Deuterium retention and desorption behavior in an advanced reduced-activation alloy

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HIGHLIGHTS

- The first result of the deuterium retention and desorption in an ARAA is presented.
- The ARAA was irradiated with helium and then continuously irradiated with deuterium.
- TDS measurements were performed in situ immediately after deuterium irradiation.
- TDS measurements were performed after exposure to air for one week.
- The effects of helium irradiation and exposure to air were investigated.

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ABSTRACT

We present the first experimental results of the deuterium retention and desorption behavior in an advanced reduced-activation alloy (ARAA) under development in Korea. For the in-situ measurement of desorbed gases from samples immediately after irradiation, a thermal desorption spectroscopy (TDS) system clustered with an inductively coupled plasma ion source has been built. Samples were and were not irradiated with helium ions at energies of 1.4, 3.5, and 5.0 keV and then continuously irradiated with 1.7-keV deuterium ions. TDS measurements were performed in situ immediately after deuterium irradiation and after exposure to air for one week. The amount of desorbed deuterium is the largest for the sample without helium irradiation from the TDS results measured in situ immediately after irradiation. Further, the amount of desorbed deuterium is significantly lowered when the helium energy is increased to 3.5 keV with no significant changes thereafter, indicating that the layer formed by implanted helium at near or deeper than the stopping range for 1.7-keV deuterium ions effectively acts as a barrier against deuterium diffusion into the depth. Because of the strong diffusivity of deuterium into the ambient atmosphere, the amounts of desorbed deuterium are greatly reduced for the samples without helium irradiation and with 1.4-keV helium irradiation after exposure to air for one week. In addition, our deuterium results for the ARAA are also compared with the results for F82H by other authors.

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1. Introduction

The development of advanced low-activation materials for the first wall and blanket structure in fusion reactors is the key issue for the realization of nuclear fusion [1–5], and the Korea Atomic Energy Research Institute has been developing an advanced reduced-activation alloy (ARAA) [6–8]. During the fusion process, the first wall is directly exposed to an environment of incident particles and the heat flux from a plasma. As a result, its surface can be damaged, and this damage has been reported to affect the retention and desorption behaviors of hydrogen isotopes, which are associated with the plasma performance, fuel control, and safety [9–24]. Some authors have reported the deuterium retention and desorption behavior of the reduced-activation ferritic/martensitic (RAFM) steel F82H by using thermal desorption spectroscopy (TDS) [15–24]. However, the number of available reports on the deuterium retention and desorption behavior of RAFM steels damaged by helium irradiation is very limited [23,24]; for example, Yamauchi

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et al. [24] have reported the results obtained for F82H damaged by 5-keV helium irradiation prior to 1.7-keV deuterium irradiation. We note that information regarding the environment and time concerning sample storage from the completion of deuterium irradiation to the start of the TDS measurement was not clearly stated in Refs. 15−24.

In this study, we built a TDS system clustered with an inductively coupled plasma (ICP) ion source and investigated the deuterium retention and desorption behavior in an ARAA. The ARAA samples were either irradiated with helium ions (1.4, 3.5, or 5.0 keV) or not irradiated and then continuously irradiated with 1.7-keV deuterium ions. Samples for the TDS measurements were divided into two groups. For the first group, TDS measurements were performed in situ immediately after deuterium irradiation. On the contrary, the samples in the second group were exposed to air for one week prior to TDS measurements.

2. Experiment

The ARAA (heat number RC4417) was supplied by POSCO Specialty Steel Co., Ltd., Korea. The ARAA is a 9Cr-1.2W-based ferritic/martensitic steel with 0.01 wt% Zr and exhibits a much longer creep-rupture time than conventional RAFM steels [7]. The chemical composition of the ARAA is given in Table 1. Further information about the ARAA can be found in the literature [6−8].

We have built a TDS system clustered with an ICP ion source. Fig. 1 shows a schematic of the custom-built system. The system consists of three modules: a module for the ICP ion source, a module for sample heating, and a module for the measurement of desorbed gases. The ion source module is separated from the heating module by a gate valve, and the heating module is connected with the measurement module. The ion source module and measurement module are evacuated by turbomolecular pumps (TMPs), and the system can reach an ultrahigh vacuum pressure of ~10^{-6} Pa. The sample is heated in the heating module for degassing and healing the damage due to polishing processes and then transferred to the ion source chamber through the gate valve without being exposed to air. The sample is irradiated in the ion source module, and the irradiated sample can be transferred to the heating module through the gate valve immediately after irradiation for the TDS measurement or can be stored in other environments prior to the TDS measurement. In the heating module, the sample can be linearly heated from room temperature up to 1273 K at a constant ramp rate. Desorbed gases are analyzed in the measurement module by using a residual gas analyzer (RGA).

The ARAA alloy was fabricated into disk-shaped coins (diameter: 20 mm, thickness: 1 mm), and the surfaces were mechanically polished by Al_2O_3 powders and then chemically cleaned with ethanol using an ultrasonic bath. The samples were degassed in a vacuum at 1173 K for 1 h. After cleaning and heating, the ARAA

Table 1
Chemical composition of the ARAA.

Element	Concentration (wt%)
C	0.10
Cr	9.0
W	1.2
Mn	0.45
V	0.20
Ta	0.07
Si	0.10
N	0.01
Ti	0.01
Zr	0.01
Fe	Balance

samples were either irradiated with helium ions (1.4, 3.5, or 5.0 keV) at a fixed fluence of 5.0×10^{21} He/m^2 at room temperature or not irradiated. The samples were continuously irradiated with 1.7-keV deuterium ions at a fixed fluence of 6.5×10^{21} D/m^2 at room temperature as well. The samples were divided into two groups for the TDS measurements. The samples in the first group were transferred to the heating module immediately after deuterium irradiation, and TDS measurements were performed from room temperature to 1100 K with a linear ramp rate of 0.5 K/s. On the contrary, the samples in the second group were exposed to air for one week after deuterium irradiation, and TDS measurements were also performed from room temperature to 1100 K with a linear ramp rate of 0.5 K/s. During heating, the gases that desorbed from the sample were quantitatively measured by the RGA (Model RGA 100, Stanford Research Systems, Inc., USA). The RGA sensitivity was calibrated by measuring the RGA signal at various gas flow rates; the measurement was performed when the signal for each flow rate reached a steady state in order to minimize the inaccuracy arising from gas adhering to surfaces. The differences in the RGA signals from the least-squares fit were within 5%. Since it is difficult to calibrate the RGA signals for HD, HDO, and D_2O with the same method, the average value of the calibration factors for those of D_2 and H_2 was used for HD, and the calibration factor for H_2O was used as the calibration factor for HDO or D_2O [9]. By integrating the desorption rate with respect to the heating time, the total amount of desorbed gases could be obtained.

3. Results

An RGA is a mass spectrometer that analyzes residual gases by measuring the mass-to-charge ratio of the ions. Most commonly, the mass of the ion (m) is approximated as the mass of its molecule in atomic mass units, and the charge of the ion is measured in units of the electron charge (e). The mass-to-charge ratio of D_2^+ corresponds to $m/e = 4$, and that of He$^+$ also corresponds to $m/e = 4$; during the TDS measurement, the signals associated with both D_2 and He appear as the signal of $m/e = 4$. We irradiated an ARAA sample with 1.7-keV deuterium ions at a fluence of 6.5×10^{21} D/m^2 and measured the time evolution of the $m/e = 4$ signal. Similarly, we irradiated an ARAA sample with 5.0-keV helium ions at a fluence of 5.0×10^{21} He/m^2 and measured the time evolution of the $m/e = 4$ signal. Fig. 2 shows examples of the time evolution of the $m/e = 4$ signal intensity during the TDS measurements for the two experiments performed independently. Specifically, Fig. 2(a) and (b) show the $m/e = 4$ signals for the samples irradiated with 1.7-keV deuterium and 5.0-keV helium ions, respectively. As shown in Fig. 2, the desorption signal of D_2 has a peak around 700 K, whereas the desorption signal of He has a peak around 850 K; the D_2 signal is clearly distinguished from the He signal. Throughout our TDS study presented afterwards, the D_2 desorption signals were selectively analyzed from the $m/e = 4$ signal.

Fig. 3 shows the thermal desorption spectra of D_2, HD, HDO, and D_2O for the ARAA samples. The samples (a, e) were not irradiated with helium ions and were irradiated with (b, f) 1.4-keV, (c, g) 3.5-keV, and (d, h) 5.0-keV helium ions at a fixed fluence of 5.0×10^{21} He/m^2. The samples (a-h) were continuously irradiated with 1.7-keV deuterium ions at a fluence of 6.5×10^{21} D/m^2. Fig. 3(a−d) show the spectra of the first-group samples that were measured in situ immediately after deuterium irradiation, whereas Fig. 3(e−h) show the spectra of the second-group samples exposed to air for one week after deuterium irradiation prior to the TDS measurement.

In Fig. 3(a−d), the desorption rate at the D_2 peak has a large value compared to the desorption rates at the other peaks of HD, HDO, and D_2O. The D_2 spectrum for the sample without helium