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## Effect of C-He simultaneous implantation on deuterium retention in damaged W by Fe implantation



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#### ABSTRACT

Deuterium (D) retention behaviors for the 3 keV Helium (He<sup>+</sup>) implanted damaged-Tungsten (W) and 10 keV Carbon (C<sup>+</sup>) - 3 keV He<sup>+</sup> simultaneous implanted damaged-W were evaluated by thermal desorption spectroscopy (TDS) to understand the synergetic effect of defect formation and C/He existence on D retention behavior for W with various damage level. For the He<sup>+</sup> implantation, the retention of D trapped by dislocation loops was controlled by 3 keV He<sup>+</sup> fluence. The D retention in the deeper region was reduced by He<sup>+</sup> implantation with higher He<sup>+</sup> fluence due to the formation of He bubbles and dense defects at the surface region which would reduce the effective D diffusion coefficient. In addition, in the case of the simultaneous C<sup>+</sup> - He<sup>+</sup> implantation, the reduction of D retention trapped in the deeper region was also found by the higher C<sup>+</sup> - He<sup>+</sup> fluence. It can be said that D retention behavior was controlled by the formation of He induced defects and accumulation of He near the surface even if the damages were introduced in the deeper region.

#### 1. Introduction

Tungsten (W) is a candidate for plasma facing materials (PFMs) in D-T fusion reactors like ITER due to good thermal properties and lower sputtering yield [1–3]. During the plasma operation, irradiation defects will be introduced into W by 14 MeV neutrons and energetic ions including hydrogen isotopes, helium (He) and impurities like carbon (C) escaped from plasma. It is well known that hydrogen isotopes are trapped by the implantation defect, leading to enhanced fuel retention in W [4,5]. In our previous study [6], the retentions of D trapped by vacancies or voids were increased as the damage concentration was increased, and voids became major and stable D trapping sites. In addition, it was reported that the large amount of C was observed, which deposited on the full metal wall after plasma operation in QUEST [7]. The existence of C in W will change the trapping behavior for hydrogen isotope. For example [8,9], preexistence of C in vacancies reduced D trapping energy. In previous experiment [10,11], it was also found that C-W layer formed by C<sup>+</sup> implantation suppressed the D diffusion

toward the bulk. However, in the actual reactor condition, He bubbles will be also formed and suppress the D diffusion toward the bulk [12]. Therefore, it is important to elucidate synergism of not only implantation defect and the existence of C but also, He bubble on hydrogen isotope retention in W. At first, this study focused on the D retention behavior for the He<sup>+</sup> implanted damaged-W with the various damage concentrations introduced by Fe ion to understand the interaction between He<sup>+</sup> implantation and implantation defects by heavy ion in the deeper region, refered as bulk. After that, the simultaneous C<sup>+</sup>- He<sup>+</sup> implantation for damaged-W by Fe<sup>2+</sup> implantation was performed to evaluate hydrogen isotope dynamics in W under actual reactor conditions by using the result of Ref. [9] and the He<sup>+</sup> implanted damaged-W.

#### 2. Experimental

A disk-type polycrystalline W with the size of 10 mm diameter and 0.5 mm thickness, purchased from Allied Material (A.L.M.T.) Corp. Ltd,

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was used as a sample. The sample was the mechanically polished to be the surface roughness of ~ 50 nm. To remove impurities and residual damages, these samples were heated at 1173 K for 30 min under ultrahigh vacuum (<  $10^{-6}$  Pa) [13].

Thereafter, 6 MeV Fe<sup>2+</sup> was implanted to prepare the damaged-W samples at room temperature with the average damage level of 0.03 and 0.3 displacement per atom (dpa) by using the 3 MV tandem accelerator, TIARA (Takasaki Ions Accelerators for Advanced Radiation Application) at QST (National Institutes for Quantum and Radiological Science and Technology). The Fe<sup>2+</sup> fluence were set to be 0.217- $2.17 \times 10^{18} \text{ Fe}^{2+} \text{ m}^{-2}$ . The damage level was calculated by SRIM (The Stopping and Range of Ions in Matter) and calculation result showed the average damage level in the region of each ion implantation depth [14]. Then, 3 keV He<sup>+</sup> implantation or the simultaneous 10 keV C<sup>+</sup> -3 keV He<sup>+</sup> implantation for these samples were performed. The ion flux for only He<sup>+</sup> implantation was  $1.0 \times 10^{17}$  He<sup>+</sup> m<sup>-2</sup> s<sup>-1</sup> up to the ion fluence of 0.1–1.0  $\times$  10<sup>21</sup> He<sup>+</sup> m<sup>-2</sup> at room temperature. The ion flux for the simultaneous  $C^+$  -  $He^+$  implantation was 1.0  $\times$   $10^{17}\,C^+\,m^{-2}\,s^{\text{--}1}$ /  $1.0 \times 10^{17}$  He<sup>+</sup> m<sup>-2</sup> s<sup>-1</sup> up to the ion fluence of  $0.1-1.0 \times 10^{21}$  $C^+ m^{-2} / 0.1 - 1.0 \times 10^{21} He^+ m^{-2}$  at room temperature. Thereafter, 3 keV  $D_2^+$  was implanted with the ion flux of 1.0–3.0  $\times$  10<sup>18</sup> D<sup>+</sup> m<sup>-2</sup> s<sup>-2</sup> up to the ion fluence of  $1.0-3.0 \times 10^{22} \text{ D}^+ \text{ m}^{-2}$  at room temperature.  $C^{+},\,He^{+}$  and  $D_{2}^{+}$  implantation were performed by the triple ion implantation system at Shizuoka University [15]. The maximum implantation depth of 10 keV  $C^+$ , 3 keV He<sup>+</sup> and 3 keV  $D_2^+$  are the same, which was found to be up to 50 nm by SRIM calculation using displacement threshold energy of 50 eV. 6 MeV Fe<sup>2+</sup> implantation depth was calculated to be up to 1.8 µm by SRIM. Finally, thermal desorption spectroscopy (TDS) measurements were performed from room temperature to 1173 K with the heating rate of 0.5 K s<sup>-1</sup> to evaluate the  $D_2$ desorption behavior.

In addition, the Transmission Electron Microscope (TEM, JEM 2000EX, JASCO Inc.) observations at Kyushu University were also applied for the simultaneous C<sup>+</sup> - He<sup>+</sup> implanted W, Only C<sup>+</sup> implanted W and He<sup>+</sup> implanted W with the C<sup>+</sup> fluence of  $1.0 \times 10^{21}$  C<sup>+</sup> m<sup>-2</sup> and He<sup>+</sup> fluence of  $1.0 \times 10^{21}$  He<sup>+</sup> m<sup>-2</sup> to observe the formation of defect and He bubble produced by these ion implantations.

#### 3. Result and discussion

## 3.1. $He^+$ fluence dependence on D retention for the sequential $He^+$ implanted damaged-W

Fig. 1 shows  $D_2$  TDS spectra for the He<sup>+</sup> implanted damaged-W and only damaged-W with the damage concentration of 0.3 dpa with  $D_2^+$  fluence of  $1.0 \times 10^{22} D_2^+ m^{-2}$ . The TDS spectra were consisted of three desorption peaks located at around 400, 600 and 780 K. Based on

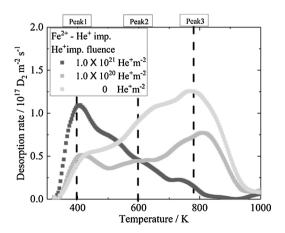


Fig. 1.  $D_2$  TDS spectra for the  ${\rm He}^+$  implanted damaged-W and only damaged-W with the damage level of 0.3 dpa.

previous studies, these desorption peaks are attributed to the desorption of D trapped by dislocation loops and/or adsorbed on the surface as Peak 1 [16,17], that trapped by vacancies as Peak 2, and that trapped by voids as Peak 3 [18,19].

The desorption of D as Peaks 3 for the He<sup>+</sup> implanted damaged-W was decreased. On the other hand, large increase of D desorption as Peak 1 was observed by He<sup>+</sup> fluence of  $1.0 \times 10^{21}$  He<sup>+</sup> m<sup>-2</sup>. The dislocation loops by He<sup>+</sup> implantation was observed in our previous studies [20,21]. It can be said that the amount of D trapped in the defect by Fe<sup>2+</sup> implantation was decreased due to the existence of He or enhancement of dislocation loops by He<sup>+</sup> implantation near surface region even if the damage level was reached to 0.3 dpa.

## 3.2. Damage level dependence on D retention for the $He^+$ implanted damaged-W

Fig. 2 shows the TEM images for He<sup>+</sup> implanted W with the He<sup>+</sup> fluence of  $1.0 \times 10^{21}$  He<sup>+</sup> m<sup>-2</sup> and the damaged-W with the damage level of 0.3 or 0.03 dpa with  $D_2^+$  fluence of  $1.0 \times 10^{22}$  D<sup>+</sup> m<sup>-2</sup>. It was clear that the density of dislocation loops (black dots in Fig.2) for He<sup>+</sup> implanted W was higher than that for the damaged-W due to the longer penetration depth of 1.8  $\mu m$  compared to that for He<sup>+</sup> of 50 nm. In addition, the density of the dislocation loops was almost the same among the damaged-W samples with 0.3 and 0.03 dpa. Fig. 3 shows the D<sub>2</sub> TDS spectra for the He<sup>+</sup> implanted damaged-W and only the damaged-W with various damage levels and He<sup>+</sup> fluences, where all the samples are damaged by  $Fe^{2+}$  implantation with the damage levels of 0 to 0.3 dpa and the He dose of  $0-1.0 \times 10^{21}$  He<sup>+</sup> m<sup>-2</sup>. The D desorption rate for Peak 1 was almost the same, even if the damage level in the deeper region was changed. Therefore, the amount of D trapped by dislocation loops would be controlled by He<sup>+</sup> fluence, which was consistent with the result of TEM images in Fig. 2. In addition, at the He<sup>+</sup> fluence of  $1.0 \times 10^{21}$  He<sup>+</sup> m<sup>-2</sup>, the retention of D for Peak 3 was almost the same for all the samples in comparison to the other samples with lower He<sup>+</sup> fluence (0 ~  $1.0 \times 10^{20}$  He<sup>+</sup> m<sup>-2</sup>), indicating that most of D was trapped by dislocation loops introduced by He<sup>+</sup> implantation with the higher fluence of  $1.0 \times 10^{21}$  He<sup>+</sup> m<sup>-2</sup>, and The D retention in the deeper region (Fe<sup>2+</sup> implantation depth of 1.8  $\mu$ m) was reduced.

## 3.3. Higher $D_2^+$ fluence effect on D retention for the sequential He<sup>+</sup> implanted damaged-W

Fig. 4 shows D<sub>2</sub> TDS spectra for the He<sup>+</sup> implanted damaged-W and only damaged-W (0.3 dpa) with D<sub>2</sub><sup>+</sup> fluence of  $3.0 \times 10^{22}$  D<sup>+</sup> m<sup>-2</sup>. The D retention as Peak 3 for the He<sup>+</sup> implanted damaged-W was decreased, which was consistent with the low D<sub>2</sub><sup>+</sup> fluence case as shown in Section 3.1, although the D desorption rate as Peak 1 was increased due to the He<sup>+</sup> implantation. Fig. 5 shows D<sub>2</sub> TDS spectra for the He<sup>+</sup> implanted damaged-W with various damage levels, He<sup>+</sup> fluence of  $1.0 \times 10^{21}$  He<sup>+</sup> m<sup>-2</sup>, and D<sub>2</sub><sup>+</sup> fluence of  $3.0 \times 10^{22}$  D<sup>+</sup> m<sup>-2</sup>, where the D retention as Peak 3 was enhanced as damage level was increased in comparison to that in Fig. 2 with He<sup>+</sup> fluence of  $1.0 \times 10^{21}$  He<sup>+</sup> m<sup>-2</sup>. The D retention as Peak 1 was also higher than that in lower D<sub>2</sub><sup>+</sup> fluence. From these results, it indicated that D trapped by the surface was saturated in the higher D<sub>2</sub><sup>+</sup> fluence ( $3.0 \times 10^{22}$  D<sup>+</sup> m<sup>-2</sup>) and D would diffuse toward the bulk.

## 3.4. Effect of the simultaneous $C^+$ - $He^+$ implantation on D retention in damaged-W

Fig. 6 shows D<sub>2</sub> TDS spectra for 0.3 dpa damaged-W followed by simultaneous  $C^+$ -He<sup>+</sup> implantation with various fluences. Large enhancement of D retention as Peak 1 was observed at higher  $C^+$  - He<sup>+</sup> fluence. In addition, the retention of D trapped as Peak 3 was decreased with increasing the  $C^+$  - He<sup>+</sup> fluence. These D retention behaviors were almost the same as that in the He<sup>+</sup> implanted damaged-W in Fig. 1.

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