J. Plasma Fusion Res. SERIES, Vol. 9 (2010)

Correlation between desorption of deuterium and recovery of irradiation defects in simultaneously deuterium and carbon ion-implanted tungsten

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(Received: 20 November 2009 / Accepted: 23 January 2010)

The experiments on simultaneous C^+ and D_2^+ implantation into a tungsten substrate was performed to investigate a synergism of the dual ion implantation on desorption of deuterium and recovery of irradiation defects. These results were compared with that for the D_2^+ ions implanted tungsten. It was found that the TDS spectrum for the simultaneous C^+ and D_2^+ implanted sample was quite different from that for the D_2^+ implanted one and the deuterium retention increased by the simultaneous C^+ and D_2^+ implantation, indicating that deuterium would be trapped by carbon and/or irradiation defects. TEM images show that the dislocations and dislocation loops were introduced into tungsten by both of the D_2^+ and simultaneous C^+ and D_2^+ implantation, and the density and size of irradiation defects for the simultaneous C^+ and D_2^+ implanted sample were higher and larger than those for the D_2^+ implanted one. During heating up to 1073K, the dislocations and dislocation loops remained for the simultaneous C^+ and D_2^+ implanted sample, although both of them were moved and/or annihilated for the D_2^+ implanted sample. It was concluded that the size and density of the defects would influence on the deuterium desorption and the defects recovery.

Keywords: tungsten, C^+ and D_2^+ simultaneous implantation, irradiation defects, TDS, TEM.

1. Introduction

Obtaining new data on tritium retention in fusion reactors is one of the critical issues for the estimation of tritium recycling. Tungsten is considered as one of the candidates for ITER divertor components due to its low sputtering yield and low tritium inventory [1-4]. Besides,, carbon is thought to be used for high heat flux regions of the plasma facing components. Thus, combination use of tungsten and carbon will be employed for the divertor region of ITER [2-4]. During plasma operations, carbon will be sputtered by energetic particles and will be implanted into the surface of tungsten accompanied with hydrogen isotopes including tritium, leading the formation of a W-C mixed layer and generation of irradiation damages [5-9]. Therefore, the dynamics of tritium inventory in the W-C mixed layer have to be elucidated to estimate the tritium recycling in D-T fusion reactor.

In our previous studies [10], experiments on simultaneous C^+ and D_2^+ implantation experiments have been carried out with various ion flux ratio of C^+/D^+. It was found that the C^+/D^+ flux ratio was one of the important factor for the determination of deuterium retention; the deuterium retention increased as the C^+/D^+ flux ratio decreased. Furthermore, it was also shown that the deuterium retention for the simultaneous C^+ and D_2^+ implanted sample with lower C^+/D^+ flux ratio was four times as large as that for the D_2^+ ion implanted sample, and what the deuterium would be trapped by the irradiation defects was suggested[11]. From these experiments, it was considered that the investigation of deuterium desorption from irradiation defects should be needed.

In the present study focused on the deuterium desorption behavior, and the correlation between the desorption of deuterium and the recovery of irradiation defects was elucidated by means of Thermal Desorption Spectroscopy (TDS) and Transmission Electron Microscopy (TEM).

2. Experimental

Stress-relieved tungsten samples with sizes of 10 mm in diameter and 0.5 mm in thickness were used. The samples were polished mechanically to mirror surface finish and pre-heated at 1173 K for 10 minutes in vacuum to remove surface impurities and damages induced by the polishing process. The simultaneous ion implantation system has been set up at Shizuoka University, and C^+, D_2^+ and He^+ could be simultaneously implanted by this system. It also features that the TDS chamber and the implantation chamber were connected through the load lock chamber. CO_2 gas was used as the C^+ source to prevent contamination of hydrogen impurity. A E×B mass separator was installed at the head of the C^+ gun. In the present study, simultaneous C^+ and D_2^+ (dual ions) implantation experiments were performed at room

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temperature with ion energies of 10 keV C^+ and 3 keV D_2^+, fluxes of 0.2 ×10^{18} C^+ m^{-2} s^{-1} and 1.0 × 10^{18} D^+ m^{-2} s^{-1} up to fluences of 2.0 ×10^{21} C^+ m^{-2} and 1.0 × 10^{22} D^+ m^{-2}. The C^+/D^+ flux ratio was fixed to be 0.2. To compare the deuterium retention and microstructure change with those obtained for the D_2^+ ion implanted tungsten, the D_2^+ (single ion) implantation experiment was also conducted with the same experimental conditions. After the ion implantation, TDS experiments were performed with a heating rate of 0.5 K s^{-1} from room temperature to 1173 K to find out the desorption behavior and estimate the deuterium retention.

Additionally,, the TEM observations with isochronal annealing experiments were performed to elucidate the characterization the irradiation defects and their annealing behaviors. For the TEM observation, small tungsten specimens with a diameter of 3 mm and thickness of 0.1mm were used. The samples were polished by the twin-jet electro-polishing method and thereafter, the single ion and dual ions implantations were performed at the same conditions mentioned above. The TEM observation (JEM 2000EX, JASCO Inc.) was performed at Kyushu University during in-situ heating from R.T. up to 1073 K at every 100 K step to clarify the recovery behavior of irradiation defects.

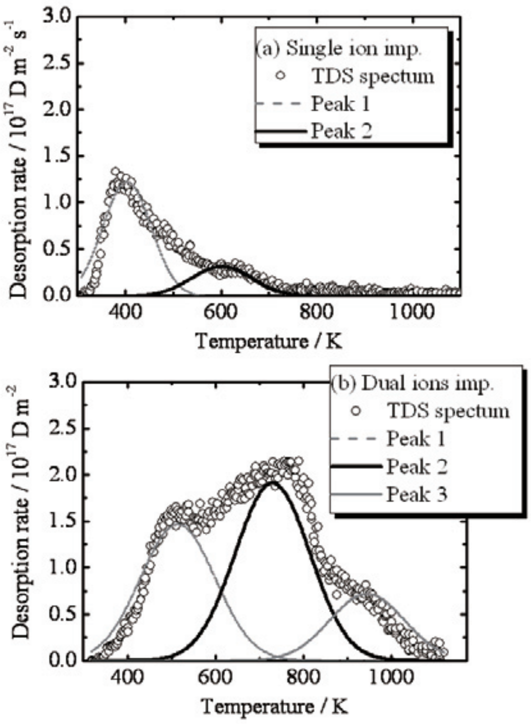

Fig. 1 Thermal desorption spectra of D_2 from
(a) Single ion implanted tungsten and
(b) Dual ions implanted tungsten.

3. Results and discussion

Figure 1 shows the D_2 TDS spectra for the single ion implanted and the dual ions implanted samples. The desorption of deuterium for the dual ions implanted sample was observed in the temperature range of 300-1173 K. The TDS spectra consisted of three major desorption stages, located at around 500, 750 and 900 K. These three stages were thought to be a desorption of deuterium retained on the surface and/or in the intrinsic defects existing in tungsten, that trapped by the ion-induced defects like dislocations and/or dislocation-loops and that trapped by carbon with forming C-D bonds, respectively [11]. In contrast, the TDS spectrum for the single ion implanted sample had two desorption stages. It was found from both figures that the temperature region of the deuterium desorption from ion-induced defects for the dual ions implanted sample was shifted about 100 K toward higher temperature side, indicating that deuterium trapped at ion-induced defects for the dual ions implanted sample could need higher temperature for the desorption compared to that for the single ion implanted one. The total deuterium retention for the dual ions implanted sample was about four times as large as that for the single ion implanted sample. In doing so, the retention of deuterium trapped at ion-induced defects for the dual ions implanted sample was much larger than that for the single ion implanted

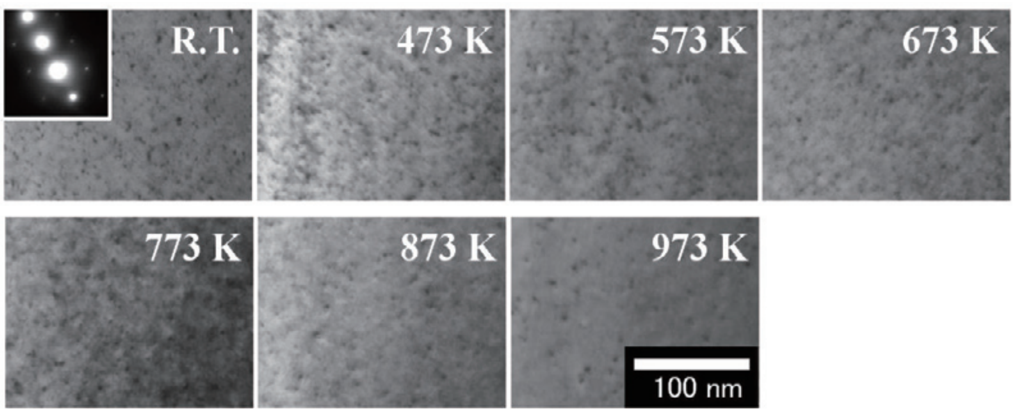

Fig. 2 TEM micrographs for the single ion (D_2^+) implanted tungsten as a function of heating temperature. The electron diffraction pattern at room temperature is also shown.

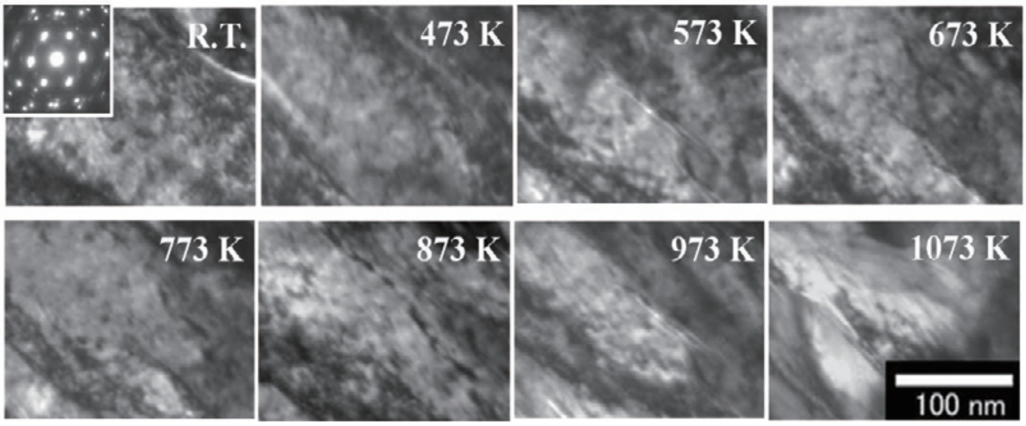

Fig. 3 TEM micrographs for the dual ions (C^+-D_2^+) implanted tungsten as a function of heating temperature. The electron diffraction pattern at room temperature is also shown.

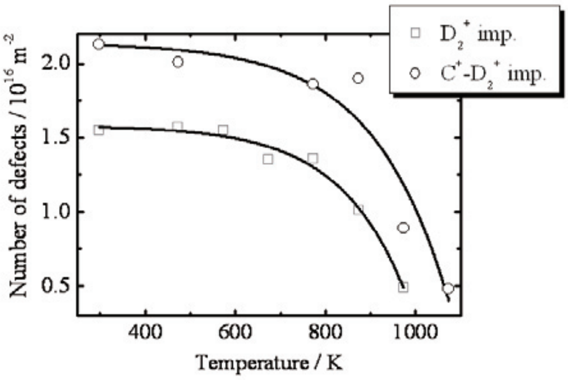

Fig. 4 Density of irradiation defects as a function of post-implantation annealing temperature

sample, while the retention of deuterium trapped by intrinsic defects was almost constant among these two samples.

From the TEM image for the samples after the implantations, it was found that there were grain boundaries but no dislocations existed. To observe the microstructure change caused by the annealing for the single ion and dual ions implanted samples, Figs 2 and 3 show the TEM images for the tungsten samples as a function of annealing temperature, including the electron diffraction pattern at room temperature. The diffraction patterns for the single ion implanted sample for the pretreated sample were similar, i.e., no disordering was observed. In contrast, the spots and halo rings were found in that for the dual ions implanted sample, indicating the formation of fine structures and amorphous near-surface layer. In the TEM images at room temperature for both the single and dual ions implanted samples, such irradiation defects as dislocations and dislocation loops appeared after the ion implantation. The density and size of the irradiation defects for the dual ions implanted sample were higher and larger than those for the single ion implanted one. Deuterium could be trapped by the dislocation and the dislocation-loops [12, 13]. Especially, the large dislocation-loop has "kink" around them. The kink held much complex dislocations. It seems that the kink would be more attractive to trap deuterium than simple dislocations. Based on the TEM data (Figs. 2 and 3), the density of the dislocation-type defects as a function of annealing temperature is shown in Fig.4. It was found that the irradiation defects like dislocations and dislocation loops started moving and remained up to 900 K. On the other hand, for the single ion implanted sample, they began clearly moving and annihilating around 800 K. It was suggested that the recovery of irradiation defects was much affected by C^+ implantation. The mean size of defects was estimated from the number of vacancy calculated by the SRIM and the density of defects for each samples. Because the interstitial atom and the vacancy are produced as a pair during ion implantation, the number of vacancies and that of interstitial atoms should be the same. The interstitial atoms get together forming dislocation-loops. The average for the number of tungsten atoms per one visible dislocation-type defect was estimated by the vacancy number and the defects density. The ratio of the average for each sample was regarded as the ratio of defects size. This result showed that the size for the dual ions implanted sample was 16 times larger than that for the single ion implanted sample. Hence, it was thought that the recovery of larger defects required the higher temperature.

When the TDS results are confronted with the TEM data, it is apparent that, the deuterium desorption from ion-induced defects and the initiation temperature of the defects recovery for the dual ions implanted sample were about 100 K higher than those for the single ion. Therefore, enlargement of the defects size caused by the C^+ implantation means that the surroundings of the defects would become complex, and the high temperature was required for the desorption of deuterium. Consequently, the recovery of irradiation defects would be yielded the shift to the higher temperature.

4. Conclusion

TDS experiments were carried out to investigate desorption of deuterium, and the TEM observations performed to observe the recovery of irradiation defects. From the TDS experiments, the total deuterium retention for the dual ion implanted sample was about four times as large as that for the single ion implanted sample. Especially, the retention of deuterium trapped by ion-induced defects increased for the dual ions implanted sample, and the temperature region of the deuterium desorption for the dual ions implanted sample was shifted toward higher temperature by about 100 K compared to that for the single ion implanted sample.TEM observations showed that the density and size of irradiation defects for the dual ions implanted sample were higher and larger than that for the single ions implanted one. For the dual ions implanted sample, the formation of fine structures and amorphous were found and initiation temperature of defects recovery for the dual ions implanted was about 100 K higher than that for the single one.

From those experimental results, a synergism of the dual ion implantation on the desorption of deuterium and the recovery of irradiation defects arose from that the surrounding states of irradiation defects.

Acknowledgement

This study has been supported by NIFS collaboration program No. NIFS07KOBA020, JSPS Kakenhi (No. 19686055 and 19055002) from MEXT, Japan, the Institute of Applied Mechanics, Kyushu University and the Center for Instrumental Analysis at Shizuoka University.

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