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Ion fluence dependence on chemical behavior of energetic deuterium implanted into oxygen-contained boron film

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Abstract

Ion fluence dependence on chemical behavior of energetic deuterium implanted into oxygen-contained boron and pure boron films was investigated by XPS and TDS. It was found that large amount of D implanted into the oxygen-contained sample was quickly desorbed as D_2O by chemical sputtering during D_2^+ implantation. The D retention for both samples increased as the D_2^+ fluence increased. The implanted D was preferentially trapped by O with forming O–D bond, and thereafter by B with forming B–D bond. These facts indicate that the stability of D bound to O is higher than that bound to B. It was concluded that the chemical sputtering of oxygen by energetic particles and the chemical stability of O–D bond should be taken into consideration for the evaluation of tritium inventory in the oxygen-contained boron film.

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Keywords: Boronization; Plasma facing materials; Thermal desorption; Tritium retention; Wall conditioning

1. Introduction

Boronization is considered as one of the most effective conditioning techniques for first walls in many plasma testing devices because of remarkable improvement of their plasma performances [1–3]. It is well known that boron can easily trap various impurities such as oxygen and carbon, which could make fusion plasma unstable. Thus, it is predicted that oxygen which is one of the typical impurities would be contained in the boron film coated on the first wall during boronization [4–7] and this oxygen-contained boron film will be exposed to energetic tritium, deuterium and helium. Therefore, it is important from the viewpoint of tritium safety to elucidate the trapping and desorption behaviors of tritium in order to estimate tritium inventory in oxygen-contained boron films.

In our previous study, the chemical behavior of energetic deuterium (D) implanted into the oxygen-contained boron films with oxygen concentration of 11%, 37% and 39% was studied [8]. For the sample with oxygen concentration of 11%, it has already been reported that the oxygen was

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dissolved into the boron film [9]. However, B_2O_3 was formed in the sample with oxygen concentration of around 37%. It was also found that the D retention significantly decreased as oxygen concentration increased. The trapping behavior of D retained in the sample was changed compared to the pure boron sample and large amount of D was preferentially trapped by oxygen. It can be said that the existence of B_2O_3 resulted in the D trapping with forming O–D bond [8]. It is also noted that the oxygen concentration of 20%, which would be a threshold concentration for the oxide (B_2O_3) formation, may be a key factor for the elucidation of trapping and retention behaviors of D in boron film. In the present study, therefore, the deuterium ion (D_2^+) fluence dependence on the chemical behavior of energetic D implanted into the oxygen-contained boron film with oxygen concentration of approximately 20% was investigated by means of X-ray Photoelectron Spectroscopy (XPS) and Thermal Desorption Spectroscopy (TDS). These behaviors for the oxygen-contained boron film were compared to those for the pure boron film.

2. Experimental

The oxygen-contained boron and pure boron films, namely Sample A and Sample B, were, respectively, deposited on silicon substrate using the Plasma Chemical Vapor Deposition (P-CVD) apparatus at Shizuoka University as described in our previous paper [10]. Before a P-CVD process, a helium discharge was performed for 30 min to clean the film surface. Thereafter, the oxygen-contained boron film was deposited using decaborane ($B_{10}H_{14}$) vapor, oxygen gas and helium carrier gas with the flow rates of 2.5, 0.1 and 5.7 sccm. The pure boron film was also prepared only using the decaborane ($B_{10}H_{14}$) vapor diluted by helium carrier gas with the same flow rate above. The substrate temperature was ambient temperature during the process and the boron sample with the thickness of 150 nm was achieved. From the XPS (ESCA 1600 Series, ULVAC-PHI Inc.) measurements using Al Kα as X-ray source, it was found that the atomic concentrations of boron, oxygen, carbon and nitrogen for Sample A were 68%, 22%, 6% and 4%, and those for Sample B, 93%, 3%, 3% and 1%, respectively.

After the preparation of the samples, they were preheated at 993 K for 10 min under an ultrahigh vacuum of $\sim 10^{-8}$ Pa, and then D_2^+ was implanted into the film with an energy of 1.0 keV and the flux of 1.0×10^{18} D^+ m^{-2} s^{-1} at ambient temperature. The D_2^+ fluence was changed between 5.0×10^{20} and 1.0×10^{22} D^+ m^{-2}. The TDS measurements were also carried out from ambient temperature up to 993 K with the heating rate of 0.5 K s^{-1}. The XPS measurements were performed at every experimental step.

3. Results and discussion

Fig. 1 shows the B-1s XPS spectra for Sample A and Sample B as a function of D_2^+ fluence. It was found that the peak position for Sample A was located at 188.3 eV whereas that for Sample B was located at 187.7 eV before D_2^+ implantation. These peaks for both samples were attributed to B–B bond [8]. A broad peak located at 192 eV for Sample A was also observed, which indicates the formation of B–O bond as boron oxide [11]. The chemical shift of B-1s by D_2^+ implantation was not observed at any fluence for both samples.

The D_2 TDS spectra for both samples after D_2^+ implantation with 1.0×10^{22} D^+ m^{-2} are shown in Fig. 2. It was found that the D retention in Sample A was much smaller than that in Sample B. These TDS spectra were analyzed by Gaussian distribution function to determine the D trapping states in both samples. It was indicated that all the D_2 desorption consisted of three stages: 400, 520 and

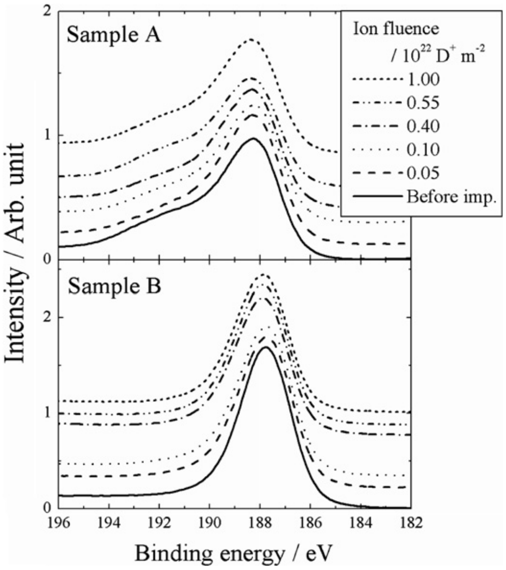

Fig. 1. The B-1s XPS spectra for Sample A and Sample B as a function of D_2^+ fluence.

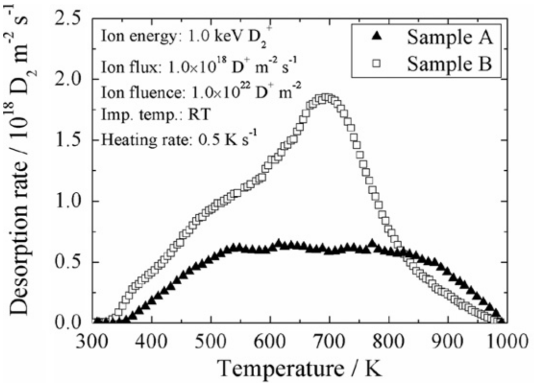

Fig. 2. The D_2 TDS spectra for Sample A and Sample B after D_2^+ implantation with the D_2^+ fluence of 1.0×10^{22} D^+ m^{-2}.

700 K for Sample B and 520, 700 and 860 K for Sample A. The first stage for Sample B was attributed to the desorption of D trapped in the interstitial site. The other two stages would be corresponded to the desorption of D with forming B–D–B and B–D bonds for Sample B. For Sample A, their peaks were the D desorption from B–D–B, B–D and O–D bonds, respectively [8,12,13]. Fig. 3 summarizes the D retentions for both samples as a function of D_2^+ fluence. It was found that the total D retentions for both samples increased as the D_2^+ fluence increased. For Sample B, the trapping of D with forming B–D bond was dominant in any fluence. For Sample A, it was not dominant up to the D_2^+ fluence of 5.5×10^{21} D^+ m^{-2}. The D trapped by O reached almost a half of the total D retention in the D_2^+ fluence less than 5.0×10^{20} D^+ m^{-2} and become constant in the further D_2^+ implantation.

The total D retention at the D_2^+ fluence of 7.3×10^{21} D^+ m^{-2} for Sample A was estimated to be 9.4×10^{20} D m^{-2}, which is almost consistent with our previous study for the samples with the oxygen concentrations of 11 and 37% [8]. The oxygen concentration decreased after deuterium implantation for all the samples. This fact suggests that the contained oxygen could be chemically sputtered and be desorbed by forming D_2O from the sample during the implantation of energetic D [8]. The SRIM 2003 code was used in order to evaluate the amount of D desorbed as D_2O. It was found that the amount of D desorbed as D_2O was estimated to be about 5×10^{20} D m^{-2} from estimation of the amount of decreased oxygen by D_2^+ implantation. This estimation was approximately consistent with difference of the D retention between Sample A and Sample B. These facts indicate that large amount of D implanted into the oxygen-contained boron film would be quickly desorbed as D_2O and some of D was trapped by oxygen. It was suggested that existence of boron oxides in the sample would lead the trapping of implanted D by forming O–D bond.

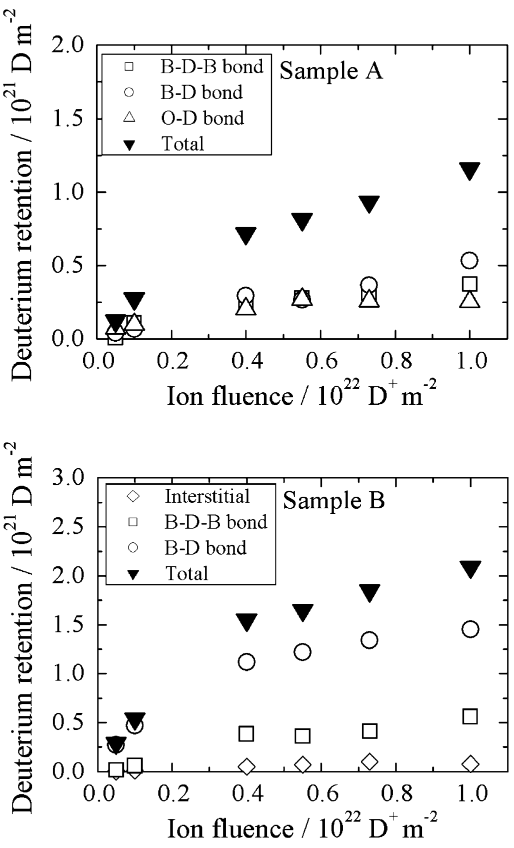

Fig. 3. The D retentions for Sample A and Sample B as a function of D_2^+ fluence.

However, the amount of O–D bond was saturated above the D_2^+ fluence of 5.5×10^{21} D^+ m^{-2}. The chemical existing states of D retained in the samples as a function of D_2^+ fluence are summarized in Fig. 4. The implanted D was preferentially trapped by oxygen with forming O–D bond for Sample A in the low D_2^+ fluence region, although that for Sample B was preferentially trapped by boron with forming B–D bond. These results indicate that the D interacts with oxygen in the initial D_2^+ implantation stage and thereafter, it is trapped by boron.

From these experimental results, it can be concluded that both of chemical sputtering of oxygen by energetic particles and chemical stability of

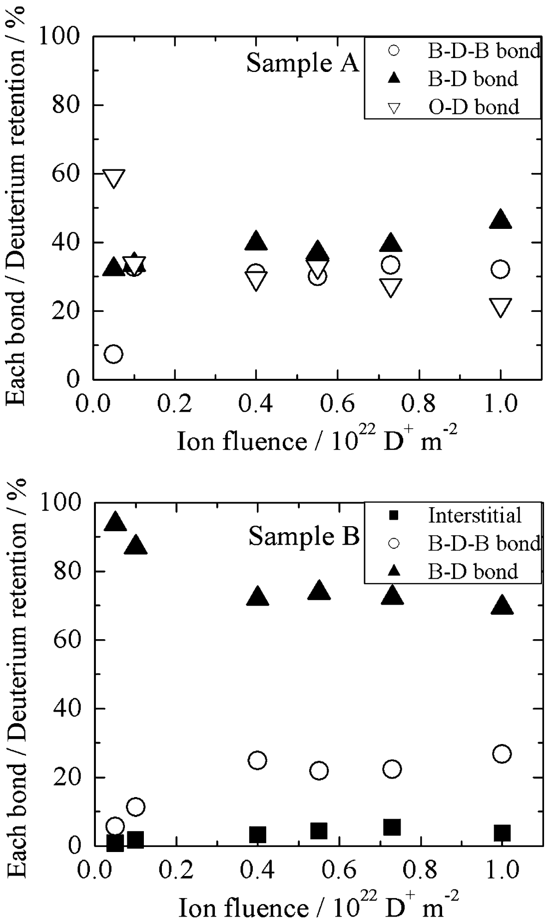

Fig. 4. The D retention of each chemical existing state as a function of D_2^+ fluence for Sample A and Sample B.

O–D bond should be taken into consideration for the evaluation of tritium inventory in the oxygen contained-boron film.

4. Conclusion

The D trapping and retention behaviors in the boron film with oxygen concentration of 20%, was investigated as a function of D_2^+ fluence using XPS and TDS. It was found that large amount of implanted D were quickly chemically sputtered as D_2O during D_2^+ implantation for the oxygen-contained film. The D retention for both of pure and oxygen-contained films increased as ion fluence increased. The implanted D in oxygen-contained boron film was preferentially trapped by oxygen with forming O–D bond, although that in pure boron film was preferentially trapped by boron with forming B–D bond. These facts indicate that stability of D bound to oxygen is higher than that bound to boron. It was concluded that the chemical stability of O–D bond should be taken into consideration for the evaluation of tritium inventory in the oxygen-contained boron film.

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