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Influence of tungsten–carbon mixed layer and irradiation defects on deuterium retention behavior in tungsten



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ABSTRACT

The D_2^+ fluence dependence on deuterium (D) retention was studied to clarify the D retention mechanism in tungsten. The additional D desorption stage was observed around 660 K in the TDS spectrum for a sample implanted with D_2^+ up to the fluence of $10^{23} D^+ m^{-2}$, which desorption stage was not observed the D_2^+ implanted sample with the fluence less than $10^{22} D^+ m^{-2}$. The TEM observation showed that the highly dense voids were formed in tungsten by D_2^+ implantation with the fluence of $10^{23} D^+ m^{-2}$, considering that the D would be trapped by voids. To understand the D trapping by voids in C^+ implanted tungsten, $C^+ - D_2^+$ sequential implantation experiments at various C^+ implantation temperatures were performed. It was found that the amount of D desorbed around 560 K was increased by increasing the C^+ implantation temperature. The formation of the voids was observed with increasing the C^+ implantation temperature by TEM, indicating that the increase of D desorption around 560 K was caused by the formation of voids. However, the desorption temperature of D trapped by voids in C^+ implanted sample was lower than that in D_2^+ implanted one. TEM observation and XPS measurement indicated that this difference was caused by the increase of void size and/or the presence of implanted carbon.

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1. Introduction

The usage of tungsten has been considered for the plasma facing material (PFM) in D–T fusion reactors. The first wall is exposed to energetic hydrogen isotopes and impurities like carbon during plasma operation and the irradiation defects will be formed with formation of W–C mixed layer on the tungsten surface. It is well known that the W–C mixed layer causes the hydrogen isotope retention on tungsten under D–T fusion circumstance [1]. Several impurities such as carbon and oxygen were detected in QUEST at Kyushu University although it is constructed with all metallic first walls [2], indicating that it is difficult to remove carbon completely even if the all metallic first walls are adopted. The knowledge of hydrogen isotope retention behaviors in PFM is quite important to understand the fuel behaviors for the operation of fusion reactors. In our previous study [3], D_2^+ implantation was performed to obtain fundamental knowledge for the retention behavior of

hydrogen isotopes in tungsten. $C^+ - D_2^+$ sequential implantations (C^+ was implanted into tungsten before D_2^+) was also performed to understand the effect of C^+ implantation on D retention behavior in tungsten. The D_2 thermal desorption spectroscopy (TDS) spectrum consisted of three desorption stages in the case of D_2^+ implantation [3]. The D desorption stage at 660 K was increased with increasing of D_2^+ fluence. It was indicated that additional D trapping site was formed as increasing D_2^+ fluence. In addition, the D desorption stage at 660 K was hardly observed for $C^+ - D_2^+$ implanted sample [4]. Therefore, the behavior of D desorption stage at 660 K is critical for understanding of D retention behavior in pure tungsten and C^+ implanted tungsten. In this study, D_2^+ implantation and $C^+ - D_2^+$ sequential implantation with various C^+ implantation temperatures were performed to clarify the trapping site of D desorbed at higher temperature region and W–C mixed layer formation on D retention behavior.

2. Experimental

A disk-type polycrystalline tungsten with stress-relieved conditions (heated at 1173 K), which was purchased by Allied Material

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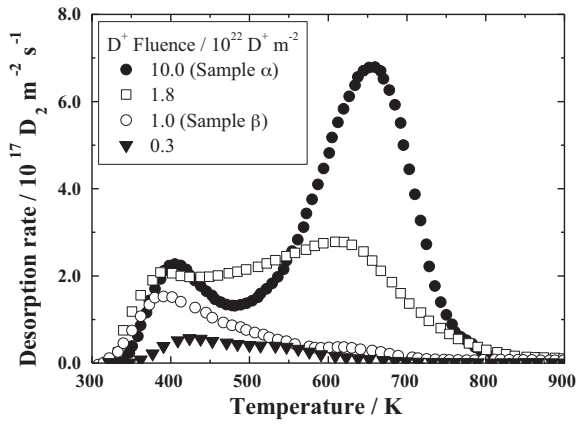


Fig. 1. D₂ TDS spectra of D₂⁺ implanted tungsten with various D⁺ fluence.

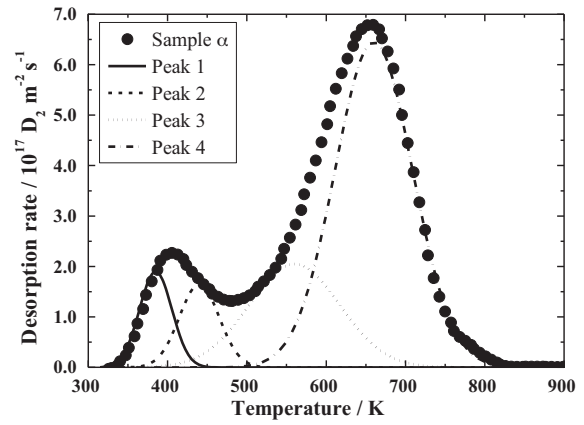


Fig. 2. D₂ TDS spectra and the separated peaks of D₂⁺ implanted tungsten.

Co. Ltd, was used as samples. The sample size was 10 mm diameter and 0.5 mm thickness. The samples were polished mechanically to the roughness of less than 1 μm by SiC abrasive papers and diamond suspensions. These samples were preheated at 1173 K for 30 min under ultrahigh vacuum (<10⁻⁶ Pa) to remove the impurities and residual damages.

The 3.0 keV D₂⁺ implantation was performed for tungsten with an ion flux of 1.0 × 10¹⁸ D⁺ m⁻² s⁻¹ and an ion fluence of 1.0 × 10²³ D⁺ m⁻² at R.T. (D₂⁺ implantation experiment), which D₂⁺ fluence was set to be higher value than that in our previous study [3] in order to observe additional trapping sites. This sample was named as Sample α.

The C⁺-D₂⁺ sequential implantation was done using 10 keV C⁺ and 3 keV D₂⁺ (C⁺-D₂⁺ sequential implantation experiment). The C⁺ implantation was performed with the ion fluence of 1.0 × 10²¹ C⁺ m⁻² at room temperature, 473 K and 673 K to introduce voids in tungsten. These samples were named as Sample A, Sample B and Sample C, respectively. Thereafter, D₂⁺ was implanted with fluence of 1.0 × 10²² D⁺ m⁻² at R.T. for all samples.

After these ion implantations, TDS measurements were performed from room temperature to 1173 K with a heating rate of 0.5 K s⁻¹ to investigate the D retention behavior. The chemical states of carbon and tungsten were evaluated by X-ray Photoelectron Spectroscopy (XPS) (ESCA1600 system, ULVAC-PHI Inc.) using Mg-Kα X-ray source (1253.6 eV). Transmission Electron Microscope (TEM) (JEM 2000EX, JASCO Inc.) observations were also performed to observed the irradiation defects and voids formed by these ion implantations at Kyushu University.

3. Results and discussion

Fig. 1 shows the D₂ TDS spectra for D₂⁺ implanted tungsten with various D⁺ fluences as Sample α. The TDS spectra with the ion fluence of 0.3–1.8 × 10²² D⁺ m⁻² were also shown in this figure, where that with the ion fluence of 1.0 × 10²² D⁺ m⁻² was named as Sample β. The D₂ desorption was observed in the temperature range of 300–750 K. These spectra could be consisted of four D desorption stages at around 380 K, 440 K, 560 K and 660 K, named as Peaks 1, 2, 3 and 4, respectively as shown in Fig. 2. The D desorptions as Peaks 1, 2 and 3 were attributed to the desorptions of D adsorbed on the surface, trapped by dislocation loops and vacancies, respectively [5,6]. In the D⁺ fluence less than or equal to 1.0 × 10²² D⁺ m⁻², the major D₂ desorption stage was observed as Peaks 1 and 2. The amount of D trapped by vacancies as Peak 3 was proportionally increased as the D⁺ fluence increased in the fluence from 1.0 × 10²² D⁺ m⁻² to 1.8 × 10²² D⁺ m⁻². However, the shape of D₂ TDS spectrum was clearly changed in the fluence of 1.0 × 10²³ D⁺ m⁻², where the major D₂ desorption stage was shifted

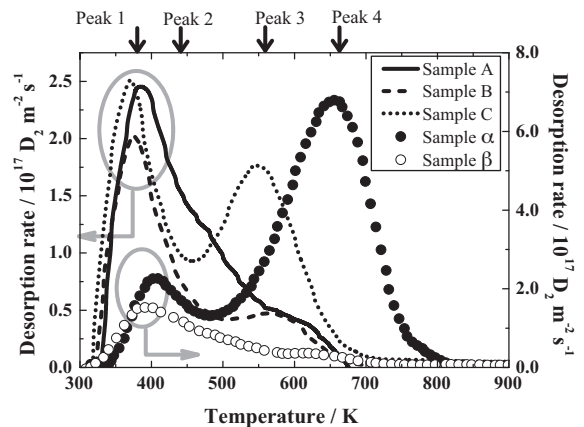


Fig. 3. D₂ TDS spectra of D₂⁺ implanted tungsten and C⁺-D₂⁺ sequential implanted tungsten with various.

toward the higher temperature of 660 K, indicating that the additional trapping site would be formed.

To understand the D trapping by voids for C⁺ implanted tungsten, C⁺-D₂⁺ sequential implantation experiments were performed and compared to only D₂⁺ implantation with higher D₂⁺ fluence. Fig. 3 shows the D₂ TDS spectra for Samples α, β, A B and C. The D₂ desorption for C⁺ implanted sample was observed at 300–700 K. These spectra were consisted of three D desorption stages as Peaks 1, 2, and 3. The D retentions for Samples α, A, B and C were summarized in Fig. 4. The D retentions as Peaks 1 and 2 were similar with the only D₂⁺ implanted samples. However, the D retention as Peak 3 was increased for Sample C compared to other C⁺ implanted

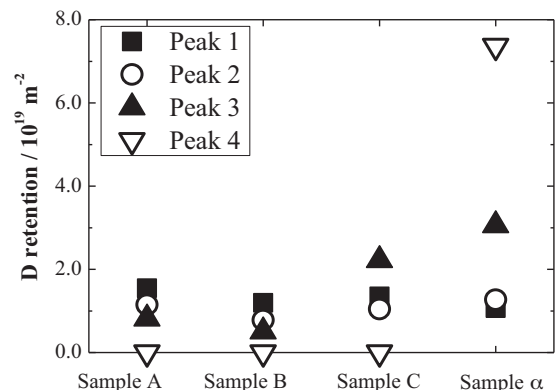


Fig. 4. The deuterium retentions of each peak in the samples.

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