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Effects of helium implantation on hydrogen isotope retention behavior in SiC

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Abstract

To elucidate the interaction between hydrogen isotopes and damaged structures in SiC, deuterium ion (D_2^+) was implanted into helium ion (He^+) implanted SiC. The chemical state of SiC was analyzed by XPS and thermal desorption behaviors of D_2 and He were observed by TDS. It was found that D desorption consisted of two stages, namely D bound to Si and that to C. The D was desorbed from both desorption stages for He^+ implanted SiC with the low D_2^+ fluence, although it was preferentially trapped by C for pure SiC. The D retention for He^+ implanted SiC was lower than that for pure SiC under the same D_2^+ fluence. The implanted He mainly forms He blister or remains in the carbon vacancies. The D trapping efficiency was changed by He^+ implantation, although the D trapping mechanism was not largely influenced.
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1. Introduction

Silicon carbide (SiC) is one of the most attractive materials for fusion first wall and/or structural materials due to its minimal activation, good thermal conductivity and thermal stability [1–4]. One of the critical issues for the usage of SiC is that lots of hydrogen isotopes including tritium were retained in a narrow surface layer of the implantation range if a few keV hydrogen isotope ions were implanted into SiC. In addition, high energetic helium (He) produced by D–T reaction is thought to interact with the fusion materials and may lead to increase of T retention. Therefore, it is important to elucidate detailed mechanism for hydrogen isotope trapping and detrapping from the viewpoint of fusion safety.

In our previous papers [5,6], the hydrogen isotope exchange behavior, and the retention and re-emission behaviors of hydrogen isotopes in SiC

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have been reported. The D and H retentions in SiC have been evaluated. The hydrogen isotope trapping and detrapping behaviors in pure SiC have been also reported that the D desorption consists of two stages, namely desorption stages of D bound to Si and C [7]. The behavior of damaged structures by He^+ implantation has been also studied [8]. Some experimental results have also been reported on hydrogen isotope desorption behavior for He^+ implanted SiC [9,10] and He^+ implantation effects for various SiC materials have been studied well [11,12]. However, detailed mechanisms for hydrogen isotope trapping and detrapping in He^+ implanted SiC have been not clearly elucidated. In this study, D_2^+ was implanted into He^+ implanted SiC and the chemical state of SiC was evaluated by the X-ray photoelectron spectroscopy (XPS) and the thermal desorption behaviors of D and He were studied by the thermal desorption spectroscopy (TDS). These behaviors for He^+ implanted SiC were discussed with taking those for pure SiC into consideration.

2. Experimental

The sample used in this study was a polycrystalline silicon carbide (β-SiC) wafer (ROICERAM-HS) supplied by Asahi Glass Co. Ltd. with a size of $\phi 10 \times 0.5\ mm^3$ and density of $3.1\ g/cm^3$. To remove residual hydrogen and impurities, the sample was heated up to 1300 K in a ultra-high vacuum chamber with a vacuum below 1×10^{-6} Pa for 10 min. After cooling the sample, 1.3 keV He^+ was implanted into SiC with an ion flux of $1.3 \times 10^{18}\ He^+\ m^{-2}\ s^{-1}$ and an ion fluence of $1.0 \times 10^{22}\ He^+\ m^{-2}$ at the room temperature. Thereafter, 1.0 keV D_2^+ implantation experiment was performed with an ion flux of $1.3 \times 10^{18}\ D^+\ sm^{-2}\ s^{-1}$ and an ion fluence between $1.3 \times 10^{21}\ D^+\ m^{-2}$ and $1.0 \times 10^{22}\ D^+\ m^{-2}$. The chemical states of Si and C for the SiC sample were evaluated by XPS (ESCA1600 system, ULVAC-PHI Inc.) [7]. To evaluate the D_2 and He desorption behaviors and their retentions, the TDS experiment was performed at a heating rate of $0.5\ K\ s^{-1}$ and at temperature up to 1300 K.

3. Results and discussion

In our previous study [7] and other reports [13,14], the TDS spectrum of D_2 for pure SiC consists of two desorption stages at the temperature of 800 K and 1000 K, which corresponds to the desorption of D bound to Si and C, respectively. Fig. 1(a) shows TDS spectra of D_2 for He^+ implanted SiC after 1.0 keV D_2^+ implantation with various D_2^+ fluence. It was found that the D desorption for both stages were observed even if the D_2^+ fluence was low. This desorption behavior was quite different from that for pure SiC. Only high temperature desorption stage was observed in the D_2^+ fluence below $0.13 \times 10^{22}\ D^+\ m^{-2}$ for pure SiC and it was almost saturated in the fluence less than $0.2 \times 10^{22}\ D^+\ m^{-2}$. After its saturation of D bound to C for pure SiC, the 1st desorption peak, which corresponds to the desorption of D bound to Si, has begun to increase and become the major desorption stage. For He^+ implanted SiC, both D desorption stages were observed even if the D_2^+ fluence was low ($0.13 \times 10^{22}\ D^+\ m^{-2}$). In the TDS spectra of He as shown in Fig. 1(b), a large sharp peak was observed at temperature around 1200 K. This desorption stage would correspond to that retained as He blister in SiC, which is almost consistent with the previous report [12]. A small broad peak was also found at temperature between 400 K and 800 K. These intensities decreased as the D_2^+ fluence increased. Therefore, the retained He would interact with the following D_2^+ implantation and be detrapped by collision processes. The D retentions (D/SiC) for pure SiC and He^+ implanted SiC as a function of D_2^+ fluence were summarized in Fig. 2. The He retention for He^+ implanted SiC was also shown in Fig. 2(b). The experimental error for the estimation of the retention was almost 10%. For the estimation of D retention, the concentration of saturated D retained with 1.0 keV D_2^+ implantation was assumed to be the same as that with 4.0 keV D_2^+ implantation and calibrated by comparing the TDS data obtained by pure SiC with 1.0 keV D_2^+ implantation of $1.0 \times 10^{22}\ D^+\ m^{-2}$ and the ERD data obtained by pure SiC with 4.0 keV D_2^+ implantation of $1.1 \times 10^{22}\ D^+\ m^{-2}$ [6]. Although D/SiC for pure SiC was almost saturated to be 0.75 in D_2^+ fluence over $0.5 \times 10^{22}\ D^+\ m^{-2}$, that for He^+ implanted SiC was reached only 0.52 and was still increasing. For the He retention, it decreased quickly in D_2^+ fluence between $0.25 \times 10^{22}\ D^+\ m^{-2}$ and $0.5 \times 10^{22}\ D^+\ m^{-2}$. These facts indicate that the implanted D interacted with retained He and D trapping efficiency was suppressed.

The peak positions of C 1s and Si 2p for He^+ implanted SiC were summarized in Fig. 3. The summary for pure SiC was also shown in Fig. 3(a) for comparison. The peak position of Si 2p was quickly

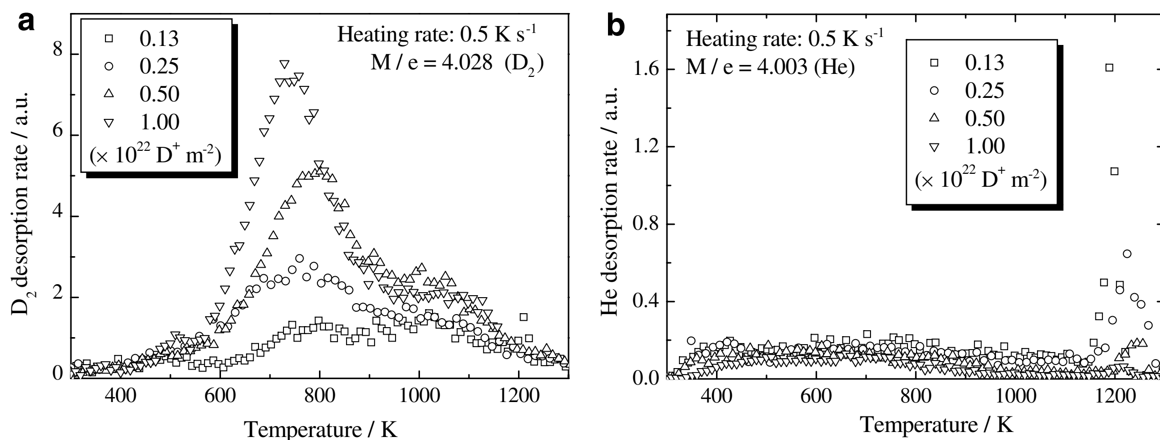

Fig. 1. TDS spectra of (a) D_2 and (b) He for He^+ implanted SiC with various D_2^+ fluences.

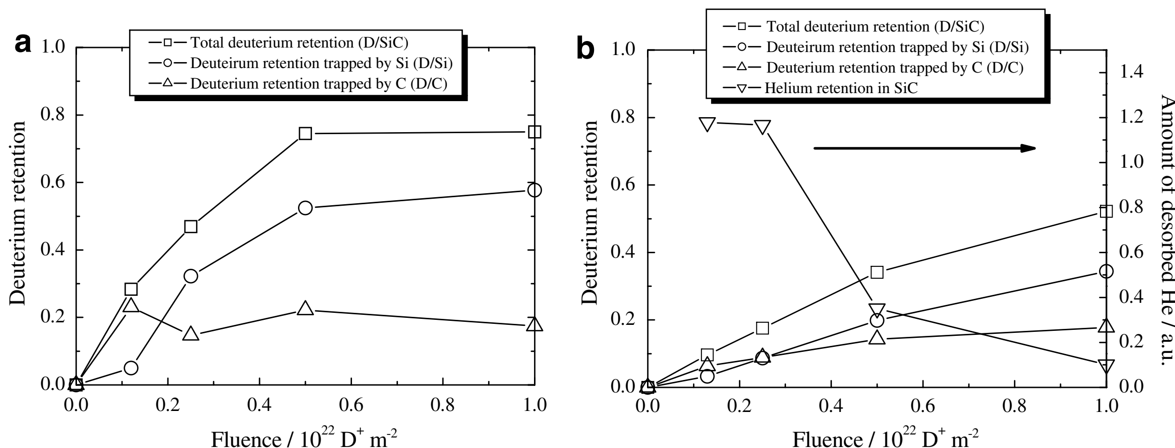

Fig. 2. D and He retentions in (a) pure SiC and (b) He^+ implanted SiC as a function of D_2^+ fluence.

shifted to the lower energy side by He^+ implantation and remained almost constant for further D_2^+ implantation. For pure SiC, negative peak shift was observed in the initial D_2^+ implantation. However, the peak position of Si 2p for pure SiC after D_2^+ implantation was slightly higher than that for He^+ implanted SiC. This difference would be caused by the interaction between Si and D. Contrary to the behavior of Si 2p, that of C 1s was shifted to the higher energy side by D_2^+ implantation. Comparing these behaviors for He^+ implanted SiC with pure SiC, the peak position of C 1s for He^+ implanted SiC was about 0.2 eV smaller than that for pure SiC after D_2^+ implantation and positive peak shift of C 1s for He^+ implanted SiC was delayed. A large peak shift was not observed in the low D_2^+ fluence region less than 0.2×10^{22} D^+ m^{-2}. Therefore, the chemical state of C 1s was also influenced by He^+ implantation.

From these experimental results, the interaction between hydrogen isotopes and damaged structures in SiC is discussed. In this study, D_2^+ and/or He^+ with high energy were introduced into SiC, where they would migrate and interact with SiC structure. It is known that the hydrogen isotopes were trapped by interstitial sites and vacancies. The stability of hydrogen isotopes trapped by the vacancies is higher than that in interstitial sites because D_2^+ was energetic, like 'hot atoms' and the activation energies of D estimated from the TDS spectra were much higher than those of D trapped by interstitial sites [7]. Therefore, following discussion is focused

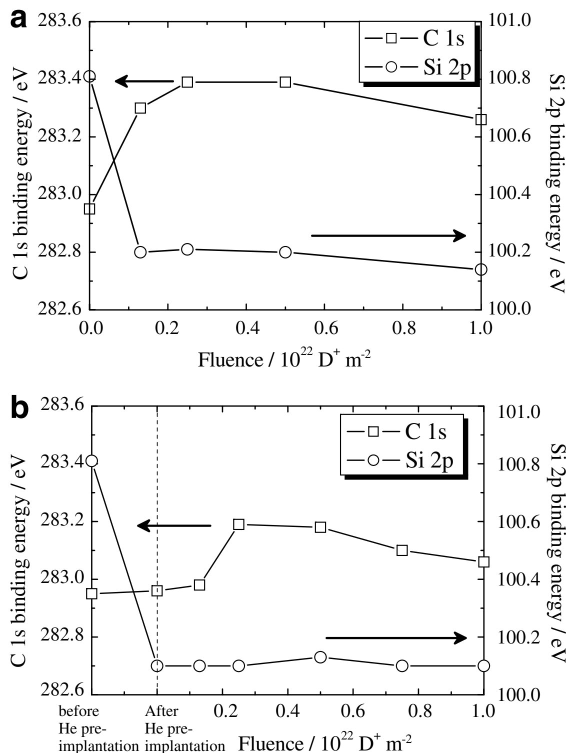

Fig. 3. Summary of XPS peak position of C 1s and Si 2p for (a) pure SiC and (b) He^+ implanted SiC as a function of D_2^+ fluence.

on interaction of hydrogen isotopes with the damaged structures produced by the ion implantation in SiC.

According to the TDS results, the D trapping behavior for pure SiC was different from that for He^+ implanted SiC. Although the D was preferentially trapped by C for pure SiC in the low D_2^+ fluence region, that for He^+ implanted SiC was trapped by both of C and Si. These results indicated that D interacted with C, namely silicon vacancies in the initial D_2^+ implantation stage and thereafter, it was trapped by Si, namely carbon vacancies. For He^+ implantation, the damaged structures, namely silicon and carbon vacancies were introduced before the D_2^+ implantation and D was trapped by both of C and Si even if the D_2^+ fluence was low. However, the implanted He was remained in SiC with forming He blister or retaining in carbon vacancies and latter He would mainly prevent the D trapping by Si. It can be said that SiC structure would be amorphized and some disordered C would exist in SiC, which has been pointed out in the previous report [13]. However, it seems that this structure change does not largely influence the hydrogen trapping site because D desorption stages for He^+ implanted SiC was the same as that for pure SiC and only D trapping efficiency and its behavior were changed. These disordered C produced by the ion implantation would migrate and desorbed as hydrocarbons form [8], which is consistent with the previous report that only hydrocarbon was desorbed from SiC after D_2^+ implantation and no silane was formed [14,15].

It can be concluded that implanted He mainly forms He blister or remains in the carbon vacancies by He^+ implantation. This He^+ implantation induces the production of disordered C in SiC, indicating the SiC would be amorphized. The D trapping efficiency and behavior were changed by He^+ implantation, although the D trapping mechanism was not largely influenced. The D interacts with the He trapped in carbon vacancies and desorbed without interacting with SiC in the initial D_2^+ implantation. Therefore, the D retention was reduced in contrast to that for pure SiC.

Further study on He^+ post implantation or He^+–D_2^+ simultaneous implantation study will reveal the total hydrogen isotope retention behavior in SiC.

4. Conclusion

For the estimation of hydrogen isotope behavior and elucidation of its interaction mechanism with damaged structures in SiC, the D_2^+ were implanted into He^+ implanted SiC. The chemical states of Si and C were studied by XPS and thermal desorption behavior of D was analyzed by TDS. These results were compared with those for pure SiC.

It was found that D desorption consisted of two stages, namely D bound to Si at low temperature stage and that to C at high temperature one. The D was desorbed from both desorption stages for He^+ implanted SiC with the low D_2^+ fluence, although it was preferentially trapped by C for pure SiC. The D retention for He^+ implanted SiC was lower than that for pure SiC under the same D_2^+ fluence. The implanted He mainly forms He blister or remains in the carbon vacancies by He^+ implantation. This He^+ implantation induces the production of disordered C in SiC, indicating the SiC would be amorphized. The D trapping efficiency and behavior were changed by He^+ implantation, although, the D trapping mechanism was not largely influenced. The D interacts with the implanted He and desorbed without interacting with SiC in the initial D_2^+

implantation. Therefore, the D retention was reduced in contrast to that for pure SiC.

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