# Deuterium retention and desorption of tungsten with inert gas plasma pre-exposure

希ガスプラズマ曝露後のタングステンの重水素保持・脱離挙動

<u>T.Nishio</u>, Y.Yamauchi, Y.Nobuta, T.Hino <u>西尾知久</u>、山内有二、信太祐二、日野友明

Laboratory of Plasma Physics and Engineering, Hokkaido University Kita13 Nishi8, Kita-ku, Sapporo, Hokkaido, Japan 北海道大学大学院工学院 プラズマ物理工学研究室 北海道札幌市北区北13条西8丁目

Tungsten samples were pre-exposed to inert gas plasma with fluence of  $2 \sim 10 \times 10^{16}$  ions cm<sup>-2</sup> and continuously exposed to deuterium gas plasma with fluence of  $5 \times 10^{16}$  ions cm<sup>-2</sup> with glow discharge device. The amounts of retained D in samples were measured by thermal desorption spectroscopy. The amount of retained D was larger than that without pre-exposure sample when the fluence of pre-exposure was  $10 \times 10^{16}$  ions cm<sup>-2</sup> in all inert gas cases. Different inert gas pre-exposure showed different pre-exposure fluence dependence of the amount of D retention.

#### 1. Introduction

Tungsten is one of candidate materials for plasma-facing walls of fusion reactor because of its high melting point and low-sputtering erosion yield. The walls of fusion reactor will be received particle loads such as neutron, helium ash and hydrogen isotope during the operation and wall conditionings such as glow discharge cleanings. Understandings of hydrogen isotope behavior in the wall materials are key issues for fuel density control and safety operation of fusion reactors. On the other hand, the particle loads during would cause the change in the surface characteristics such as distributions of atomic composition and defects. These might lead to the change in the hydrogen isotope behavior in the wall materials.

However, these effects on the hydrogen isotope behaviors have not been understood sufficiently. The objective of the present study is to investigate the effect of inert gas plasma pre-exposure on deuterium retention and desorption of tungsten by sequential exposures to inert gas and deuterium plasmas.

## 2. Experiment

The sample used was powder-metallurgy tungsten with 99.95 % purity manufactured by Nilaco Corp. The sample size was 10 mm x 20 mm x 0.1mm. The samples were subjected to mechanical polishing and ultrasonic cleaning in ethanol.

The sample was exposed to He, Ne or Ar plasma, and subsequently exposed to  $D_2$  plasma in the glow plasma discharge device [1] at room

temperature. Discharge gas was introduced using mass flow meter. The discharge pressure was 8 Pa and the discharge voltage was 300 V. The fluence of the inert gas plasma pre-exposure were ranged between 2-10 x  $10^{16}$  ions cm<sup>-2</sup>. The fluence of the deuterium plasma was 5 x 10<sup>16</sup> ions cm<sup>-2</sup>. After the deuterium plasma exposure, exposed, the deuterium desorption/retention behavior was evaluated by thermal desorption spectrometry (TDS). In the TDS, the sample was heated from room temperature to 1273 K with a heating rate of 0.5 K/s and kept at 1273 K for 10 min. during the heating, the desorption rate of deuterium from the sample was quantitatively measured with a quadruple mass spectrometer (OMS). То distinguish helium from deuterium, a high resolution QMS was also used when in the case of He pre-exposure. Scanning electron microscopy (SEM) and auger electron spectroscopy (AES) were also carried out to observe surface morphologies and atomic compositions, respectively.

## 3. Results

Fig.1 shows the amounts of desorbed  $D_2$  as functions of pre-exposure fluence. The amount of retained D was larger than that without pre-exposure sample when the fluence of pre-exposure was 10 x 10<sup>16</sup> ions cm<sup>-2</sup> in all inert gas cases. In the He pre-exposed samples, the desorption amount slightly decreased when the fluence of pre-exposure was 2 x 10<sup>16</sup> ions cm<sup>-2</sup>, and then increasing with the He pre-exposure fluence. In the Ne pre-exposed samples, the desorption amount temporally decreased with the increase of the pre-exposure fluence, and then increased. In the Ar pre-exposed samples, the decrease of the desorption amount by the pre-exposure was larger than other inert gas cases when the fluence of pre-exposure was  $2 \times 10^{16}$  ions cm<sup>-2</sup>, and then increased with the pre-exposure fluence.

Fig.2 shows thermal desorption spectra of  $D_2$  for all inert gases cases with 10 x  $10^{16}$  ions cm<sup>-2</sup> fluence pre-exposure. Desorption peaks from tungsten and tungsten carbide [2] were observed at around 450 K. In the He pre-exposure, the desorption intensity around 500K was slightly larger than other inert gas cases.

Fig.3, 4 show the depth profiles of atomic compositions for W with the He and the Ar plasma exposure, respectively. The pre-exposure fluence was 5 x  $10^{16}$  ions cm<sup>-2</sup>. According to the TRIM calculation [2], the Ar ions irradiated to W could be implanted to a depth of a few nm, and cause elastic collision and irradiation damages. In the Ar pre-exposed sample, carbon impurities slightly decreased in a few nm from the top surface. This result would be the reason why the result in the Fig.2 was caused. Also this result would indicate that the amount of desorbed D<sub>2</sub> with the Ar pre-exposure could increase with pre-exposure fluence.

In this experiment, no irradiation damage was observed at the top surface from SEM images.



25with the He pre-exposure [10<sup>11</sup> molec. cm<sup>-2</sup> s<sup>-1</sup>] 20**Desorption rate** • With the Ne pre-exposure 15• with the Ar pre-exposure 10 $\mathbf{5}$ 0 500900 1100 300 700 **Temperature** [K] Fig.2. Thermal desorption spectra of  $D_2$ 100 80 Atomic Composition v 60 40 [%] 0 20 0 0 10

Depth [nm]

Fig.3. Atomic Composition of W with the He plasma exposure



Fig. 4. Atomic composition of W with the Ar plasma exposure

#### References

- Y. Yamauchi, K. Takeda, Y. Nobuta, T. Hino J. Nucl. Mater., 390–391 (2009), pp. 1048–1050.
- [2] Wenmin Wang et al, Journal of Nuclear Materials, 241-243 (1997), p.1087.