富山大学水素同位体科学研究センター研究報告29・30：65－70，2010.

Effect of simultaneous implantation on deuterium retention in tungsten

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Deuterium retention in tungsten irradiated simultaneously with deuterium, helium and carbon ions was investigated by TDS. Effect of irradiation pre-damage on the deuterium retention was examined by microstructure observation with taking account of the TDS results. It was found that deuterium was preferentially trapped by intrinsic defects for simultaneous C^+ and D_2^+ implanted tungsten. The deuterium trapping by ion-induced defects was enhanced by the ion implantation.

In the case of simultaneous implantation with He^+, the deuterium retention has largely changed. Especially, in the case of He^+ and D_2^+ simultaneous implantation, the deuterium retention increased compared to the sequential implantation. However, the triple ions (He^+-D_2^+-C^+) implantation, the deuterium retention was almost the same as that for the only D_2^+ implanted tungsten, indicating the dynamic desorption would be enhanced.

Keywords: tritium retention, plasma-surface interactions, tungsten, simultaneous implantation

I. Introduction

Plasma facing materials play an key role in maintaining high purity D-T plasma and their selections are quite important. Recently, the combination of several different materials as plasma facing materials for the first wall, divertor and baffles was deemed to be one of the best solutions [1-3]. Therefore, the assessment of tritium retention under simultaneous ions implantation circumstance is one of the critical issues. Tungsten is considered as one of the candidates for ITER divertor components because of its low sputtering yield and low tritium retention [1-4]. In addition, carbon is considered to be used for high heat load regions of the plasma facing components.

To predict hydrogen isotope recycling, including tritium, it is important to elucidate the effects of simultaneous implantation of carbon and/or helium on hydrogen isotope retention and/or recycling for tungsten. The sputtering yield of W under implantation with C and D ions as a function of the C fraction in the incident flux has been studied and it was shown that deuterium retention is limited by the number of available traps [5]. Besides, implanted carbon exists in several chemical forms in tungsten. At higher temperatures, WC layer is formed [6], which was confirmed by chemical shift of XPS [7]. In addition, the implantation of helium produced by the D-T nuclear reaction will introduce damages and/or bubbles into the tungsten, indicating that the deuterium retention is also expected to change upon simultaneous He^+ irradiation. In the present study, the deuterium retention behaviors for various ion implantation conditions were studied and simultaneous implantation effect on deuterium retention is discussed. The correlation between these retention behaviors and microstructure change is also discussed.

II. Experimental

Stress-relieved tungsten samples with size of 10 mm in diameter and 0.5 mm in thickness were used. Prior to implantation, the samples were polished mechanically to mirror finish surfaces and pre-heated at 1173 K for 10 minutes in vacuum to remove surface impurities and damages induced by the polishing process. The triple ions implantation system has been set up at Shizuoka University. This system also features a TDS chamber which is connected with an implantation chamber through a gate valve via a load lock chamber. CO_2 gas was used as a C^+ source to prevent hydrogen impurity contamination. A E×B mass separator was installed at the head of the C^+ gun. 10 keV C^+, 3 keV D_2^+ and 3 keV He^+ ions were independently controlled. The D_2^+ flux was set to be 1×10^{18} D m^{-2} s^{-1} and its fluence was reached to be 1×10^{22} D m^{-2}. The angles of incidence with respect to the surface normal were 0 degrees for the C^+ ions and 15 degrees for the others. The implantation area was set to 4 mm × 4 mm. The sample holder is equipped with a ceramic heater to heat the samples up to 1300 K. The ion implantation experiment was done at room temperature. After an ion implantation, the sample was transferred into an TDS chamber. The sample was heated up to 1173 K with a heating rate of 0.5 K s^{-1}, and Desorbed gaseous molecules were detected by quadruple mass spectrometers. The TEM observations (JEM 2000EX, JASCO Inc.) were also performed at the Institute of Applied Mechanics, Kyushu University to analyze the microstructure change by various ion implantations. The depth profiles of D and C were analyzed by GD-OES at University of Toyama.

III. Results and discussion

Figure 1 shows the D_2 TDS spectra for tungsten simultaneously implanted with C and D ions. The C^+/D^+ flux ratio was fixed at 0.2, 1, and 2. In the case of C^+/D^+ = 0.2, the TDS spectrum was clearly different from other spectra. The TDS spectrum was characterized by desorptions peaks at ~500 and ~800 K with a shoulder at ~950 K.; Note that this TDS spectrum is similar to the spectrum observed for tungsten sequentially implanted with C and D ions. For the C^+/D^+ = 1 and 1.8 cases, the higher desorption stage at ~ 800 K disappeared and only one large desorption peak was found at ~ 350 -500 K, which was similar to the desorption observed for tungsten.

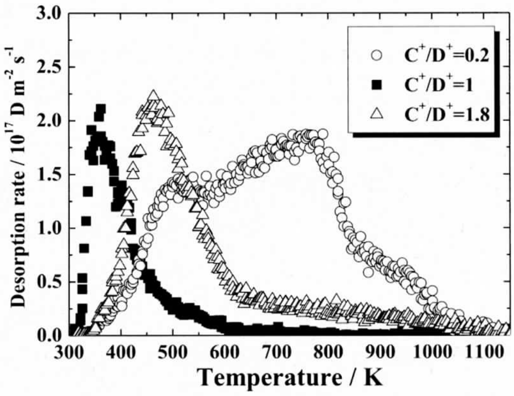

Fig.1 The D_2 TDS spectra for simultaneously implanted tungsten with D and C ions with various C^+/D^+ flux ratio.

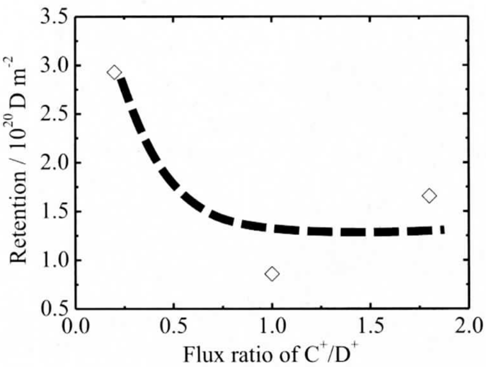

Fig.2 Summary of deuterium retention for the simultaneously implanted tungsten with various C^+/D^+ flux ratio.

The deuterium retentions for the simultaneously implanted tungsten with various C^+/D^+ flux ratios are summarized in Fig. 2. It was clear that the highest deuterium retention was achieved for the sample with C^+/D^+ flux ratio of 0.2. The data was scattered as increasing the C^+/D^+ flux ratio. However, we believe that the deuterium retention would be converged above 10^{17} D m^{-2} by increasing the C^+/D^+ flux ratio. Fig. 3 shows microstructure changes caused by isochronal annealing (10 min.) for the C^+-D_2^+ simultaneous implanted sample. It was found that the dislocations and the dislocation loops appeared after the ion implantation. Microscopic damage at matrix was gradually annihilated around 973 K and most of damage was annihilated at 1073 K. However, the dislocations existed at 1073 K. The electron diffraction pattern for C^+-D_2^+ implanted tungsten after annealing at 1073 K (Fig. 4) was clearly different from the original tungsten indicating the formation of a WC layer after the simultaneous implantation.

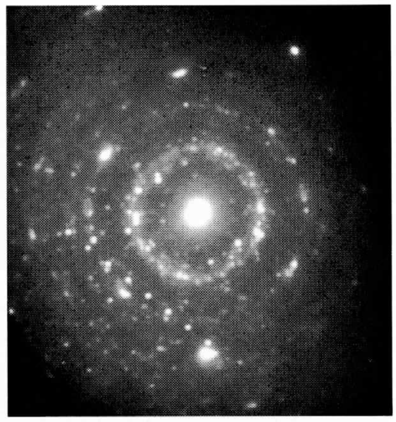

Fig. 4 The diffraction pattern for the C^+-D_2^+ simultaneous implanted sample after annealing at 1073 K.

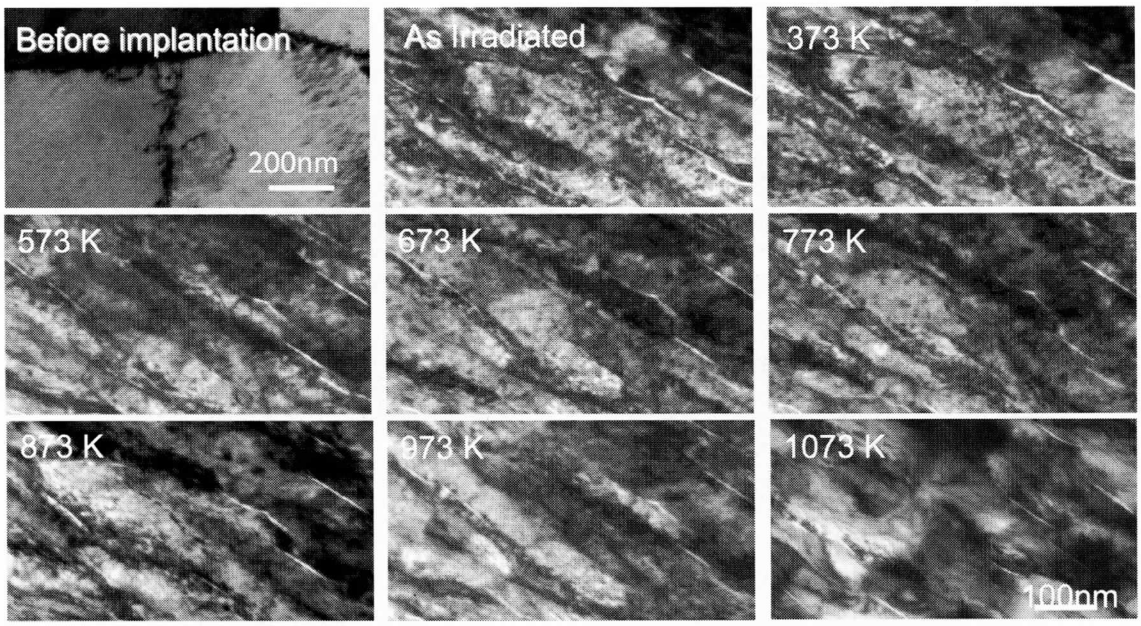

Fig. 3 Microstructure change caused by isochronal annealing for the C^+-D_2^+ simultaneous implanted sample.

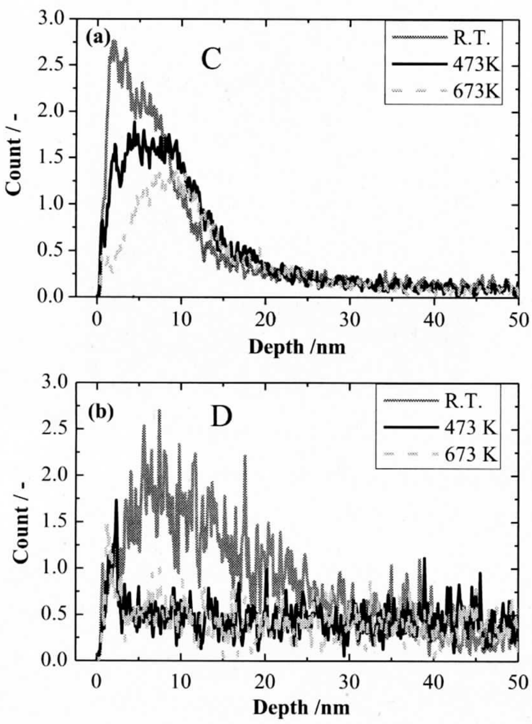

Fig. 5 The depth profiles of C and D for tungsten simultaneously implanted with C^+ and D_2^+ at various temperatures.

The depth profiles of C and D after various implantation temperatures were observed by GD-OES. As shown in Fig. 5, carbon was aggregated near the surface region around 20 nm,

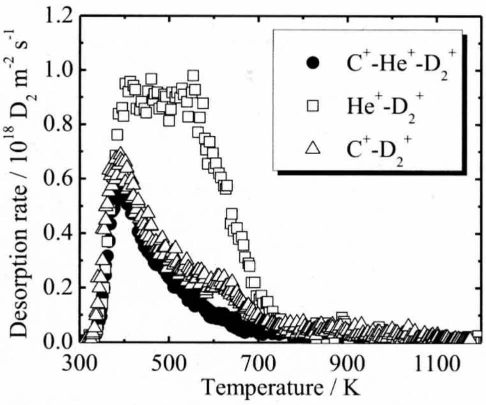

Fig. 6 The D_2 TDS spectrum for triple ions (C^+-He^+-D_2^+) implanted tungsten. The TDS spectra for the dual ions (He^+-D_2^+) and (C^+-D_2^+) implanted tungsten samples were also shown for comparison.

which is almost consistent with the estimation by SRIM code. The carbon concentration near the surface region decreased at temperature of 673 K, indicating the dynamic desorption toward the outside of the sample. The amount of retained deuterium decreased as the implantation temperature increased. These results show that the dynamic desorption as CD_x would be occurred.

To evaluate helium implantation effect, triple

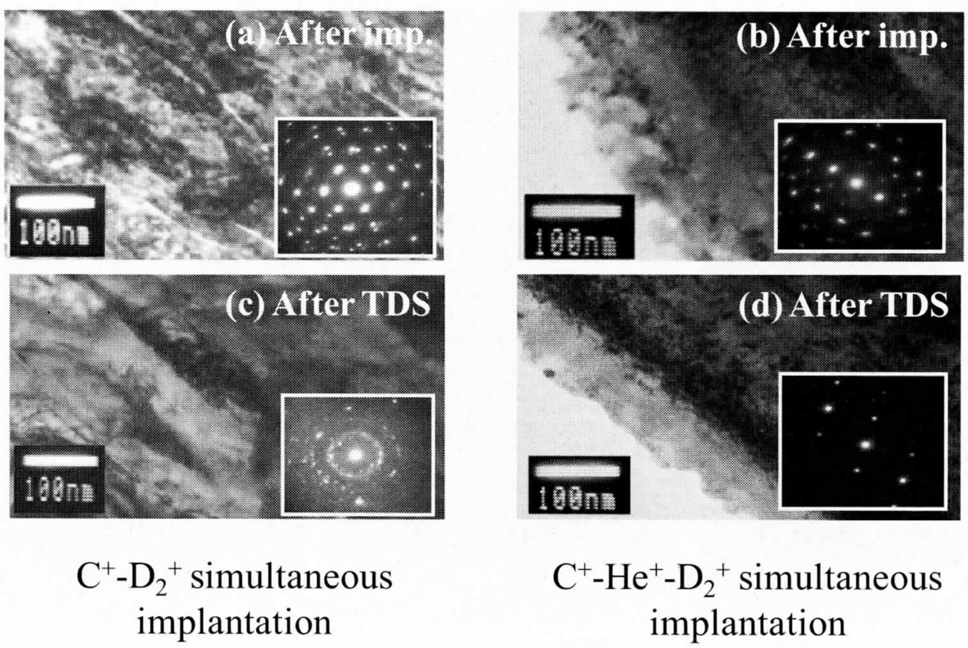

Fig. 7 TEM micrographs for C^+-D_2^+ and C^+-He^+-D_2^+ simultaneous implanted tungsten samples after ion implantation and TDS experiment

ions (C^+, He^+ and D_2^+) implantation was performed. Fig. 6 shows the D_2 TDS spectra for triple ions implanted sample with the flux ratio of C^+:He^+:D^+:=0.2:0.2:1. The D_2 TDS spectra for the He^+-D_2^+ (He^+:D^+=0.2:1) and C^+-D_2^+ (C^+:D^+=0.2:1) simultaneous implanted samples were also shown for comparison. It was found that the D_2 desorption temperature was concentrated in lower temperature side less than 1100 K. The deuterium retention by dual ions implantation of C^+-D_2^+ and triple ions were almost the same. However, the deuterium retention by He^+-D_2^+ implantation was about 2.5 times as large as those by triple ions implantation and C^+-D_2^+ dual ions implantations. These results indicate that the dynamic desorption as CD_x during the triple ions implantation reduces the deuterium retention. Fig. 7 summarizes TEM micrographs for the C^+-D_2^+ and C^+-He^+-D_2^+ simultaneous implanted tungsten samples after ion implantation and TDS experiment. It has been found that He bubble was formed in the C^+-D_2^+ simultaneous implanted tungsten and this bubbles were aggregated by heating. The diffraction pattern after TDS experiment was quite different among these samples. The different structure was formed for the dual ion implanted sample, although the no clear change of structure by triple ions implantation was found for the C^+-He^+-D_2^+ simultaneous implanted tungsten. These results show that the dynamic trapping and detrapping of deuterium by simultaneous ion implantation would be occurred. Therefore, additional experiments will be required for the elucidation of tritium dynamics at the tungsten surface.

IV. Conclusions

The deuterium retentions for tungsten with simultaneous ion implantations were evaluated by TDS. Effects of irradiation damage on deuterium retention was also evaluated by microstructure observation with taking account of TDS results. It was found that the deuterium was preferentially trapped by intrinsic defects including grain boundary for simultaneous C^+ and D_2^+ implanted tungsten. The deuterium trapping by ion-induced defects was enhanced by the ion implantation.

In the case of simultaneous implantation with He^+, the deuterium retention has largely changed. Especially, in the case of He^+ and D_2^+ simultaneous implantation, the deuterium retention increased compared to the sequential implantation. However, the triple ions (He^+-D_2^+-C^+) implantation, the deuterium retention was almost the same as that for the only D_2^+ implanted tungsten, indicating the dynamic desorption would be enhanced.

Acknowledgements

This study was supported by a Grand-in-aid for scientific research in fusion by Ministry of Education, Science and Culture, Japan, by the joint usage/research program of the Hydrogen Isotope Research Center, University of Toyama (No.HRC2009-02 &03) and by the Center for Instrumental Analysis at Shizuoka University.

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