Retention and Desorption of Deuterium in Heavily Irradiated W 重照射損傷を受けたタングステンの水素同位体保持・放出特性 Shiori NAITOU¹, Naoki FUTAGAMI¹, Shouji MIYAMOTO², Tomonori TOKUNAGA¹, Hideo WATANABE³, Naoaki YOSHIDA³ <u>内藤梓小里¹</u>, 二神直樹¹, 宮本正二², 徳永知倫¹, 渡辺英雄³, 吉田直亮³ ¹IGSES Kyushu Univ., ²NSK Co. Ltd., ³RIAM Kyushu Univ. 九大総理工¹、日本精工²、九大応力研³ 6-1, Kasugakoen, Kasuga-City, Fukuoka 816-8580, Japan 〒816-8580 福岡県春日市春日公園6-1

In this study behavior of implanted deuterium in tungsten samples with different type of defects were examined. In case of tungsten irradiated by energetic heavy ions, it was identified that not only vacancies and dislocations but also fine vacancy clusters such as nano-voids and small complex, act as strong trapping site of deuterium by comparing the microstructure and TDS spectrum.

1. Introduction

Plasma facing materials in a fusion reactor a large number of lattice defects are formed by the heavy irradiation of fast neutrons. On the other hand, hydrogen isotopes from the plasma penetrate into the material through the surface and are trapped by the radiation induced defects. Estimation of tritium inventory in the plasma facing material in fusion reactors is very important from the standpoint of safety. In order to estimate the amount of the retained tritium, nature of the accumulated defects and their interaction with hydrogen isotopes must be understand well.

Tungsten is a potential candidate of plasma facing materials for protecting heavy heat load and particle loads. In this work, therefore, several kinds of tungsten samples with different treatments such as heating, plastic deformation and heavy ion irradiation are prepared. By comparing the behavior of implanted deuterium and microstructure in these samples, interaction of hydrogen isotopes with the defects were examined.

2. Experimental procedures

In this study, tungsten sheets manufactured by Nilaco Co. (99.95% purity, 0.1 mm-thick) were used as a sample. After cutting into a size of 5×10 mm from the sheet, mechanical polishing, annealing (if necessary) and electro-polishing were carried out successively.

Some samples were annealed for 30 min at temperatures from 1173 K to 1323 K. They were irradiated at room temperature with 0.2keV-deuterium ions to $1x10^{21}ions/m^2$ and TDS experiments were performed successively. Microstructure was also observed by TEM to look for the responsible defects for trapping of

deuterium.

Some of the recrystallized samples were irradiated at room temperature with 2.4 MeV-copper ions. After the irradiation, TEM observation and TDS experiment were also carried out.

3. Results and Discussions

TDS spectra of D_2 from the annealed samples and mechanically deformed samples are shown in Fig.1. Desorption from the as received sample is similar to that of 1173K/30min-sample. Remarkable desorption peaks at around 330-420K (peak A) and 420-560K (peak B) become smaller by increasing the annealing temperature. Desorption from the re-crystallized samples is very small and desorption at the peak B almost vanished. Fig.2 shows TDS spectrum of DH from the annealed samples. It is worth to note that a new peak around 600-900K (peak C) appear in addition to peak A and Peak B.

After heavy ion irradiation up to 0.01dpa, 0.1dpa and 1dpa at room temperature, TDS experiments were performed. As shown in Fig.3, large peaks A, B and C appeared.

In case of copper ion irradiation, dense defects are accumulated above 0.1dpa. Fig.4 shows micrograph of the pre-thinned tungsten irradiated up to 1dpa. As can be seen in the micrograph, not only dislocation loops but also dense nano-voids are formed. It is considered that very dense single vacancies and their small complex are also accumulated in the sample in addition to the observable defects. Formation of fine vacancy clusters and nano-voids even at low temperatures where vacancies can not migrate thermally is very important from the standpoint of tritium inventory. In case of irradiation with high energy heavy ions and neutrons, in which cascade collisions occur often, fine vacancy clusters are formed even at low fluence and at low temperature.

Mechanically deformed tungsten has also desorption peak C at 850 K, because the vacancies and their fine clusters are formed by the crossing of dislocations under the plastic deformation. To diminish the strong trapping of hydrogen isotope at peak C, the mechanically deformed tungsten should be annealed well above 1223 K.

From the experimental results of TDS and TEM we assign the trapping site of each desorption peaks as followings:

<u>Peak A $(330 \sim 420 \text{K})$ </u>: In addition to adsorption at the surface, single vacancies and their very fine clusters act as trapping for D.

<u>Peak B (420~560K)</u> : Kinks of dislocations is one of the candidate of the corresponding trapping sites.

<u>Peak C (660~900K)</u> : Very fine vacancy complex and nano-voids for D_2 . Trapping energy was estimated to be 1.8eV in the previous work.

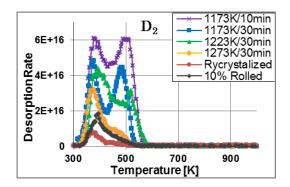


Fig.1 Thermal desorption of D2 from the annealed samples.

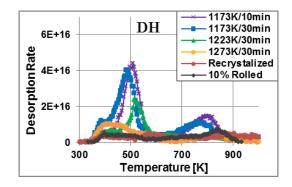


Fig.2 Thermal desorption of DH from the annealed samples.

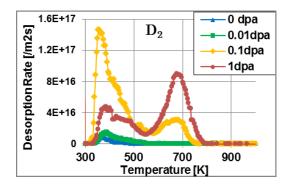


Fig.3 Thermal desorption of D_2 from the Cu ion irradiated samples.

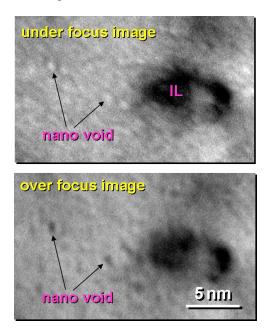


Fig.4 Nano-voids and dislocation loops formed by copper ion irradiation