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# Deuterium retention of ferritic steel irradiated by energetic hydrogen ions

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# ABSTRACT

Ferritic steel (F82H) was irradiated by energetic hydrogen ions to make a irradiation damage. The damaged ferritic steel was irradiated by deuterium ions with energy of 1.7 keV in order to investigate the retention and desorption behavior of deuterium. The deuterium ion fluence was taken up to  $5 \times 10^{18} \text{ D/cm}^2$ . The amount of retained deuterium increased as the deuterium ion fluence and saturated at  $1 \times 10^{18} \text{ D/cm}^2$ . It was clearly seen that the amount of retained deuterium increased as the damage caused by the hydrogen ion irradiation. For example, the amount of retained deuterium in the sample with 4 dpa was several times larger than that in the sample without damage. It was also seen that the desorption temperature approximately 70 K increased compared with that without damage. These results suggest that the number of trapping site and trapping energy for fuel hydrogen increases owing to the irradiation damage. The present results show that both fuel hydrogen recycling and in-vessel tritum inventory increases by the irradiation damage.

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#### 1. Introduction

Low activation ferritic steel is one of candidate materials for first walls and blanket structure components in demonstration reactors. Energy confinement of fusion plasma and in-vessel tritium inventory depend on fuel hydrogen retention of first walls. Fuel hydrogen retention of low activation materials without damage by ion irradiation has been investigated for ferritic steel, vanadium alloy and SiC/SiC composite so far [1]. Fuel hydrogen (deuterium) retention of ferritic steel (F82H) was investigated and compared with that of stainless steel, 316L SS [2]. The amount of retained deuterium in F82H was lower than that of 316L SS, and the desorption temperature of retained deuterium was smaller than that of 316L SS. F82H was also employed as first walls in [FT-2M [3,4]. The inside of vacuum vessel was fully covered with the ferritic plates. The effect of ferromagnetism on plasma production, control, confinement and stability was investigated. No deleterious effect was observed. These results encourage an application of ferritic steel to first walls of a demonstration reactor.

The first wall in the demonstration reactor is exposed to energetic ions/neutrals and fusion neutrons. The fuel hydrogen retention of the ferritic steel may change owing to the damage caused by these particle bombardments. Thus, it is of important to investigate the effect of damage on the fuel hydrogen retention to evaluate the fuel hydrogen recycling and the in-vessel tritium inventory. In the present study, ferritic steel (F82H) is irradiated by energetic hydrogen ions with an energy of 23–28 keV at different irradiation fluences and temperatures, in order to introduce the damage at the surface. Following that, the damaged ferritic steel is irradiated by deuterium ions with an energy of 1.7 keV in order to implant the deuterium to the damaged region. Then, the retention and desorption behavior of deuterium is examined, and the influence of irradiation damage on fuel hydrogen retention is investigated by using a technique of thermal desorption spectroscopy, TDS.

### 2. Experiments

Reduced activation ferritic steel (F82H) was exposed to energetic hydrogen ions using high flux ion source (DATS) at Japan Atomic Energy Agency (JAEA). The sample size was  $20 \times$  $20 \times 1$  mm. Two or three samples were placed on the sample holder (Fig. 1). The ion energy was in the range from 23 keV to 27.3 keV. The irradiation with a pulse of 100 - 150 ms was repeated to change the ion fluence. The number of pulse was in the range from 30 to 70. The temperature of the sample during the ion irradiation was monitored by an IR camera. The highest temperature was in the range from 573 K to 973 K. The base temperature was in the range from 503 K to 623 K. Table 1 shows the ion fluence, ion energy and irradiation temperature (highest temperature) for the irradiated samples. In order to evaluate the damage in displacement per atom (dpa), the calculation was conducted using SRIM-code2003 [5]. In Fig. 2, the number of vacancy per hydrogen ion with energy of 23 keV or 27.3 keV is plotted to the depth of





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Fig. 1. Locations of samples 1–7 at sample holder.

Table 1Hydrogen ion fluence, energy and irradiation temperature for damaged samples 1 – 7.

	Fluence (10 <sup>18</sup> H/cm <sup>2</sup> )	H <sup>+</sup> energy (keV)	Irradiation temp. (K)
Sample <sup>①</sup>	0.74	27.3	973
Sample <sup>②</sup>	1.05	27.3	973
Sample <sup>③</sup>	0.53	27.3	873
Sample <sup>(4)</sup>	1.92	23.0	823
Sample <sup>(5)</sup>	0.96	23.0	623
Sample⑥	0.96	23.0	823
Sample⑦	0.48	23.0	573



Fig. 2. Number of vacancy per ion versus depth of F82H.

F82H. The damage is largest in the depth at 150 nm. For the damaged samples, the deuterium ion irradiation at a room temperature, RT, with ion energy of 1.7 keV is conducted before the TDS measurement. The damage due to the deuterium ion is also shown in Fig. 2. The projected range and maximum range of 1.7 keV deuterium ion in F82H are 8.4 nm and 40 nm, respectively. In the present study, the value at 8.4 nm was employed to calculate the dpa. The dpa at 8.4 nm is approximately  $1 \times 10^{-3} \text{ dpa}/10^{-8} \text{ cm/H}$ . The ion fluence required to make 1 dpa becomes approximately  $1 \times 10^{18}$  H/cm<sup>2</sup>. Fig. 3 shows the dpa and the hydrogen ion irradiation temperature of seven samples. Here, group 1 shows the damaged samples 1, 2 and 3. These samples were placed at the same sample holder, a top figure shown in Fig. 1. Group 2 shows the damaged samples 4 and 6. These samples were placed at the center of sample holder, a middle and a bottom figures shown in Fig. 1. The annealing effect of the hydrogen ion irradiation temperature



Fig. 3. Maximum irradiation temperature and damage in dpa for the damaged samples.

on the number of vacancy was not observed in the present irradiation temperature.

The damaged samples were irradiated by 1.7 keV deuterium ions using an ECR ion source at Hokkaido University. No damaged sample was also irradiated for the comparison. The deuterium ion irradiation temperature was RT. The deuterium ion fluence was changed and the range was from  $0.1 \times 10^{18}$  D/cm<sup>2</sup> to  $5 \times 10^{18}$  D/cm<sup>2</sup>. After the deuterium ion irradiation, the sample was transferred into the chamber of TDS analysis. The sample was heated up to 1073 K with a heating rate of 30 K/min. Outgas species and the desorption rate were measured using a quadruple mass spectrometer, QMS. It is noted that the annealing during the TDS does not affect the deuterium desorption behavior since the deuterium ion irradiation was conducted before the TDS analysis. The depth profiles of surface atomic composition before and after the deuterium ion irradiation were also examined using Auger electron spectroscopy, AES.

## 3. Results

Thermal desorption spectroscopy for the damaged samples and no damaged sample was conducted also before the deuterium ion irradiation to examine the background gas species. Outgas species were H<sub>2</sub>, H<sub>2</sub>O, CO, CO<sub>2</sub> and CH<sub>4</sub>. The amounts of desorbed H<sub>2</sub>, H<sub>2</sub>O and CO were large. After the deuterium ion irradiation, outgas species containing deuterium were observed. These are D<sub>2</sub>, HD, D<sub>2</sub>O and HDO. The amount of desorbed HDO was largest and the desorption peak was approximately 550 K. The amount of desorbed D<sub>2</sub> was secondary largest and the desorption peak was approximately 500 K. After the deuterium ion irradiation, the amounts of desorbed H<sub>2</sub>, H<sub>2</sub>O, CO, CO<sub>2</sub> and CH<sub>4</sub> were a few times decreased.

The deuterium ion irradiations with an ion fluence of  $1 \times 10^{18} \text{ D/cm}^2$  were conducted for the damaged samples 1, 2, 3, 4 and 6, and no damaged sample. The TDS analysis was conducted after the deuterium ion irradiation. The amount of retained deuterium was obtained by integrating the desorption rates of D<sub>2</sub>, HD, D<sub>2</sub>O and HDO with respect to heating time. The amounts of retained deuterium for the damaged samples 4 and 6, and no damaged samples 4 and 6, and no damaged samples 4 and 6 were placed at the center of sample holder and the irradiation temperature was same, 800 K. It is clearly seen that the amount of retained deuterium is 2.4 times increased. The damaged samples 1, 2 and 3 were irradiated by energetic hydrogen ions in the same irradiation sequence. The irradiation temperature of these samples was



Fig. 4. Amounts of retained deuterium for the samples in Group 2 (a) and the samples in Group 1 (b).

approximately same, 900–1000 K. Since the position is different, the damage is different. Fig. 4(b) shows the amount of retained deuterium versus damage in dpa. It is seen that the amount of retained deuterium has a tendency to increase as increase of dpa. These results suggest that the amount of retained deuterium significantly increases by the damage.



Fig. 6. Amount of retained deuterium versus deuterium ion fluence for the damaged sample 4 and no damaged sample.

The deuterium ion irradiation was conducted at different deuterium ion fluence. Fig. 5 shows the amount of retained deuterium to deuterium ion fluence for the damaged sample 4 and no damaged sample. The amount of retained deuterium saturates at the deuterium ion fluence of approximately  $1 \times 10^{18}$  D/cm<sup>2</sup> for both the damaged and no damaged samples. The saturated value of the damaged sample is several times larger than that of no damaged sample. Fig. 6(a) and (b) shows the thermal desorption spectra of gases containing deuterium for the damaged sample and no damaged sample, respectively. It is seen that both the desorption rate and the desorption temperature increase by the damage. The peak temperatures of HDO and D<sub>2</sub> were approximately 70 K increased by the damage. This result suggests that a number of trapping site and the trapping energy increase by the damage.

Fig. 7(a)-(c) shows the depth profiles of atomic composition for no damaged sample and damaged samples 6 before and after the deuterium ion irradiation, respectively. The content of oxygen at the surface was large in no damaged sample. The carbon content at the surface was large after energetic hydrogen ion irradiation. This carbon might have deposited during the hydrogen ion irradiation. After the deuterium ion irradiation, the surface impurity layer became very thin, less than the projected range of deuterium ion. The reduction of impurity layer is consistent with the TDS data after the deuterium ion irradiation. Then, it is regarded that the deuterium might have been trapped in the bulk region of F82H.



Fig. 5. Thermal desorption spectra of gases containing deuterium for the damaged sample 4 (a) and no damaged sample (b).



Fig. 7. Depth profiles of atomic composition for no damaged sample (a), damaged samples 6 before (b) and after deuterium ion irradiation (c).

#### 4. Conclusion

The reduced activation ferritic steel, F82H, was damaged by using energetic hydrogen at different ion fluence and the irradiation temperature. The damage in dpa was calculated using SRIM code. For the damaged F82H samples and no damaged F82H sample, the deuterium ion irradiation was conducted at room temperature. The amount of retained deuterium was obtained using a technique of thermal desorption spectroscopy. The amount of retained deuterium was several times increased by the damage even if the damage was only several dpa. The desorption temperature of gases containing deuterium became higher compared with that of no damaged sample. These results suggest that the number of trapping site and the trapping energy increase by the damage. In the present experiment, the damage by energetic hydrogen ion bombardment was only a few dpa since the irradiation time was limited. If the damage is extended to 100 dpa and similar experiment is carried out, we can obtain the highest amount of fuel hydrogen.

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# References

- T. Hino, in: 18th IAEA Fusion Energy Conference, CN-77/FTP1/08, CD-ROM, Sorrento, 2000.
- [2] T. Hino, K. Yamaguchi, Y. Yamauchi, Y. Hirohata, K. Tsuzuki, et al., Plasma Sci. Technol. 17 (2005) 2737.
- [3] K. Tsuzuki, H. Kimura, Y. Kusama, M. Sato, H. Kawashima, et al., Fus. Sci. Technol. 49 (2006) 197.
- [4] K. Tsuzuki, K. Kamiya, K. Shinohara, M. Bakhtiari, H. Ogawa, et al., Nucl. Fus. 46 (2006) 966.
- [5] <http://www.srim.org/>.