

Deuterium retention in tungsten coating on the CFC tiles exposed to JT-60U divertor plasmas

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Surface morphology and deuterium retention in vacuum plasma splayed (VPS) tungsten-coated tiles exposed to JT-60U divertor plasmas were investigated. It was found from surface observation that the surface of the tungsten coating would be eroded due to the sputtering although the total erosion was below the resolution of the cross-sectional observation. No significant blistering was observed on the surface of the tungsten coating. The deuterium retention in the tungsten-coated tiles was poloidally uniform at $\sim 10^{22}$ D/m². The deuterium depth profiles in the tungsten coating were similar to the carbon depth profiles in the coating. This suggests that the implanted deuterium during plasma discharge is trapped predominantly by the carbon impurity contained in the tungsten coating and deuterium retention depends on the amount of carbon impurities in the coatings.

Keywords: Tritium retention, Blistering, Tungsten, Vacuum plasma spraying, Divertor plasmas, JT-60U

1. Introduction

Fuel retention in a future deuterium-tritium (DT) fusion device is one of major concerns in terms of tritium (T) safety and economy. In ITER, an administrative limit of in-vessel T retention is assumed to be ~ 700 g. Carbon (C) based materials such as carbon fiber composites (CFC) have been widely used as armor materials [1]. However, C deposition layers containing hydrogen isotopes were found in the tokamak experimental devices, for example, the inner divertor and the outer dome wing in JT-60U [2, 3] and the louver of the cryogenic pumps in JET [4]. Therefore, if the C based materials are used as the armor materials in future DT fusion devices, the in-vessel T retention will reach the administrative limit.

Tungsten (W) based materials are promising candidates for plasma facing components (PFCs) for future DT fusion devices such as ITER and beyond due to the low T retention compared with C, low sputtering yield, and high melting temperature. In the current ITER design, W is used for the upper baffles and the divertor dome in the initial operation phase [5]. In the DT operation phase of ITER, a full W divertor with beryllium (Be) first wall and all-W machine will be planned to reduce in-vessel T retention [5]. It is reported that the T retention in the ITER vacuum vessel reaches the administrative limit within 100-300 shots of 400-s discharges at $Q = 10$ for the initial material choice of CFC/W/Be due to co-deposition of C

and T, and about 25000 shots for all-W machine (excluding neutron damage effects) [5]. However, this estimation was based on the basic experimental results obtained from plasma simulators and ion beam irradiation devices. In order to predict the in-vessel T retention in ITER, it is essential to investigate the mechanism of D retention in the tokamak devices from the results of basic experiments and to extrapolate the tokamak experimental data to ITER conditions.

Hydrogen isotope retention and surface modifications on W materials were investigated using several tokamak devices such as ASDEX-Upgrade [6, 7] and TEXTOR [8, 9]. However, the plasma confinement capability and stored energy, therefore, the particle flux toward the armor materials and the surface temperature of these devices may be different from those of ITER. Therefore, in order to more accurately estimate the in-vessel T retention in ITER, more studies are necessary for hydrogen isotope retention in W using large confinement tokamak devices.

In JT-60U, the equivalent DT fusion gain of 1.25 and the stored energy of 10.9 MJ have already been achieved [10, 11]. Hence, it is valuable to understand hydrogen isotope retention and its mechanism in W materials in high confinement plasmas which could be extrapolated to ITER conditions. Recently, W migration at the divertor region [12, 13] and the mechanism of W accumulation to the core plasmas [14] have been studied in JT-60U, but surface modifications and D retention have not been studied yet. In

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this work, surface modification, for example, erosion, deposition, and blistering and D retention in W-coated CFC tiles exposed to JT-60U divertor plasmas were investigated by means of surface analysis techniques. The mechanism of blister suppression and D retention in the W-coated tiles are discussed.

2. Experimental Setup

2.1 Tungsten-coated Tiles

Figure 1 (a) shows a cross-sectional view of a W-coated tile. Rhenium and tungsten (Re/W) multilayer with $\sim 15 \mu\text{m}$ thickness was deposited by physical vapor deposition (PVD) techniques on the CFC tiles. Tungsten with a thickness of about $50 \mu\text{m}$ was coated by vacuum plasma sputtering (VPS) techniques on the Re/W multilayer.

Figure 1 (b) shows surface morphology of the W coating. The disk-like W with a diameter of $10\text{--}40 \mu\text{m}$ was stacked on the tiles. Surface roughness, R_a , measured by a surface profilometer was $R_a = 5\text{--}7 \mu\text{m}$.

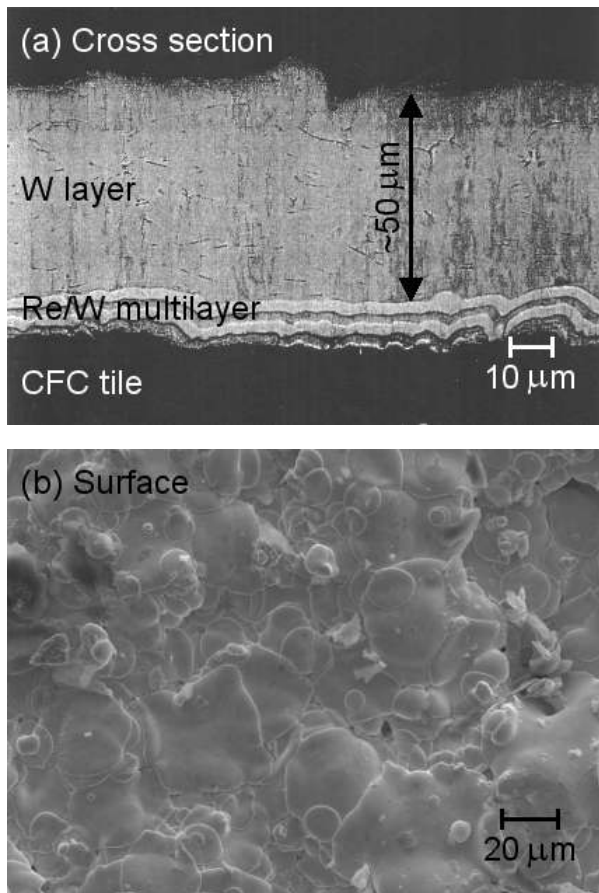


Fig.1 (a) A Back Scattered Electron (BSE) image of a polished cross section of the W-coated tile with a Re/W multilayer. (b) A Secondary Electron (SE) image of the W-coated tile surface. These images were obtained from virgin tiles.

In JT-60U, twelve W-coated CFC tiles were installed on the upper array of the outer divertor tiles before initiation on the experimental campaign 2003-2004 as shown in Fig. 2 (a). 978 discharges were performed with usual magnetic configurations (“Usual configuration” in Fig. 2(b)) and 25 discharges with a strike point positioned on the W-coated tile array (“W configuration” in Fig. 2(b)). In order to remove surface T, 105 shots of hydrogen discharges with hydrogen neutral beam injection (NBI) heating (HH discharge) were performed before air ventilation. After the experimental campaign, two W-coated tiles at the end of the tile array were taken out from the vacuum vessel for post-mortem analysis.

The incident ion fluences to the W-coated tiles during the campaign were measured by Langmuir probe array installed in the divertor tiles, which were installed in a different toroidal position from the W-coated tiles. The surface temperature of the W-coated tiles during the plasma discharges was calculated by finite element methods (FEM) based on the temperature measured by

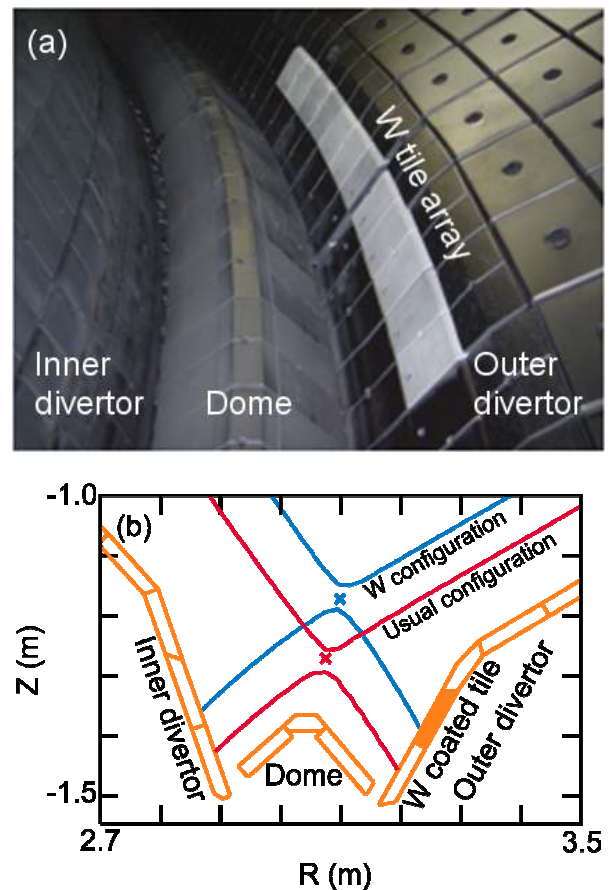


Fig.2 (a) Photograph of the W-coated tile array installed in the JT-60U divertor region. (b) Poloidal cross section of the divertor region with typical separatrix of a usual and a W experimental magnetic configuration. The filled tile represents the W-coated tile.

thermocouples embedded at ~ 6 mm below the tile surface.

2.2 Surface Analysis

The W-coated tiles were cut to a size of about $8 \times 8 \times 1$ mm³. Surface morphology and cross section were observed by a Scanning Electron Microscope (SEM). Deuterium retention in the samples was evaluated with Thermal Desorption Spectroscopy (TDS). The samples were heated from 300 to 1373 K at a heating rate of ~ 0.42 K/s and kept at 1373K for 10 min. The desorbed species were analyzed by a quadrupole mass spectrometer (QMS). The details of the TDS measurement system can be found in Ref. [15]. Depth profiles of D and main impurities, C and oxygen (O), were obtained by Secondary Ion Mass Spectroscopy (SIMS) with a primary Cs⁺ ion beam of 5 keV and 200 nA. The ion beam was raster-scanned over an area of 300×300 μm^2 and the secondary ions were detected from 3 % of the center of the sputtered area in order to suppress a crater edge effect. The surface roughness and the depth of the SIMS sputtered crater were calibrated by a surface profilometer.

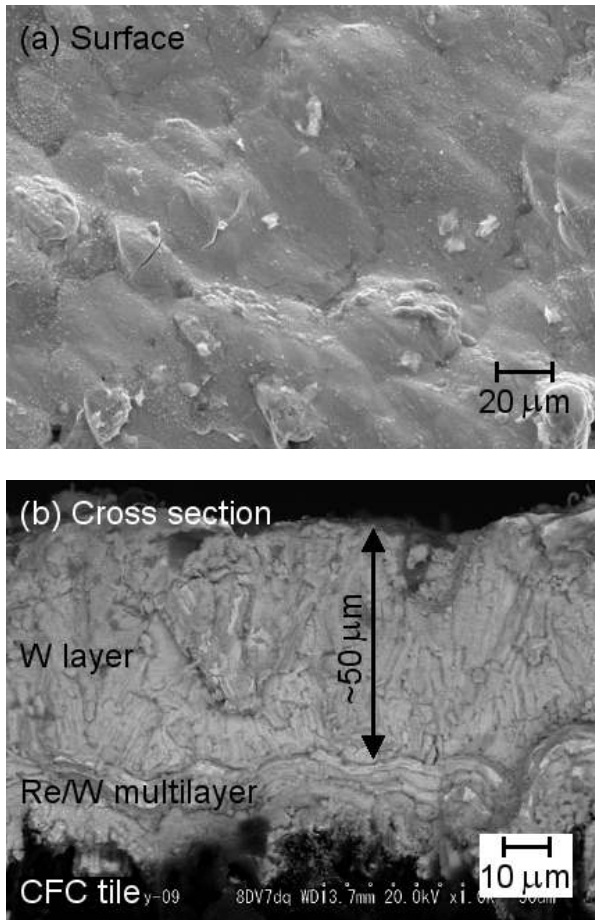


Fig.3 (a) A SE image and (b) a BSE image of the W-coated tiles after the exposure to JT-60U divertor plasmas.

3. Results

Figure 3 (a) shows surface morphology of the W-coated tiles after the plasma exposure. The surface roughness was decreased to $R_a = 3\text{--}5$ μm , which is lower than that of the virgin tile surface ($R_a = 5\text{--}7$ μm). However, no significant blistering, which was seen on polycrystalline W surface in many basic experiments, for example, Ref. [16], was observed.

Figure 3 (b) shows fracture cross section of the W-coated tile after the plasma exposure. The W coating still remained ~ 50 μm thickness and this indicates that the erosion of the W coating exposed to the plasmas, which had a total ion fluence of $10^{25}\text{--}10^{26}$ m⁻² in the campaign, was low.

Figure 4 (a) shows a poloidal distribution of retained D obtained from TDS measurement. The total incident ion fluence during the D discharge with D NBI heating (DD discharge) and the typical surface temperature at the end of

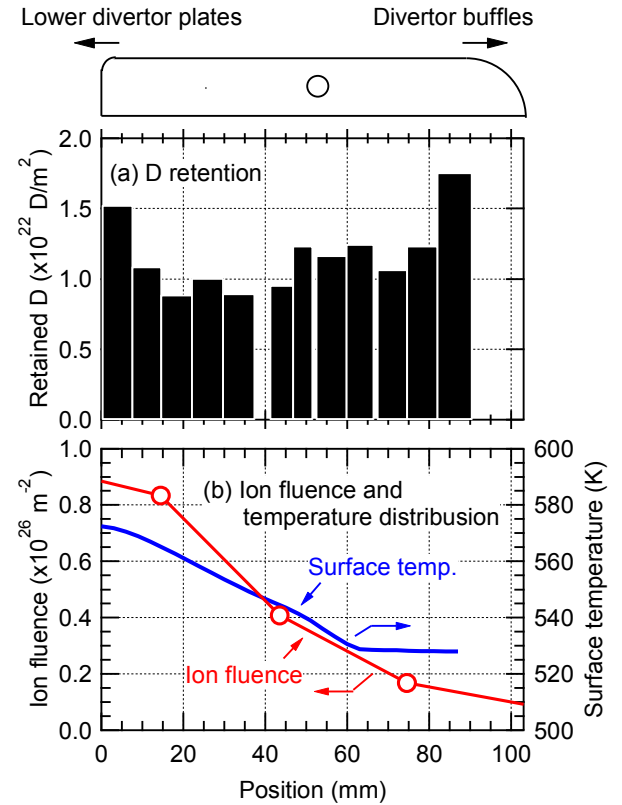


Fig.4 (a) Poloidal distribution of retained D in the W-coated tile obtained by the TDS measurement. (b) Total incident ion fluences during the D discharges with D NBI heating and typical surface temperature at the end of a plasma discharge was also shown with right axis. The origin of the horizontal axis is corresponding to the lower tile edge of the W-coated tile.

a plasma discharge are shown in Fig. 4 (b). The D retention within ~ 1 mm thickness was poloidally uniform with $\sim 10^{22}$ D/m², although the incident ion fluence and the surface temperature depended on the poloidal position of the W-coated tiles. Note that the surface temperature shown in Fig. 4 (b) was estimated on the assumption of no coating, i.e. plain CFC tiles. Hence, the surface temperature of the W-coated tiles are considered to be higher to some extent than that of the CFC because of the lower thermal conductivity of the W coating compared with CFC materials and thermal contact resistance among VPS-W, PVD-Re/W layer and CFC substrates [17].

4. Discussion

Before installation of the W-coated tiles into the JT-60U divertor regions, thermal heat load tests were carried out in the electron beam irradiation facility JEBIS [18]. The microstructure of the surface after testing for 230 cycles at ~ 7 MW/m² for ~ 5 s is shown in Fig. 5. Many spherical W particles, which did not melt during the plasma spraying process, are seen on the surface. Although the surface temperature rose to ~ 1500 K during the thermal heat load tests, no melt layer was observed. In contrast, the maximum thermal heat load on the W coating during the tokamak plasma discharge was ~ 6 MW/m² for ~ 7 s in the experimental campaign. Since the heat loads between the two experiments, the thermal heat load tests and the tokamak plasma exposure, were similar, the reduction in surface roughness of the W-coated tile used in JT-60U would not due to melting caused by the heat load on the W coating during plasma discharge. Hence, the reduction in the surface roughness might be caused by surface sputtering. However, the effects of transient heat loads such as edge-localized mode (ELM) and disruption on

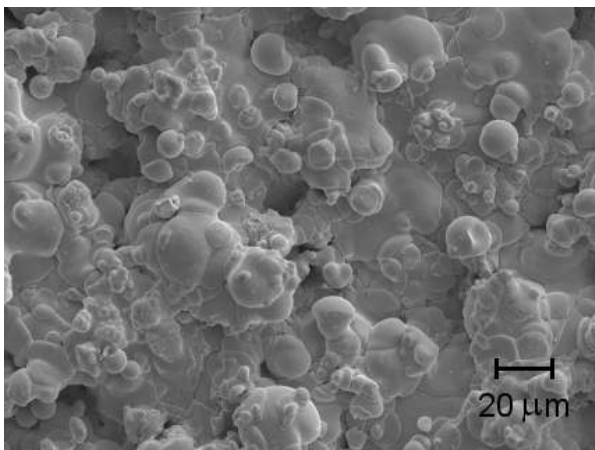


Fig.5 A SE image of surface morphology of the W coating after the thermal heat load tests. Heat flux of 230 cycles at ~ 7 MW/m² for ~ 5 s was deposited by electron beam irradiation.

surface melting have to be investigated.

As shown in Fig. 3 (a), no significant blistering was observed on the W coating. Miyamoto *et al.* reported that no blisters were formed on the toughness enhanced, fine-grained W-1.1 wt% TiC (TFG W-TiC) with a grain size of ~ 1 μ m [19]. The reason they proposed is that since the TFG W-TiC contains more grain boundaries compared with commercially-used W, a larger fraction of implanted D atoms can easily diffuse back to the surface along grain boundaries and can desorb from the surface during plasma exposure. The grain size of the W coating observed by a transmission electron microscope (TEM) was 0.5-2 μ m [20, 21], which was similar to that of TFG W-TiC. Hence, since the implanted D atoms in the W coating easily diffuse back to the surface along the grain boundaries, local accumulations near the surface, for example, grains, grain boundaries and intrinsic defects was suppressed, resulting in no blister formation on the W coating. However, one order of magnitude higher D retention compared with the W-coated samples exposed to pure D plasma in a basic experimental facility, PIECES-B [22], was observed in the W-coated tiles used in JT-60U.

In order to investigate the reason for this high D retention in the W-coated tiles, depth profiles of the implanted D and the main impurities, C and O, in the W coating were measured by SIMS. Figure 6 shows SIMS depth profiles at (a) ~ 11.5 and (b) ~ 33.7 mm from the

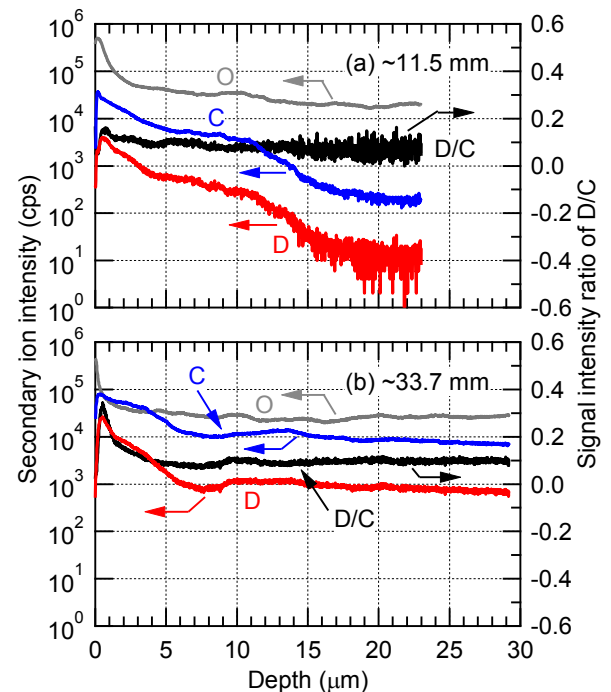


Fig.6 SIMS depth profiles of D, C, and O signal for poloidal positions at (a) ~ 11.5 mm and (b) ~ 33.7 mm from the tile edge. Signal intensity ratios of D/C calculated from the signal intensities are also shown with right axis.

lower tile edge. The D depth profiles were different at the positions: The D signal intensity was highest near the surface and decreased with increase in depth for both measurement positions. At the position of ~ 11.5 mm, the D signal intensity decreased to $\sim 10^1$ cps at ~ 20 μm deep, while at the position of ~ 33.7 mm, the D signal intensity was kept $\sim 10^3$ cps down to ~ 30 μm . The depth profiles of O impurities were constant and were different from those of D. The C impurities were also detected and the depth profiles were similar to the D depth profiles. However, as shown in Fig. 3 (b) no clear C deposition layer was observed on the plasma exposed surface of the W-coated tiles. The depth profiles of the signal intensity ratio of D/C evaluated from the SIMS signals were constant as shown in Fig. 6. These results suggest that implanted D is trapped by the C impurities which are contained in the W coating and that the D retention in the W coating depends on the amount of C impurities.

Three possible sources of C impurities in the W coating are considered; one is the implantation of C eroded from C armor tiles, another is contamination with C impurities during the manufacturing process of the W coating and the other is C diffusion from the CFC substrates. If the C is diffused from CFC substrates, the C signal intensity is highest near the interface between CFC substrate and Re/W interlayer and decreases toward the surface. However, as shown in Fig. 6, since the C signal intensity decreased with increasing depth, C diffusion from the CFC substrates is not dominant within the detection area.

The diffusion coefficient of C in bulk W [23] indicates that the implanted C at the initial discharge of the experimental campaign can diffuse down to few tens of nm, assuming a surface temperature of 700 K and a NBI heating duration of 8420 s including the HH discharge. This is much shallower than the position of the C impurities obtained from the SIMS measurement. This discrepancy indicates that the C diffusion rate in the W coating is much faster than that in the bulk W. In order to investigate the diffusion length of C impurities in the W coating exactly, the C diffusion coefficient in the W coating is required.

Depth profiles of C impurities in the virgin W coating were measured by SIMS as shown in Fig. 7. These depth profiles were obtained from two arbitrary measurement points ("A" and "B"). The C impurities are contained within at least ~ 25 μm deep in the virgin W coating. This indicates that the C impurities come from the manufacturing process of the W coating. However, the C signal intensities obtained from the virgin tiles were about one order of magnitude lower than that from the tiles exposed to plasmas, see Fig. 6. As shown in Fig. 7, the C depth profiles obtained from the virgin W coating were different from each other. The reason for the difference between positions "A" and "B" is probably due to

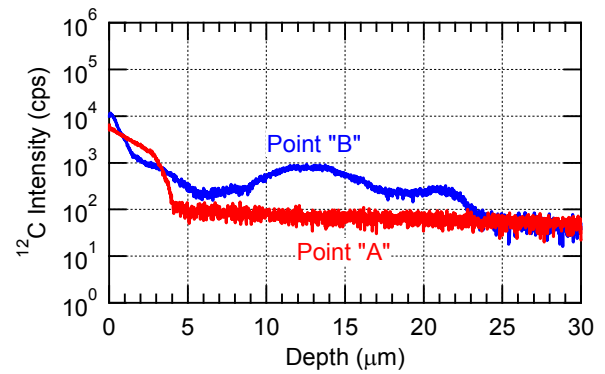


Fig.7 SIMS depth profiles of C impurities in the virgin W coating. Letters "A" and "B" correspond two arbitrary measurement points.

non-uniform distribution of the C impurities compared with the detection area of SIMS measurement ($\sim 3 \times 10^{-3}$ mm^2). Hence, in order to investigate the C impurity sources in the W coating after plasma exposure, it is necessary to obtain the C depth profiles over a wide area.

4. Conclusion

Surface morphology and D retention in the W-coated CFC tiles exposed to JT-60U divertor plasmas were investigated. Surface observations found that the surface of the W coating would be eroded predominantly by sputtering. However, it is necessary to investigate the effect of transient heat loads such as ELM and disruption. Total erosion in the one experimental campaign (total incident ion fluences of 10^{25} - 10^{26} m^{-2}) was below the resolution of cross-sectional observation. No blisters were formed on the W coating. Considering that since more grain boundaries exit in the W coating, the implanted D diffuses back along the grain boundaries more easily and escapes from the surface during the plasma discharges.

Although the total ion fluence and the surface temperature during the experimental campaign depended on the poloidal position of the W-coated tiles, the poloidal distribution of the D retention within ~ 1 mm thickness was uniform at $\sim 10^{22}$ D/ m^2 . This was due to D trapping by the C impurity particles accumulated in the W coating. Two possible C sources in the W coating were considered; one is implantation of C eroded from C armor tiles and the other is contamination of C during the manufacturing process of the W coating on the CFC substrates.

From the results of the present work we suggest that it is necessary to reduce the C impurities in the vacuum vessel or W coating for reduction of the T inventory in the W PFMs.

- [1] G. Federici *et al.*, Nucl. Fusion **41**, 1967 (2001).
- [2] Y. Hirohata *et al.*, J. Nucl. Mater. **363-365**, 854 (2007).
- [3] Y. Hirohata *et al.*, J. Nucl. Mater. **367-370**, 1260 (2007).

- [4] J.P. Coad et al., Nucl. Fusion **46**, 350 (2006).
- [5] J. Roth et al., J. Nucl. Mater. **390-391**, 1 (2009).
- [6] D. Schleußner et al., J. Nucl. Mater. **266-269**, 1296 (1999).
- [7] K. Sugiyama et al., “Deuterium retention in tungsten coatings exposed to the ASDEX Upgrade divertor plasma and irradiated by low-energy ion beams” presented at ICFRM-14.
- [8] T. Tanabe et al., Fusion Eng. Design **49-50**, 355 (2000).
- [9] M. Rubel et al., J. Nucl. Mater. **307-311**, 111 (2002).
- [10] S. Ishida and JT-60U Team, Nucl. Fusion **39**, 1211 (1999).
- [11] K. Tobita and JT-60U Team, Plasma Phys. Control. Fusion **A41**, 333 (1999).
- [12] Y. Ueda et al., J. Nucl. Mater. **363-365**, 66 (2007).
- [13] Y. Ueda et al., Nucl. Fusion **49**, 065027 (2009).
- [14] T. Nakano et al., Nucl. Fusion **49**, 115024 (2009).
- [15] T. Shibahara et al., Nucl. Fusion **46**, 841 (2006).
- [16] W. Wang et al., J. Nucl. Mater. **299**, 124 (2001).
- [17] X. Liu et al., J. Nucl. Mater. **363-365**, 1299 (2007).
- [18] M. Seki et al., Fusion Eng. Design, **15**, 59 (1991).
- [19] M. Miyamoto et al., Nucl. Fusion **49**, 065035 (2009).
- [20] Y. Yahiro et al., J. Nucl. Mater. **386-388**, 784 (2009).
- [21] Y. Niu et al., Proceedings of JSPS-CAS Core University Program Seminar on PWI-PFC and Fusion Technologies, ed. T. Hino and J. Chen, (NIFS-PROC-76, Toki, Japan, 2009) p. 123.
- [22] K. Tokunaga, et al., J. Nucl. Mater **337-339**, 887 (2004).
- [23] W. Eckstein et al., Instr. and Meth. B **153** (1999) 451.