# Desorption Behavior of Hydrogen Isotopes Implanted into Stainless Steel by Glow Discharge

グロー放電によりステンレス鋼に注入した水素同位体の脱離挙動

<u>Masamitu Kondou</u><sup>1)</sup>, Kiyohiko Nishimura<sup>2)</sup>, Msahiro Tanaka<sup>2)</sup>, Nobuaki Noda<sup>1)</sup> and Masao Matsuyama<sup>1)</sup> <u>近藤将充</u><sup>1)</sup>, 西村清彦<sup>2)</sup>, 田中将裕<sup>2)</sup>, 野田信明<sup>1)</sup>, 松山政夫<sup>1)</sup>

 Hydrogen Isotope Research Center, University of Toyama, Gofuku 3190, Toyama 930-8555, Japan 富山大学水素同位体科学研究センター,〒930-8555 富山市五福 3190
National Institute for Fusion Science, 322-6 Oroshi-cho, Toki 509-5292, Japan 核融合科学研究所,〒509-5292 土岐市下石町 322-6

Desorption behavior of hydrogen and deuterium implanted into type 316L stainless steel by glow discharge has been studied by experiments and numerical calculation based on a diffusion-limited process. Desorption spectra of hydrogen and deuterium observed by experiments agreed with the numerically calculated spectra. This result suggests that desorption rate of hydrogen isotopes implanted by glow discharge is limited by diffusion rate of hydrogen isotope in the bulk. Furthermore, no large isotope effects were observed on the desorption behavior.

#### 1. Introduction

Control of fuel particles in the reactor core is indispensable to make steady operation of a fusion reactor. A part of fuel particles is implanted into the plasma-facing materials (PFM's) and released from them during the operation. Therefore, it is of a great important issue to make clear the desorption behavior of hydrogen isotopes from PFM's from viewpoint of stable fuel balance.

From this view point, desorption behavior of hydrogen isotopes implanted into stainless steel by a glow discharge has been studied using temperature desorption spectroscopy (TDS), and analysis of desorption spectra was carried out by a numerical calculation method.

#### 2. Experimental

A square plate of type 316L stainless steel was used as a sample and its size was  $15x15x0.5 \text{ mm}^3$ , which was mechanically polished and rinsed with acetone before use.

The sample was set in the experimental apparatus, which was specially designed and manufactured for the glow discharge and desorption experiments. Schematic diagram of the apparatus is shown in Fig. 1. This apparatus has an advantageous point that the sample exposed to glow discharge in the chamber of Glow-2 of hydrogen isotope atmosphere can be moved to Glow-1, where it



Fig. 1 Schematic diagram of the experimental device used to investigate desorption behavior.

is analyzed with TDS without air exposure. Conditions of glow discharge are as follows:

- (1) Pressure of hydrogen isotope: 130 Pa
- (2) Applied voltage: 250 V (H<sub>2</sub>), 270 V (D<sub>2</sub>)
- (3) Total current: 20 mA
- (4) Exposure time after discharge: 60 min
- (5) Sample temperature: Room temperature

After glow discharge in the Glow-2, the sample was kept exposing to hydrogen isotope. Subsequently, it was evacuated below  $5 \times 10^{-5}$  Pa and the sample was transferred to Glow-1. Evacuation time was changed in the range from 1 to 5 hours. After evacuation for a given time, the sample temperature was linearly raised from room temperature to 613 K at a given temperature ramp. Each partial pressure of hydrogen isotopes desorbed from the sample was

measured by a conventional quadrupole mass spectrometer.

# 3. Numerical Calculation

Numerical calculations were carried out by applying a simple diffusion model as the first approximation. It was assumed that hydrogen isotope atoms diffuse in stainless steel with no trapping or agglomeration and the diffusion of them simply obeys the Fick's Law with a source term as shown in the following equation:

$$\frac{\partial C(x,t)}{\partial t} = D(T(t))\frac{\partial^2 C(x,t)}{\partial x^2} + G(x) \quad , \quad -\dots \quad (1)$$

where C(x, t) is the concentration of hydrogen isotope at a distance x from the surface at time t, T(t)the absolute temperature of the sample , D(T(t)) the diffusivity of hydrogen isotope at a given temperature, and G(x) the source term. The diffusivity can be expressed by the following equation:

$$D(T(t)) = D_0 \exp(-E_d / RT(t))$$
 .....(2)

#### 4. Results and Discussion

A typical desorption spectrum of deuterium is shown in Fig. 2. It was observed under the following conditions: exposure time was 90 min at room temperature, and evacuation time was 60 min. The sample was heated using an infrared heating device. Heating rate was 20 K/min. A single desorption peak was observed and peak temperature was around 400K [1, 2]. Desorption species consisted of D<sub>2</sub>, HD and H<sub>2</sub>, and a small amount of water species (HDO and D<sub>2</sub>O) was also observed.

Best fit to the observed deuterium spectrum was obtained when  $E_d$  and  $D_0$  were assumed to be 51.0



Fig. 2 Desorption spectra observed and calculated for deuterium glow discharge.



Fig. 3 Comparison of desorption spectra observed <sup>10</sup> for hydrogen and deuterium.

kJ/mol and  $1.7 \times 10^{-7}$  m<sup>2</sup>/s, respectively. These diffusion parameters almost agreed with the values reported by Wilson and Baskes [1]: 52.9 kJ/mol and  $1.7 \times 10^{-7}$  m<sup>2</sup>/s for as-received sample of type 316 stainless steel.

All of the desorption spectra observed under various conditions of discharge time, evacuation time and heating rate could be reproduced using the same diffusion parameters except for a low temperature region. Therefore, these results indicate that desorption rate of deuterium implanted into the sample by glow discharge is limited by diffusion rate in the bulk.

Comparison of two desorption spectra for hydrogen and deuterium is shown in Fig. 3. Both spectra were measured under the same conditions: namely, 90 min discharge, 60 min evacuation, and 20 K/min heating rate. As clearly seen from this comparison, there were no large differences in both peak temperature and desorption rate. These results suggest that isotope effects upon desorption behavior of hydrogen isotopes are negligibly small above room temperature.

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## References

- K.L. Wilson and M.I. Basakes: J. Nucl. Mater. 74 (1978) 179.
- [2] R.E.Clausing, L.C.Emerson, L.Heatherly, R.J.Colchin: Proc. Intl. Conf. on Plasma Wall Interactions, Julich, 1976, p.573.