A Study on Thermal Desorption of Deuterium in D-loaded SS316LN for ITER Tritium Removal System

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1. Introduction

Type B radwaste (Intermediate level and long lived radioactive waste) is generated from the vacuum vessel inner structure of ITER Tokamak building when its structural components are replaced for maintenance. Because Type B radwaste includes tritium on its inside, especially at vicinity of surface, tritium removal from the radwaste is a matter of concern in terms of the radwaste processes. Tritium behavior in materials is related with temperature. Considering a diffusion process, it is expected that tritium removal efficiency is enhanced with increasing baking temperature. However, there is a limitation about temperature due to facility capacity and economic aspect. Therefore, it is necessary to investigate the effect of temperature on the desorption behavior of Tritium in ITER materials.

2. Methods and Results

In the present study, Deuterium (D) was substituted for tritium due to the safety concern. To investigate the removal behavior of D during heating, D-loaded SS316LN, which is ITER structural material, was heated in thermal desorption spectroscopy (TDS) chamber up to 900°C. During continuous heating, D desorption was detected by residual gas analyzer (RGA) of TDS.

2.1 Deuterium Loading

The sample was $\Phi 4 \times 5$ mm cylindrical shape. D loading was performed in high pressure D₂ ambient (25 bar) at three temperatures of 120, 240, and 350°C for 24 h. (D-loaded samples were prepared and provided by *Hanyang University*.)

2.2 TDS

Because TDS is equipment for investigating thermal desorption behavior of material during continuous heating, it was utilized to investigate the behavior of D desorption in terms of temperature. The pressure of TDS chamber was initially about 10^{-8} torr. Maximum heating temperature for investigating D_2 desorption behavior was 900°C and the ramp rate of TDS heater was set to 0.5 °C/s. To calculate D concentration in the sample, D_2 calibrated leak was installed at TDS to

convert partial pressure to mol or wppm. Before the TDS test, the leak was opened to flow regular D_2 and conversion factor was calculated from calibration leak rate and detected partial pressure of D_2 by RGA of TDS. Using the conversion factor, detected RGA data was converted to quantitative data. Then, the area of TDS curve of D_2 desorption was integrated to calculate the D_2 concentration in each samples.

2.3 D₂ desorption behavior

TDS analysis was performed to investigate the desorption behavior of D in D-loaded SS316LN samples. Fig. 1 shows the concentration of desorbed D_2 from SS316LN loaded at 120 °C. D_2 desorption started at 750 sec and the maximum peak was 0.002 wppm. D desorption was kept up to 1,500 sec. Fig. 2 shows the concentration of desorbed D_2 from SS316LN loaded at 240 °C. D_2 desorption started at 800 sec and the maximum peak was 0.01 wppm. Desorption of D_2 was kept up to 2,050 sec. Fig. 3 shows the concentration of desorbed D2 from SS316LN loaded at 350 °C. D_2 desorption started at 800 sec and the maximum peak was 0.02 wppm. Desorption of D_2 was kept up to 2,200 sec.

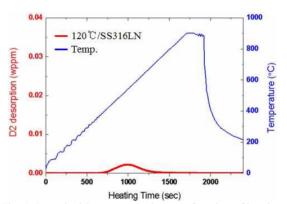


Fig. 1. Desorbed D₂ concentration as a function of heating time (120 °C loaded SS316LN)

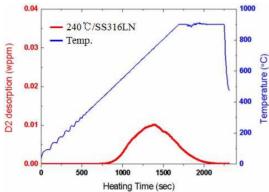


Fig. 2. Desorbed D₂ concentration as a function of heating time (240 °C loaded SS316LN)

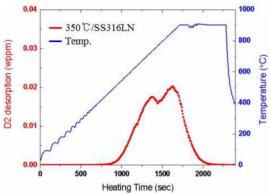


Fig. 3. Desorbed D₂ concentration as a function of heating time (350 °C loaded SS316LN)

The comparison of D_2 desorption as a function of temperature in SS316LN samples loaded at 120 °C, 240 °C and 350 °C is shown in Fig. 4. The peak temperatures were 538, 741, and 862 °C in SS316LN loaded at 120, 240, and 350 °C, respectively. The peak temperature and the maximum intensity at this point increased with increasing loading temperature. In case of 120 °C loaded SS316LN, the D_2 desorption finished before reaching to 900 °C. However, in 240°C and 350 °C loaded SS316LN, D_2 desorption finished after holding 900 °C for a while.

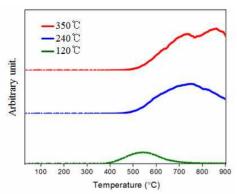


Fig. 4. Comparison of D_2 thermal desorption curves in SS316LN loaded at 120, 240 and 350 °C

When the area of curve was integrated, the concentrations of D_2 were 0.8, 6.2, and 12.2 in SS316LN loaded at 120, 240, and 350 °C, respectively. Table 1 shows D2 concentration of SS316LN loaded at 120, 240 and 350 °C measured by KAERI (heating up to 900 °C) and HYU (800 °C). The measured data from KAERI for SS316LN loaded at 120, 240 and 350 °C was 0.8, 6.2 and 12.2 wppm, respectively, while the results from HYU was 0.7, 6.3 and 14.7 wppm, respectively. Because both data shows similar value, it was considered that there was reliability in measurement method between KAERI and HYU.

Table 1. D_2 concentration of SS316LN measured by KAERI and HYU

Loading Temp. (°C)	Desorption Completion Temp. (°C)	D ₂ amount (wppm) KAERI	D ₂ amount (wppm) HYU
120	797	0.8	0.7
240	900	6.2	6.3
350	900	12.2	14.7

2.4 Trap activation energy

According to the previous study [1], it has known that peak temperature of desorption decreased with decreasing ramp rate. Using the peak shift, trap activation energy of absorbed element in the material can be calculated. When we remove D in the samples, measuring trap activation energy can be helpful to compare the removal behavior with other materials. In here, three ramp rates of 0.166, 0.332, and 0.5 °C/sec were used. The shift of peak temperature as various ramp rates was measured, and then trap activation energy of D was calculated by peak shift method.

In TDS test results with the ramp rates of 0.166, 0.332, and 0.5 °C/sec, the peak temperatures were 717, 790, and 862 °C, respectively. The graph for trap activation energy of D in SS316LN was drawn using the given ramp rates and peak temperatures obtained from each test, as shown in Fig. 5. In here, T_c means peak temperature and Φ represents ramp rate. When draw a linear line for each points, the slope was calculated to -6,700. The slope multiplied by gas constant R=8.314 kJ/mol and the trap activation energy was calculated to 56 kJ/mol. The slope can be changed considerably with little shift of the point. If more exact energy value is needed, TDS test with slower ramp rate will be more proper. However, in the present study, the energy driven from the ramp rates around 0.5 °C/sec was considered to be helpful to compare the removal behavior of D₂ in other ITER candidate materials approximately.

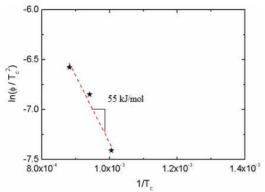


Fig. 5. Trap activation energy of D in SS316LN loaded at $350\,^{\circ}\text{C}$

3. Conclusions

TDS analysis was performed in SS316LN loaded at 120, 240 and 350 °C. D_2 concentration and the desorption peak temperature increased with increasing loading temperature. Using peak shift method with three ramp rates of 0.166, 0.332, and 0.5 °C/sec, trap activation energy of D in SS316LN loaded at 350 °C was 56 kJ/mol.

REFERENCE

[1] W. Y. Choo and J. Y. Lee, J. Mater. Sci., 17 (1982)