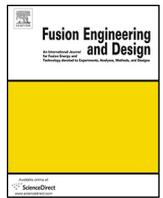




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Effect of sequential Fe²⁺ – C⁺ implantation on deuterium retention in W

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HIGHLIGHTS

- D retention behavior for the sequential Fe²⁺-C⁺ implanted W were evaluated by TDS and β –ray-induced X-ray spectroscopy (BIXS).
- The retention of D trapped by dislocation loops was controlled by the damage concentration introduced by C⁺ fluence.
- The amount of D trapped in the bulk was increased due to the defect formed by Fe²⁺ implantation.

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ABSTRACT

Deuterium (D) retention behavior for the sequential 6 MeV iron (Fe) and 10 keV carbon (C) implanted tungsten (W) were evaluated by thermal desorption spectroscopy (TDS) and β-ray-induced X-ray spectroscopy (BIXS) to understand the synergetic effect of defect formation and C existence on D retention behavior for W under various damage distribution profiles. The experimental results indicated that retention of D trapped by dislocation loops was controlled by 10 keV C⁺ implantation. The D retention was reduced in the sequential Fe²⁺ – C⁺ implanted W with higher C⁺ fluence in comparison to that with lower C⁺ fluence due to the formation of C-W layer which suppressed D diffusion toward the bulk and dense defects at the surface which reduce effective D diffusion coefficient. On the other hand, the amount of D trapped by the defects in the deeper region than C⁺ implantation region (50 nm) was increased due to the formation of dense defects by 6 MeV Fe²⁺ implantation within the depth of 1.5 μm.

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1. Introduction

Tungsten (W) is thought to be a candidate for plasma facing materials (PFMs) in D-T fusion reactors like ITER due to higher melting point and lower sputtering yield. [1–3] During the plasma operation, W will be exposed to energetic particles, such as hydrogen isotopes, neutron, and impurities like carbon (C) escaped from plasma. It was reported that large amount of C was precipitated on the full metal first wall after plasma discharge in QUEST [4]. It is well

known that hydrogen isotope is trapped by the damages produced by energetic particle irradiation and the existence of C will change the hydrogen isotope trapping behavior. In our previous study [5], the retentions of D trapped by vacancies or voids were increased as the damage concentration was increased, and voids became major D trapping sites. However, the formation of C-W layer by C⁺ implantation suppressed the D diffusion toward the bulk [6,7]. In addition, the existence of C in vacancies or voids affected on the stability of D trapping [8]. Therefore, it is quite important to evaluate synergetic of vacancies and C on D retention in W. This study focuses on the D retention behavior in the sequential Fe²⁺ – C⁺ implanted W with the various damage concentrations to comprehend hydrogen isotope dynamics in actual reactor conditions.

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2. Experimental

A disk-type polycrystalline W with the size of 10 mm diameter and 0.5 mm thickness, which was purchased from Allied Material (A.L.M.T.) Corp. Ltd, was used as a sample. To remove impurities and residual damages, these samples were preheated at 1173 K for 30 min under ultrahigh vacuum ($< 10^{-6}$ Pa) [9].

Thereafter, 6 MeV Fe^{2+} was implanted at room temperature with the damage concentration of 0.005, 0.05 and 0.5 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^{2+} fluence were set to be $7.25 \times 10^{19} - 7.25 \times 10^{21} \text{Fe}^{2+} \text{m}^{-2}$. (The damage concentration was calculated by SRIM (The Stopping and Range of Ions in Matter) code with "Ion Distribution and Quick Calculation of Damage" mode and calculation result showed the average damage concentration in the region of each ion implantation depth [10]. Then, 10 keV C^+ implantation for these samples and non-implanted W were performed with the ion flux of $1.0 \times 10^{17} \text{C}^+ \text{m}^{-2} \text{s}^{-1}$ up to the ion fluence of $1.0 \times 10^{20} \text{C}^+ \text{m}^{-2}$ and $1.0 \times 10^{21} \text{C}^+ \text{m}^{-2}$ at room temperature with the damage concentration of 0.6 and 6.0 dpa, respectively. Thereafter, 3 keV D_2^+ was implanted with the ion flux of $1.0 \times 10^{18} \text{D}_2^+ \text{m}^{-2} \text{s}^{-1}$ up to the ion fluence of $1.0 \times 10^{22} \text{D}_2^+ \text{m}^{-2}$ at room temperature. C^+ and D_2^+ implantation was performed by the triple ion implantation system at Shizuoka University [11]. The maximum implantation depth of 10 keV C^+ and 3 keV D_2^+ were found to be 50 nm by SRIM calculation using displacement threshold energy of 50 eV 6 MeV Fe^{2+} implantation depth was also calculated to be 1.5 μm by SRIM. Finally, thermal desorption spectroscopy (TDS) measurements were performed from room temperature to 1173 K with the heating rate of 0.5Ks^{-1} to evaluate the D_2 desorption behavior.

On the other hand, to validate tritium depth profile, tritium (T) implantation was performed for the sequential $\text{Fe}^{2+} - \text{C}^+$ implanted W with the damage concentration of 0.5 dpa by Fe^{2+} implantation and only C^+ implanted W at University of Toyama. The detail information of the T ion implantation system was found in Ref. [12]. The T ion fluence and ion energy was estimated to be $5.67 \times 10^{17} \text{T}^+ \text{m}^{-2}$ ($1.34 \times 10^{19} \text{D}^+ + \text{T}^+ \text{m}^{-2}$) and 1.8 keV T^+ (3 keV $\text{D}^+ + \text{T}^+$), respectively. After T ion implantation, β -ray induced X-ray spectroscopy (BIXS) [13,14] was performed to elucidate the depth profile of T in the damaged W for 19 h.

In addition, the Transmission Electron Microscope (TEM, JEM 2000EX, JASCO Inc.) observations at Kyushu University were also applied for the sequential $\text{Fe}^{2+} - \text{C}^+$ implanted W with the C^+ fluence of $1.0 \times 10^{20} \text{C}^+ \text{m}^{-2}$ and $1.0 \times 10^{21} \text{C}^+ \text{m}^{-2}$ to observe the defect distribution produced by the sequential implantation.

3. Result and discussion

3.1. C^+ fluence dependence on D retention for the sequential $\text{Fe}^{2+} - \text{C}^+$ implanted W

Fig. 1 shows D_2 TDS spectra for the sequential 0.05 dpa $\text{Fe}^{2+} - \text{C}^+$ implanted W and only 0.05 dpa Fe^{2+} implanted W. The damage concentration of C^+ implantation fluence of $1.0 \times 10^{21} \text{C}^+ \text{m}^{-2}$ and C^+ fluence of $1.0 \times 10^{20} \text{C}^+ \text{m}^{-2}$ were 6.0 and 0.6 dpa, respectively. The TDS spectra were consisted of three desorption peaks located at about 400, 600 and 780 K. According to the 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 [15,16], that trapped by vacancies as Peak 2, and that trapped by voids as Peak 3 [17,18].

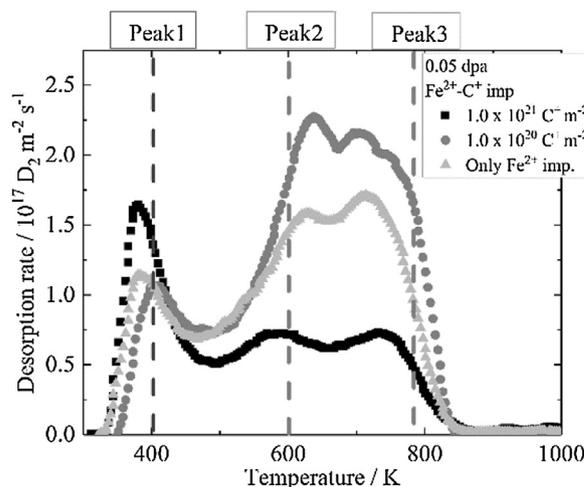


Fig. 1. D_2 TDS spectra for the sequential $\text{Fe}^{2+} - \text{C}^+$ implanted W and Fe^{2+} implanted W.

The desorptions of D as Peaks 2 and 3 for the sequential $\text{Fe}^{2+} - \text{C}^+$ irradiated W with lower C^+ fluence (C^+ fluence of $1.0 \times 10^{20} \text{C}^+ \text{m}^{-2}$) were increased compared with that for the only Fe^{2+} implanted W due to accumulation of vacancies by C^+ implantation. However, the desorption rate of D more than 600 K for the sequential $\text{Fe}^{2+} - \text{C}^+$ implanted W at higher C^+ fluence (C^+ fluence of $1.0 \times 10^{21} \text{C}^+ \text{m}^{-2}$) was reduced, indicating that the formation of C-W layer would suppress D diffusion toward the bulk [6,7] and the defect density produced by C^+ implantation with higher fluence reduces the effective diffusion coefficient. In addition, it was reported that trapping energy of hydrogen with existence of C in W reduced its trapping energy and D desorption as Peak 2 for the sequential $\text{Fe}^{2+} - \text{C}^+$ implanted W with the C^+ fluence of $1.0 \times 10^{21} \text{C}^+ \text{m}^{-2}$ was shifted toward lower temperature side. [19].

Fig. 2 shows the TEM images for the sequential Fe^{2+} and C^+ implanted W with the C^+ fluence of $1.0 \times 10^{20} \text{C}^+ \text{m}^{-2}$ and $1.0 \times 10^{21} \text{C}^+ \text{m}^{-2}$. It was clear that the density of dislocation loops (black dots in Fig. 2) was increased as the C^+ fluence was increased, and this result is consistent with the present TDS results that the desorption of D as Peak 1 (400 K) was increased as C^+ fluence was increased.

3.2. Fe^{2+} fluence dependence on D retention for the sequential $\text{Fe}^{2+} - \text{C}^+$ implanted W

The Hydrogen Isotope Diffusion and Trapping (HIDT) simulation [5] was applied to analyze D trapping states for sequential Fe^{2+} and C^+ implanted W and only C^+ implanted W. The simulation parameters used in this study were summarized in Table 1.

Figs. 3 and 4 and 4 overlapped the TDS spectra for the sequential $\text{Fe}^{2+} - \text{C}^+$ implanted W or only C^+ implanted W with the C^+ fluence of $1.0 \times 10^{21} \text{C}^+ \text{m}^{-2}$ and HIDT simulation results. The D desorption rate as Peak 1 for the sequential $\text{Fe}^{2+} - \text{C}^+$ implanted W and only C^+ implanted W were almost the same in spite of increasing the Fe^{2+} fluence. It can be said that the retention of D trapped by dislocation loops was not controlled by the damage concentration introduced by Fe^{2+} implantation. On the other hand, the desorption of D_2 as Peak 3 was increased as damage concentration by Fe^{2+} implantation was increased, indicating that D was trapped by the defects like vacancies or voids formed by Fe^{2+} implantation.

By assuming the trapping energies of Peaks 1–3 to be 0.5, 0.7–1.2, 1.5 eV, respectively, it was clear that the simulation could represent the experimental results well. In particular, two different trapping energies at Peak 2 were selected to demonstrate the effect of C existence near surface region. One was trapping

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