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Depth profile and retention of hydrogen isotopes in graphite tiles used in the W-shaped divertor of JT-60U

Y. Hirohata [a,*], Y. Oya [b], H. Yoshida [a], Y. Morimoto [c], T. Arai [d], K. Kizu [d], J. Yagyu [d], K. Masaki [d], Y. Gotoh [d], K. Okuno [c], N. Miya [d], T. Hino [a], S. Tanaka [b], T. Tanabe [e]

[a] *Department of Nuclear Engineering, Faculty of Engineering, Hokkaido University, Kita-13, Nishi-8, Kita-ku, Sapporo 060-8628, Japan*
[b] *The University of Tokyo, Yayoi, Bunkyo-ku, Tokyo 113-0032, Japan*
[c] *Shizuoka University, Ohya, Shizuoka 422-8529, Japan*
[d] *Japan Atomic Energy Research Institute, Naka-gun, Ibaraki 311-0913, Japan*
[e] *Nagoya University, Nagoya 464-8603, Japan*

Abstract

Depth profiles of deuterium and hydrogen retained in graphite tiles placed in the divertor region of JT-60U were investigated by secondary ion mass spectroscopy. The deuterium in the near surface of all graphite tiles was replaced by hydrogen due to exposure to hydrogen plasmas at the final stage operations. The depth profiles of the $H/^{12}C$ and $D/^{12}C$ signal intensity ratios varied not only with the location of the tile but also with the existence of redeposited layers. The integrated intensity of $(H+D)/^{12}C$ within the depth of 1.7 μm for the deposition dominated tiles was much smaller than that for the erosion dominated tiles. This suggests that thermal contact of the redeposited layer might be too poor to remove the heat loads from plasma to the substrate resulting in the surface temperature of the redeposited layer becoming much higher than that of the substrate.

1. Introduction

It is a very important issue to evaluate retention properties of tritium and deuterium in in-vessel components of current devices from a viewpoint of safety for the next fusion reactor [1–4]. There have been many investigations for the retention properties of hydrogen isotopes in in-vessel components of current devices. Recently, we have been studying the retention of hydrogen isotopes (H,D,T) in plasma facing graphite tiles used in the W-shaped divertor of JT-60U with inner side pumping [3,5–9] and with both side pumping [10,11] by using various method. We found that most of tritium was implanted to depths in the micrometer range, tritium distribution was independent of carbon deposition profiles, while H and D distributions seem to be very much influenced by the carbon deposition as well as the surface temperature of the tiles. It is known that, for JT-60U, erosion is dominant in the outer divertor region and redeposition in the inner divertor region [8,11].

In this study, the depth profiles of hydrogen and deuterium retained in the graphite tiles with and without a redeposited layer used in the W-shaped divertor of JT-60U were investigated by secondary ion mass spectroscopy (SIMS). All tiles were exposed to deuterium and hydrogen discharges from June 1997 to October 1998.

* Corresponding author. Tel./fax: +81-11 706 7108.
E-mail address: hirohata@qe.eng.hokudai.ac.jp (Y. Hirohata).

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2. Experiments

Fig. 1 shows the cross-sectional view of the JT-60U W-shaped divertor. The W-shaped divertor was pumped from the inner slot by cryopumps. All tiles received 3600 shots of deuterium discharges during two discharge campaigns. After each of two-deuterium discharge periods, a hydrogen discharge experiment, of totally 700 shots, was carried out for reduction of tritium before air ventilation. Boronization was conducted twice during these campaigns. Thirteen thermocouples were embeded in the divertor tiles 6 mm beneath the tile surface, to monitor the tile temperatures during the discharge experiments. Compared to full deuterium discharges, the power loads of hydrogen discharges were lower. Hence the maximum tile temperatures were more than 50 K lower for the H discharges than those for the D discharges.

Samples (5 mm × 10 mm × 1 mm) for SIMS measurements were cut from the tile surface area just above the thermocouples; four samples (ID1–ID4) from the inner divertor tiles, three samples (OD1–OD3) from the outer divertor tiles, four samples from the baffle plates (BP1, BP11, BP2, BP3), and nine samples (DM1–DM9) from the dome units were prepared (see Fig. 1). The outer divertor tiles were mostly eroded (up to 20 μm) while the inner divertor tiles were covered by deposited layers with maximum thickness of 60 μm [8]. On the dome top tile no significant deposition or erosion was observed. For ID1, an additional measurement after removing some surface deposited layers was also conducted. Depth profiles of hydrogen, deuterium, boron and oxygen retained in graphite tiles were analyzed by SIMS using Cesium ions (Cs^+) as a probing beam. The sputtering rate by the probing beam was approximately 1 μm/h. The beam of 32 μm in diameter was rastered over 400 × 400 μm^2 area. In order to eliminate the effect of crater edges, the signal of the secondary ion was collected only from the center of the rastered area. The negative secondary ion intensities of hydrogen isotopes, boron, and oxygen were normalized by that of ^{12}C for comparison. In order to compare the concentration of retained hydrogen (deuterium) in the tile, time-integrated intensity ratios of $H/^{12}C$ and $D/^{12}C$ were obtained within the sputtering time of 6000 s, corresponding to the depth of 1.7 μm. These values are referred named as $\sum H/^{12}C$ and $\sum D/^{12}C$.

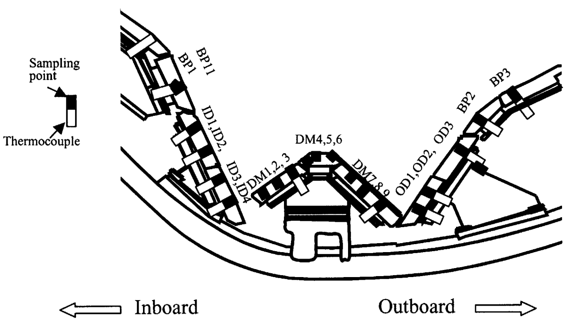

Fig. 1. Cross-sectional view of the divertor region in JT-60U with the W-shaped divertor (inner slot pumping system).

3. Results

Fig. 2 compares a typical depth profiles of $H/^{12}C$, $D/^{12}C$ and $(H + D)/^{12}C$ signal intensity ratios for (a) the

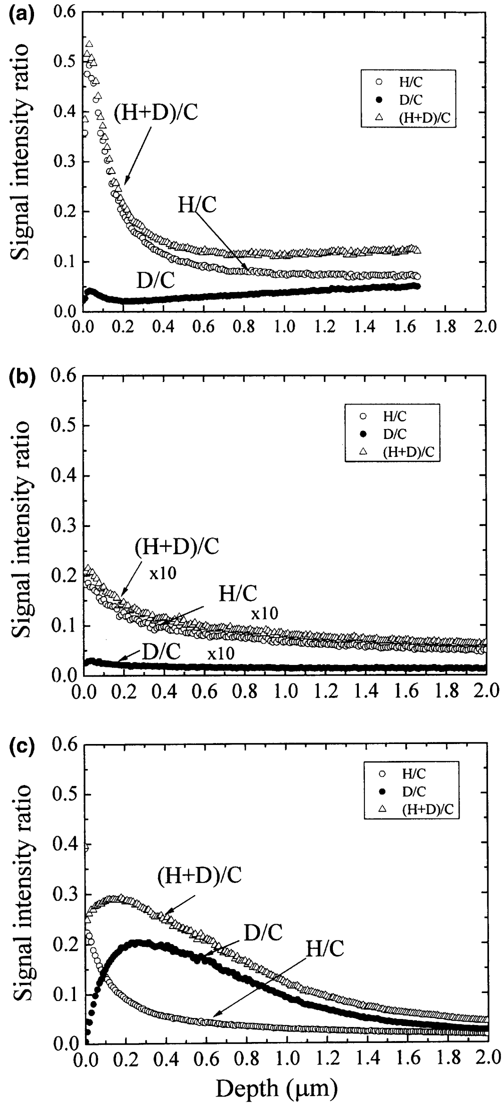

Fig. 2. Typical depth profiles of signal intensity ratios of $H/^{12}C$, $D/^{12}C$ and $(H + D)/^{12}C$ for (a) the erosion dominated region of the outer divertor tile (OD3), (b) the deposition dominated region of the inner divertor region (ID3) and (c) the plasma facing dome top tile (DM5).

erosion dominated region of the outer divertor tile (OD3), (b) deposition dominated region of the inner divertor tiles (ID3) and (c) the plasma facing dome top tile (DM5). On the outer divertor, no remarkable redeposition layers were found; erosion with a maximum depth of about 20 μm was found [8]. One can see that the H/^{12}C ratio near the surface was approximately one order of magnitude larger than that of D/^{12}C indicating effective replacement of deuterium retained in near surface regions. In addition, total retention or H retention (because hydrogen retention dominates in most cases) clearly depends on the deuterium retention, i.e. higher the D retention, higher the H retention. Fig. 3 compares (a) H and D retention within 1.7 μm depth, (b) tritium profiles determined by imaging plate technique [9] and (c) erosion deposition profiles of divertor regions [8]. The deuterium retention in the divertor region is rather consistent with tritium profiles; lower in both (inner and outer) divertor and higher in dome and baffle regions. It is very important to note that deuterium and hydrogen retentions were quite low in deposited areas, which is completely different from the observations in JET and other low temperature operating machines, where hydrogen isotope were mostly codeposited with carbon [12–14]. Separate quantitative measurements of hydrogen isotope retention by nuclear recoil detection technique by Hayashi et al. [15] for the divertor tiles used in JT-60U and TDS by Shibahara et al. [16] for those used in JT-60 and JT-60U have indicated that D/C or H/C in the deposited layers is below 0.07.

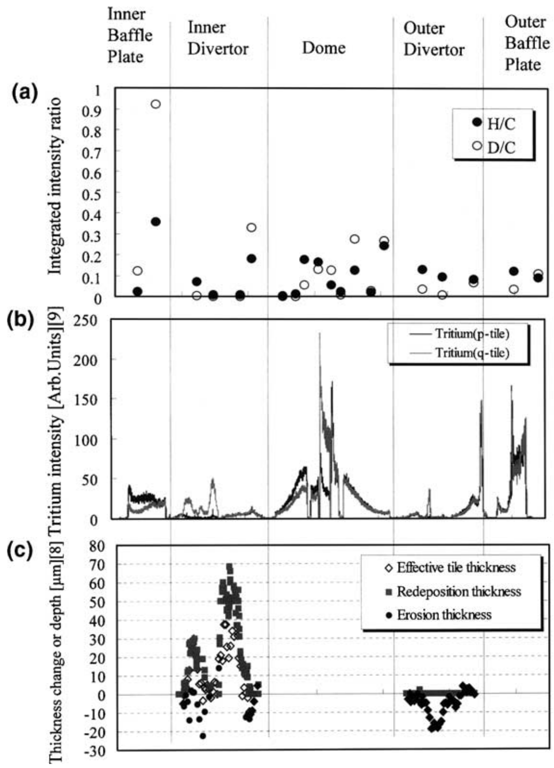

Fig. 3. Retention of hydrogen (●) and deuterium (○) within 1.7 μm measured by SIMS (a), tritium profiles measured by imaging plate technique [9] (b) and the deposition or erosion profile measured by SEM and a profile meter [8] (c) as a function of the tile position. The p and q tiles indicated in Fig. 3(b) mean the row number of the tiles in JT-60U. The p-tile is the same as that measured by SIMS, and is adjacent tile to the p-tile.

The deuterium retention characteristic in the dome top tile (Fig. 2) and baffle plates were different from that in the divertor, i.e. deuterium retention is high and penetrated very deep in both tiles, particular BP1 (see Fig. 3) shows the highest deuterium retention. However, no appreciable deposition was observed in the dome tile and only small amount of deposition in the baffle plate. On the other hand, the inner divertor tile was covered by thick redeposited layers but less deuterium was retained. All these indicate that deuterium retention is not necessarily correlated to the deposition. Probably because the temperature of deposited layer in the inner divertor tile was high enough to release large part of the retained deuterium in the deposited layers as discussed below.

4. Discussion

Although hydrogen discharges performed for the reduction of tritium makes hydrogen isotope behavior very complex, we have found characteristics of hydrogen retention in JT-60U. The most significant observation is that deuterium retention in deposited area on the inner divertor, i.e. ID1–ID3 samples, was quite low, less than those in the erosion dominated outer divertor. To confirm this, we have made additional depth profile measurements for the sample from the inner divertor tile (ID1), of which some surface deposited layers were removed mechanically and the result is shown in Fig. 4. Surprisingly both H and D retentions are higher after removing the surface layers. In the profile measurements, Gotoh et al. have found graphitic layers parallel to the substrate on the top of the deposited layers, and in between the layers and the substrate porous deposited layers remained [8]. Since such graphitic layers could be grown only by subjecting very high heat load, we can conclude that very poor thermal conductance of the redeposited layer or poor contact with the substrate forced temperature increases of the deposited surface under high plasma heat load. Consequently deuterium once retained in the deposited layers was mostly released. Once such well graphitized layers were grown on the top surface, porous deposited layers remained on the substrate, and deuterium retention must be very small for succeeding discharges.

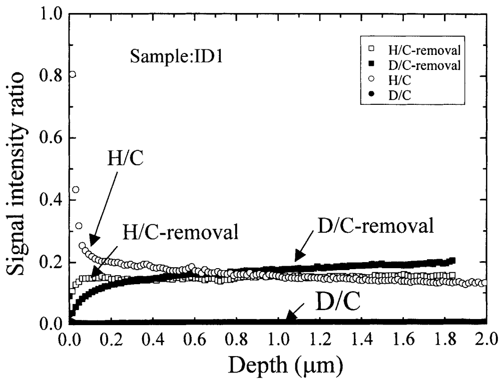

Fig. 4. Depth profiles of signal intensity ratio of $H/^{12}C$, $D/^{12}C$ for the ID1 before and after the removal of the deposition layer.

Deuterium retention in the outer divertor tiles are still low, as mentioned above, with D/C below 0.05. The most probable explanation for such low hydrogen retention is the temperature increase of the tiles. In JT-60U, the vacuum vessel was usually kept at 573 K and divertor area was easily raised above 673 K by the plasma heat load during full deuterium discharges. Sometimes surface temperatures of the outer divertor were above 1273 K. Hence deuterium retained could be released. The consistency of the tritium profiles with the deuterium profile in the divertor regions could also be attributed to a temperature increase.

Additionally, one should note that the surface of the outer divertor was always eroded due to chemical sputtering, which could make the deuterium profiled sharper than that of the inner divertor, as actually observed in Fig. 2. In Fig. 2(a) one can see a very sharp hydrogen profile decay from the surface, compared to the flat deuterium retained profile.

The question still remains of how hydrogen discharge replaced deuterium in the tiles. One can see in Fig. 3(a) hydrogen retention within 1.7 μm from the surface are mostly parallel to that of deuterium and hydrogen profile was peaked on the top surface (Fig. 2). Thus hydrogen replaced deuterium on the near surface very easily during the discharges, but cannot easily penetrate deep, i.e. the tile temperature was not high enough to allow hydrogen to diffuse in. All these results indicate that the deep deuterium profile keeps their original retention characteristics.

It should be also noted that hydrogen retention seems very much parallel to that of deuterium in Fig. 3(a), the higher deuterium retention is the higher hydrogen retention, but the hydrogen retention seems saturated at a certain level. This again shows hydrogen can easily exchange with deuterium as far as deuterium remains, irrespective of the characteristics of the tile surfaces. But once deuterium was mostly replaced, hydrogen cannot penetrate deeper.

The higher deuterium retention and deeper penetration of dome area tiles are a little puzzling. Similar results were observed in the dome unit tiles with both side pumping of JT-60U [11]. Their temperature must be lower than those for the divertor tiles and the incoming deuterium flux during discharge must be lower also. Since these tiles were facing the main plasma more directly than the divertor tiles, high-energy neutrals, which could be implanted deep and accumulated, might play a role that is not understood at present.

5. Conclusions

The depth profiles of deuterium and hydrogen in tiles placed in the divertor region of JT-60U were investigated by SIMS. The depth profiles of hydrogen isotopes were quite different for erosion dominated outer divertor tiles and deposition dominated inner divertor tiles. In particular, retention of H and D in the deposited surface layers on the inner divertor tile was quite small, even smaller than that in the erosion dominated outer divertor tile. This is quite different from the deuterium retention characteristics of JET or other low temperature operating machines. Most probably, the poor adhesion or porous nature of the deposited layer inhibited plasma heat load conduction to the substrate, resulting in temperature increases of the surface deposited layers. Actually, the depth profiles and retention of hydrogen isotopes for both divertors are inversely correlated to the heat load, i.e. frequency of the separatrix strike point hits. Such deuterium retention profiles in the divertor region are found to be rather consistent with tritium profiles in the divertor tiles.

Hydrogen discharges employed for reduction of tritium seems to work well to exchange most of deuterium retained in near surface regions, irrespective of tile positions. However, deuterium deeply retained in the tiles was not effectively replaced by hydrogen discharges, particularly for the dome top tile for which high D retention and deep penetration were not well explained at this stage.

The present results indicate that tritium retention in redeposited carbon layers in the ITER first wall could be significantly smaller than the present estimation based on JET results, if the surface temperature could be above 673 K.

References

[1] G. Federici et al., J. Nucl. Mater. 266–269 (1999) 14.
[2] J.P. Coad et al., J. Nucl. Mater. 290–293 (2001) 224.
[3] T. Tanabe et al., J. Nucl. Mater. 313–316 (2003) 478.
[4] C. Stan-Sion et al., J. Nucl. Mater. 290–293 (2001) 491.

[5] Y. Hirohata et al., Phys. Scr. T 103 (2003) 15.
[6] Y. Oya et al., J. Nucl. Mater. 313–316 (2003) 209.
[7] K. Masaki et al., J. Nucl. Mater. 313–316 (2003) 514.
[8] Y. Gotoh et al., J. Nucl. Mater. 313–316 (2003) 370.
[9] K. Sugiyama et al., Phys. Scr. T 103 (2003) 56.
[10] Y. Oya et al., Phys. Scr. T 108 (2004) 57.
[11] N. Miya, these Proceedings, doi:10.1016/j.jnucmat.2004.04.219.
[12] R.-D. Penzhorn, J.P. Coad, et al., Fusion Eng. Des. 56&57 (2001) 105.
[13] M. Mayer, V. Philipps, et al., J. Nucl. Mater. 290–293 (2001) 381.
[14] V. Rohdle, M. Mayer, the ASDEX Upgarde Team, J. Nucl. Mater. 313–316 (2003) 337.
[15] T. Hayashi, et al., private communications.
[16] T. Shibahara, et al., private communications.