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The trapping behavior of deuterium in F82H ferritic/martensitic steel

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A B S T R A C T

In the present work, the trapping behavior of deuterium in F82H under irradiation were studied by means of thermal desorption spectrometry (TDS) and TEM. The specimens were first irradiated with 5 keV He^+ at 600 °C followed by the additional D_2^+ irradiations with the energy of 0.5 keV at *RT*. Some of the specimens were also irradiated with relatively higher energy of 1 MeV for He^+ and 650 keV for D_2^+ ions. According to thermal desorption measurements of deuterium and helium after irradiations, deuterium desorption peak shift to higher temperatures of about 30 K were observed for the case with helium irradiations, which attributed to the deuterium trapped by a surroundings of the helium bubbles. In addition, it is also concluded from the thermal desorption measurements of deuterium on re-tempered and as-received specimens without helium irradiations that the latently existing carbides can acts as a potential trapping site for deuterium in F82H.

1. Introduction

For the development of fusion reactor materials, the characterization of trapping and releasing behavior of hydrogen and its isotopes is one of the key issues considering the influences on plasma parameters and tritium inventory. Recent movements show the consideration of using reduced-activation ferritic/martensitic steels for plasma facing components, such as first wall. Meanwhile, ferritic/martensitic steels are also a candidate materials for liquid-cooled high power spallation target for Japanese accelerator driven nuclear transmutation system (ADS), whereas the issues on the material development is also the hydrogen and helium gases produced by high energy proton bombardments.

In ADS, the beam window will be used for the boundary between high intensity proton accelerator and the reactor. The beam window for ADS will suffer both from high energy proton irradiations and neutron bombardment from spallation target. In such environment, crucial amount of helium, hydrogen and its isotopes will be produced in the materials, which production rate is much higher compared to conventional neutron irradiation. Introduction of these gases in materials is known to be one of the causes for radiation embrittlement, hardening and swelling. Thus, characterization of trapping behavior of these gases is also important for ADS in order to contribute to facility design.

Reduced activation ferritic/martensitic steel F82H is one of a candidate alloys for the first wall of fusion reactors as well as for the beam window of spallation target for ADS. In this study, trapping behavior of deuterium in F82H was studied following helium and/or deuterium irradiations by means of thermal desorption spectrometry (TDS) and microstructural observation by TEM.

2. Experimental procedure

The reduced activated F82H ferritic/martensitic steel used in this study was IEA-heat 420 prepared for Japanese fusion reactor material program. The specimens with a dimension of 6×10 mm were mechanically cut to the thickness of around 2 mm. Then the surfaces of the specimens were mechanically polished up to buff polishing with 0.03 μm colloidal silica for low energy ion irradiations. Some samples were re-tempered at 790 °C for 30 min to reduce the dislocation densities in order to subtract the influence of deuterium trapping by dislocations for comparison with the as-received materials, which were annealed at 1050 °C for 1 h followed by the tempering at 750 °C to 30 min. The re-tempering condition was estimated from former studies [1–3].

Low energy helium irradiations and following deuterium irradiations were performed at Research Institute for Applied Mechanics (RIAM), Kyushu University. The specimens were first irradiated with 5 keV He^+ at 600 °C up to 1×10^{20} m^{-2} appmHe followed by the D_2^+ irradiation with an energy of 0.5 keV at *RT* to 1×10^{21} m^{-2} appmD. The deuterium energy of 0.5 keV is that without introduction of point defects into Fe-based alloys. Some of the specimens were also irradiated with relatively higher energy of 1 MeV He^+ at 400 °C up to 1×10^{20} m^{-2} appmHe followed by 650 keV D_2^+ irradiation at RT to 1×10^{21} m^{-2} appmD for comparison. The irradiation was performed at High Fluence Irradiation Facility (HIT), University of Tokyo.

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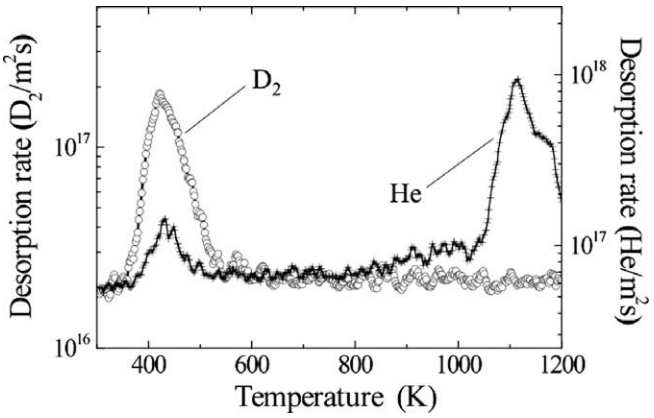

Fig. 1. Thermal desorption spectra of D_2 and He from as-received specimens irradiated with 5 keV He^+ and 0.5 keV D_2^+.

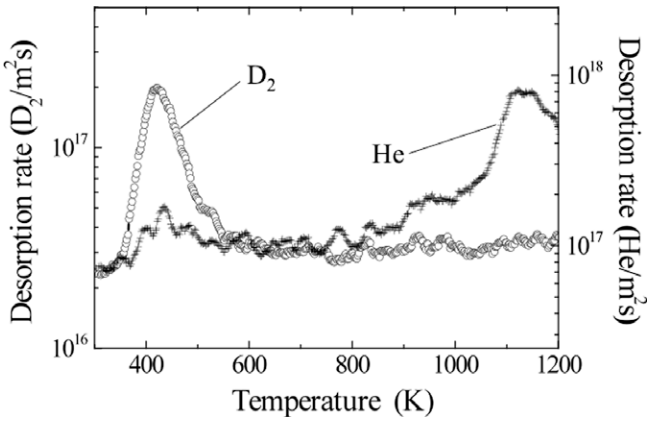

Fig. 3. Thermal desorption spectra of D_2 and He from re-tempered specimens irradiated with 5 keV He^+ and 0.5 keV D_2^+.

After the irradiations, thermal desorption of D_2 and He under heating with a ramping rate of 1 K/s were measured with high resolution quadruple mass spectrometer [4]. This high resolution spectrometer enables to distinguish slight differences in the mass between He ($m = 4.0026$) atom and D_2 ($m = 4.0282$) gas. The desorption rates of He and D_2 were quantitatively calibrated by using standard He leak with specific relative ionization efficiency. At the same time, the samples for the microstructural study by TEM were prepared using focused ion beam system (FIB) from the same specimens.

3. Results and discussion

3.1. Deuterium trapping behavior on He irradiated specimens

Thermal desorption spectra of He and D_2 from specimens irradiated with 5 keV He^+ and 0.5 keV D_2^+ are given in Figs. 1–4. Figs. 1 and 2 shows the spectra from the as-received specimens and Figs. 3 and 4 shows that from re-tempered specimens. Microstructures observed by TEM are given in Fig. 5. In Figs. 2 and 4, the deuterium desorption spectrum from the specimen with only D_2^+ irradiation, which is without helium irradiation, is also shown for comparison.

Helium irradiation on F82H alloys shows remarkable effect on the trapping behavior of deuterium. Normally, for the case of no helium irradiation, large desorption stage from deuterium appears between the temperatures from 350 K to 500 K. On the other hand, in the case with helium irradiation, the desorption stage around this temperature region shifts to higher temperatures for about 30 K, as shown in Figs. 2 and 4. In addition, the large desorption stage appears only in the low temperature region and most of the deuterium desorption ends at this temperature region, as can be seen in Figs. 1 and 3. According to the microstructure observation given in Fig. 5, large helium bubbles are densely formed near the surface of the specimens. If all implanted deuterium was trapped inside of these helium bubbles, deuterium desorption peak should appeared in the higher temperature region which corresponds to that of the helium desorption peak, which is above 1000 K where stagnant helium bubbles at lower temperature become mobile. Therefore, the result indicates that the implanted deuterium was trapped in some other weaker trapping sites, which expected to be the surrounding of highly pressurized helium bubbles.

These types of peak shift on hydrogen gas release behavior for high energy proton irradiated F82H alloy was also seen on the former studies on gas release measurement and microstructural investigation of STIP irradiated materials [5,6]. According to these studies, the desorption stage which should be attributed to the release of hydrogen gases from helium babbles appears more clearly on the specimens irradiated to 20.3 dpa at around 400 °C where formation of high-density small helium bubbles occurs, compared to that irradiated to the lower dose of 10 dpa at around 100 °C where no visible helium bubble formation was observed, which consequently appears as a peak shift. In addition, the same types

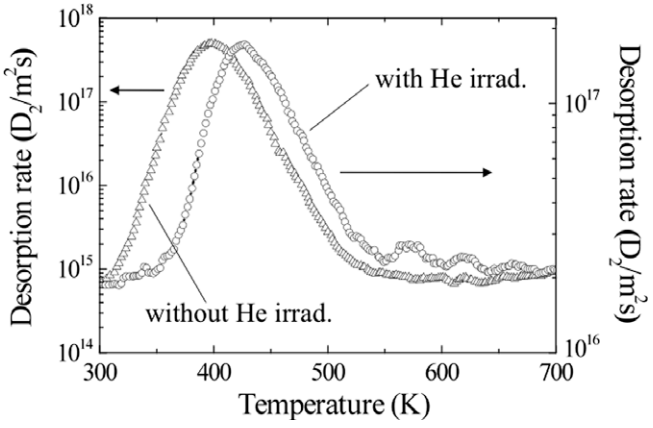

Fig. 2. Thermal desorption spectrum of D_2 from the 5 keV He^+ and 0.5 keV D_2^+ irradiated as-received specimen. The spectrum from the specimen without helium irradiation is also shown for comparison.

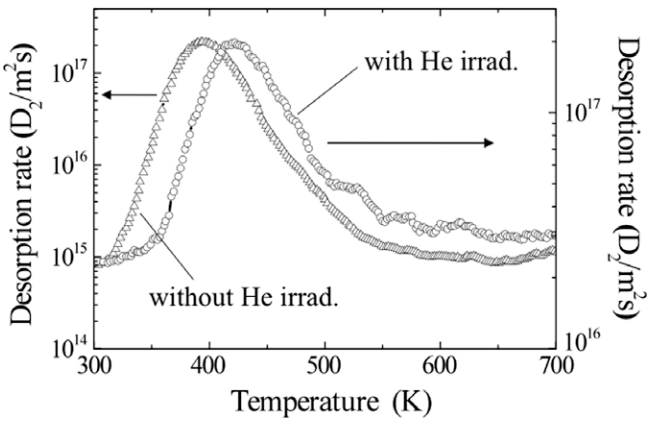

Fig. 4. Thermal desorption spectrum of D_2 from 5 keV He^+ and 0.5 keV D_2^+ irradiated re-tempered specimen. The spectrum from the specimen without helium irradiation is also shown for comparison.

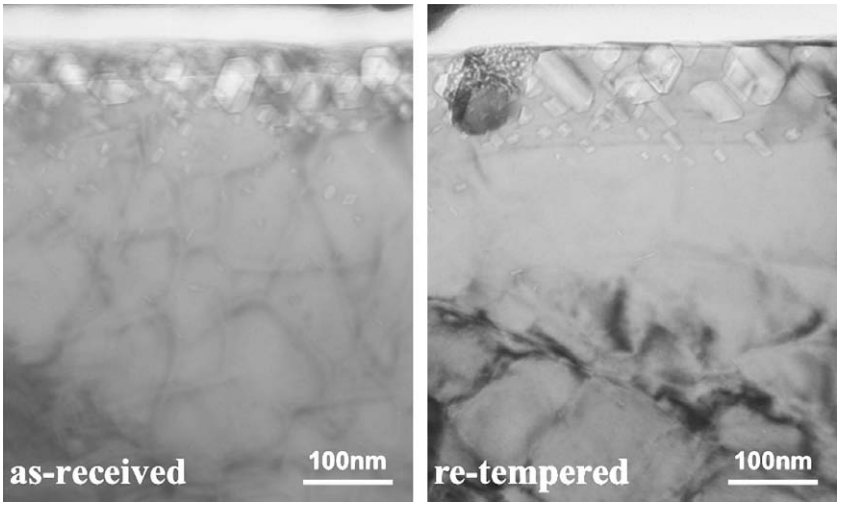

Fig. 5. The microstructures of as-received and re-tempered specimens irradiated with 5 keV He^+ at 600 °C.

of deuterium release behavior are also observed on helium irradiated specimens at relatively higher energy in the present work. Fig. 6 shows the deuterium desorption spectra from the specimen irradiated with 1 MeV He^+ at 400 °C followed by D_2^+ irradiation at RT with the energy of 650 keV. The deuterium desorption spectrum from the specimen with only deuterium irradiation is also shown for comparison. The peak shift of about 50 K and the peak broadening to higher temperature is clearly observed for this case. The peak shift can also be explained by the release of deuterium trapped by the surroundings of helium bubbles, and the broadening is the result of migration from deeper region of the specimen, since the peak region for helium bubble formation in this case is about 1μm deep from the surface according to microstructural observations.

3.2. Potential trapping sites for deuterium on F82H

Thermal desorption spectra and total desorption of deuterium from re-tempered and as-received specimens irradiated with D_2^+ at RT to 1×10^{21} m^{-2} appmD are given in Figs. 7 and 8. Note that D_2^+ irradiation was also carried out with very low energy of 0.5 keV, where no displacement damage introduction occurs in Fe-based alloys. The microstructures for these specimens before irradiations are shown in Fig. 9, where the grain growth and reduction of number densities of carbides and dislocations can clearly be seen in the re-tempered specimen.

There were two desorption stages for deuterium observed for F82H. One appears between the temperatures from 350 K to 500 K and the other from around 800 K to 1000 K. These stages were seen on both re-tempered and as-received specimens. Desorption stage at lower temperature is typical for Fe–Cr based

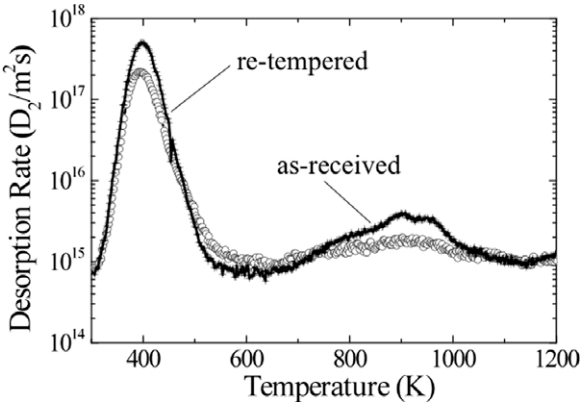

Fig. 7. Comparison of D_2 desorption spectra between as-received specimen and re-tempered specimen irradiated with 0.5 keV D_2^+.

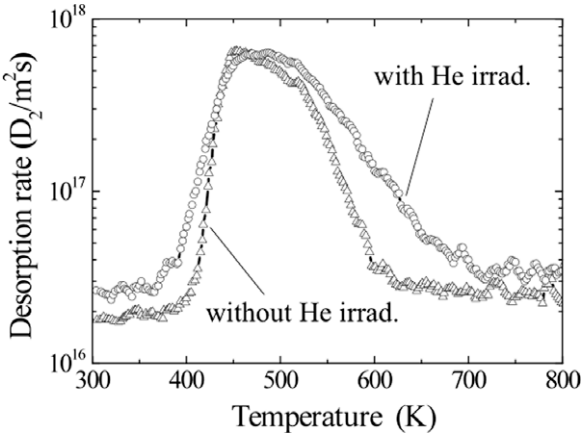

Fig. 6. Thermal desorption spectra of D_2 and He from as-received specimen irradiated with 1 MeV He^+ and 650 keV D_2^+.

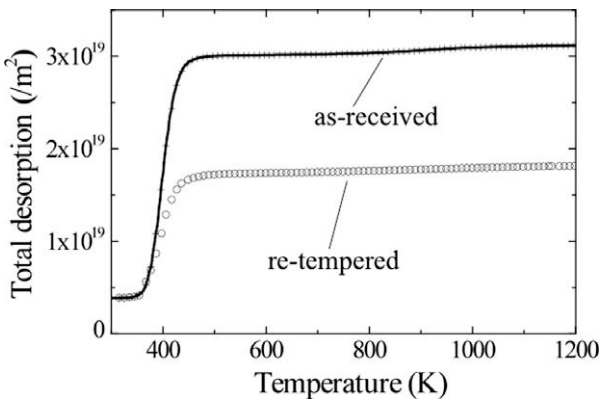

Fig. 8. Total desorption of D_2 from re-tempered specimen and as-received specimen irradiated with 0.5 keV D_2^+ to 1×10^{21} m^{-2} appmD.

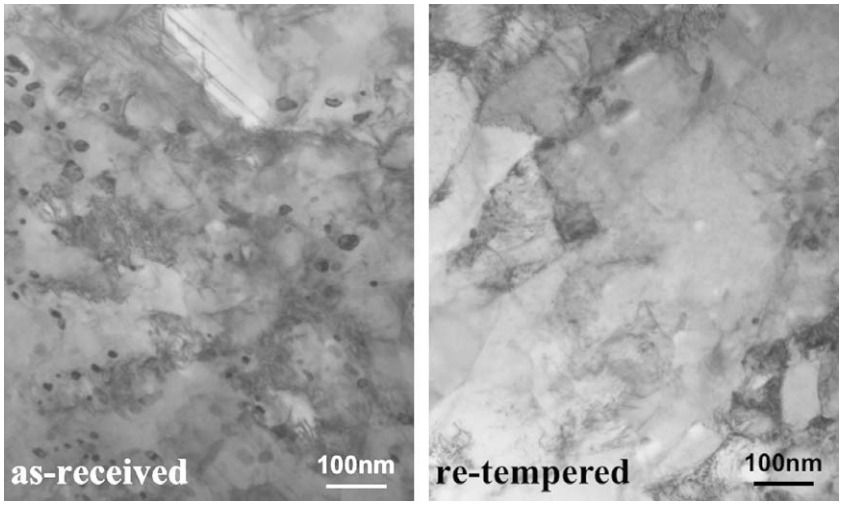

Fig. 9. The microstructures of as-received and re-tempered specimens before 0.5 keV D_2^+ irradiations.

alloy. This stage is assumed to be a release of deuterium mainly trapped on dislocations, since the stage also appears on the cold worked Fe–9Cr alloy though it disappears after annealing [7]. On the other hand, the stage at higher temperature is unique to F82H. From the microstructural observations, growth of the grins and reduction in dislocation density was observed after tempering at 790 °C for 30 min. In addition, the density of carbides decreased rapidly after tempering. Since the stage at higher temperature appears on both re-tempered and as-received specimens and total desorption decreased to about half on tempered specimens, this stage at higher temperature is mainly attributed to the deuterium trapped by the carbides.

4. Conclusion

Trapping behavior of deuterium on F82H ferritic/martensitic steels were studied by means of thermal desorption spectrometry and microstructural observations following He^+ and/or D_2^+ irradiations. In addition, deuterium desorption measurements on re-tempered and as-received materials with only D_2^+ irradiations were also performed in order to evaluate the potential trapping sites for deuterium on F82H. The results are summarized below.

1. Desorption stage for deuterium at around 350–500 K were shifted to higher temperature of about 30 K for the case of helium irradiated specimens compared to which without irradiation.
2. Since large helium bubbles were densely formed near the surface after irradiations and helium desorption stage mainly appears at temperatures above 1000 K, where helium bubbles become mobile, the shift of the deuterium desorption stage can be attributed to the deuterium trapped at surroundings of highly pressurized helium bubbles.
3. Desorption stage at around 350–500 K observed on the specimens without helium irradiations is typical to Fe–Cr based alloys. On the other hand, desorption stage appeared at around 800–1000 K is unique to F82H alloys.
4. Desorption stage at higher temperatures observed in F82H is assumed to be attributed to the deuterium trapped by the carbides, since desorption stage at lower temperature should attributed to the deuterium trapped by dislocations.

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