of D injected by NBI could escape from the core plasma due to ripple-loss and/or orbital loss processes without fully loosing its initial energy and can be implanted directly into a certain depth of the plasma facing tiles. The simulation using OFMC code has shown that the escaping energetic ion flux is high near the mid-plane and the divertor dome region, with an energy of ~50 keV [17]. For 50 keV D, the range in carbon is not more than ~0.7 μm. For the eroded samples (DM1, OD, and first wall samples), surface recession due to the erosion would make depth profile shallower than the implanted depth. Hence, we have to consider long time hydrogen penetration as follows. Since the saturation concentration of hydrogen would decrease with increasing the surface temperature, and the incident hydrogen flux from boundary plasma is extremely high on the eroded area, surface hydrogen could easily be saturated during a discharge. Further injection would simply increase the thickness of the hydrogen saturated layers. Thus the thickness of the hydrogen saturated layers in the eroded area could easily exceed the range of the incident hydrogen particles.

Fig. 4 shows the amount of $H + D$ of those samples retained within 1 μm depth. It is found that H is dominantly retained at the top surface. This is caused by the H discharges for wall cleaning

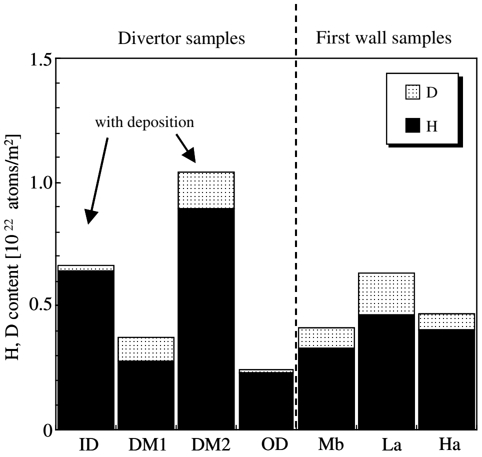

Fig. 4. The total amount of H + D retained within 1 μm depth.

(tritium removal) carried out after regular D discharge phase, i.e. D retained in the near surface was replace by H due to large isotope exchange effect. This result indicates that the isotopic exchange is effective for removal of the tritium retained in the top surface of plasma facing area. As found in (H + D)/C profiles, (H + D)/C ratios were ~0.07 in the DM1, OD, 0.08–0.15 in the first wall samples at maximum. The averages from top to 1 μm were ~0.02 in the OD, and around 0.04 in the DM1 and first wall samples. The primary factor to determine hydrogen concentration in the redeposited carbon layers must be their temperature during a discharge. The hydrogen saturation level in carbon is about 1–0.4 below around 500 K, while above 500 K, it decreases exponentially with increasing temperature [18]. In this respect, it is reasonable that the sample OD showed the lowest (H + D)/C ratio because it was taken from the outer target tile where the temperature rose easily over 1000 K during a discharge [19]. DM1 and first wall samples showed a little higher (H + D)/C ratio than OD, however, it was still a lower level, because JT-60U operates with 574 K baking temperature.

4.3. H, D retention characteristics in deposition samples

The depth profiles from the deposition-dominated samples (ID and DM2) were relatively constant, and H was dominant as well. In JT-60U, the deposition rate at the divertor during the DD discharge phase was estimated to be 6 nm/s [17]. Although the input power during the HH discharge phase was lower than during the DD discharge phase, the deposition growth could be more than 1 μm during the HH discharge phase. Therefore, the depth profile is attributed to continuous growth of the redeposited layer with constant H/C ratio during the H discharge phase.

As mentioned above, hydrogen retention in carbon is strongly influenced by the temperature. The (H + D)/C ratio in the redeposited layer on the sample ID was ~0.07. To attain such low concentration, the required temperature of the deposited layer must be higher than 800 K. This is reasonable again because the maximum temperature measured by thermocouples installed in the inner divertor tiles at a depth of 6 mm was about 750 K, thus, the temperature of the top surface should be more than 800 K. From TDS analysis of the thick redeposited layers on the inner divertor tiles, the (H + D)/C ratio was determined to be ~0.03 [8], which was nearly half of the present result. As already mentioned, we can neglect the extra hydrogen uptake from the moisture in the atmosphere. Plasma heat load during the HH discharges was less than half, and the temperature increase during the HH discharge would be less than that for the DD discharges. This indicates that the H/C ratio in the carbon deposition layers was higher than the D/C ratio in the deposition layers formed during the DD discharges. The sample DM2 showed somewhat higher (H + D)/C ratio (~0.13) than ID. DM2 was from the bottom of the dome outer wing tile situated at the private flux region. This position is regarded as a quasi-shadowed area where the temperature elevation is smaller. From the measurement by the thermocouples, the maximum temperature was about 650 K [19]. This temperature difference between ID and DM2 could cause the difference in (H + D)/C.

5. Conclusion

The H and D retention characteristics in the near top surface of the JT-60U plasma facing tiles were investigated by means of an ERD analysis. H was dominantly retained within 1 μm depth, which is due to H discharges for wall cleaning carried out before the removal of sample tiles. This kind of wall cleaning using the hydrogen exchange is effective for removal of the tritium retained in the top surface of plasma facing areas without carbon deposition. At the deposited area, H depth profile was relatively constant, indicating a continuous growth of the redeposited layer during the H discharge phase.

(H + D)/C ratios were not very much high: it was ~0.07 at a maximum in the divertor samples except for the deposition on the dome outer wing tile which was ~0.13, and it was ~0.15 at a maximum in the first wall samples. JT-60U operates with 300 °C baking temperature and the temperature of the divertor tiles during a discharge can rise to much higher values. Accordingly smaller (H + D)/C ratios were attained in JT-60U compared to those in other tokamaks. From this result, tritium incorporation in carbon at plasma facing area is not so large in ITER as expected from previous experiments. Hence its retention at plasma shadowed areas is more important for the mitigation of the in-vessel tritium inventory.

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