Journal of Nuclear Materials 502 (2018) 289–294

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Helium retention behavior in simultaneously He^{+}-H_2^{+} irradiated tungsten

Qilai Zhou [a,*], Keisuke Azuma [b], Akihiro Togari [b], Miyuki Yajima [c], Masayuki Tokitani [c], Suguru Masuzaki [c], Naoaki Yoshida [d], Masanori Hara [e], Yuji Hatano [e], Yasuhisa Oya [a]

[a] Faculty of Science, Shizuoka University, Shizuoka, 422-8529, Japan
[b] Graduate School of Science, Shizuoka University, Japan
[c] National Institute for Fusion Science, Oroshi, Toki, Gifu, 509-5292, Japan
[d] Research Institute for Applied Mechanics, Kyushu University, Kasuga, Fukuoka, 816-8580, Japan
[e] Hydrogen Isotope Research Center, University of Toyama, 930-8555, Japan

HIGHLIGHTS

- High temperature TDS (~1773 K) was used to study He retention in simultaneously He^{+}-H_2^{+} irradiated W.
- Growth of He bubbles was significantly suppressed 1.0 keV H_2^{+}.
- He retention was enhanced when the H_2^{+} energy was higher due to the additional damage.

ARTICLE INFO

Article history:
Received 25 November 2017
Received in revised form
21 February 2018
Accepted 22 February 2018
Available online 24 February 2018

Keywords:
Simultaneous irradiation
He retention
TDS
Tungsten
Hydrogen isotope

ABSTRACT

The purpose of this study is to elucidate helium (He) retention behavior in tungsten (W) under simultaneous He and hydrogen (H) irradiation. Polycrystalline-W was irradiated by He^{+} and H_2^{+} simultaneously with the energy of 1.0 keV and 3.0 keV. He^{+} fluences were (0.5, 1.0, 10) × 10^{21} He^{+} m^{-2} and H_2^{+} fluence was 1.0 × 10^{22} H^{+} m^{-2},respectively. After irradiation, He desorption behavior was investigated by high temperature thermal desorption spectroscopy (HT-TDS) in the temperature range of R.T.-1773 K. Micro-structure changes of W after irradiation were observed by TEM. It was found that simultaneous irradiation with different H_2^{+} energy significantly changed He retention behavior. 1.0 keV H_2^{+} suppressed the He bubble growth and no bubbles can be observed at room temperature. On the other hand, 3.0 keV H_2^{+} facilitated the formation of He bubbles and increased the He retention due to the additional damage introduction by energetic H_2^{+}.

1. Introduction

Due to its low hydrogen isotope retention and high melting point, tungsten (W) is considered as the candidate plasma-facing materials (PFMs) for the ITER divertor and other fusion devices [1–3]. In ITER, divertor materials will be exposed to high density ions irradiation with high fluence and low energy ranging from 10 eV to several keV [4]. In the recent long-pulse He discharges in Large Helical Device (LHD), the ion flux of typical divertor plasma were measured to be ~10^{23} ions $m^{-2}s^{-1}$ and on the other hand, the flux and energy of the incident particles on the first-wall surface would be ~2 keV and ~10^{19} ions $m^{-2}s^{-1}$ [5]. As tritium (T) and deuterium (D) will be used as the fuels in the fusion reactor, irradiation of these energetic ions on W lead to the retention of T in W [6–8]. Since T resources are very limited and radioactive, T retention results in the fuel cost and causes safety concerns [9]. Understanding of hydrogen retention and recycling is necessary for the design of fusion reactor. Recycling is the return rate of the cold hydrogen isotope to the plasma, controlling the fuel rate and lowering the plasma temperature [3]. On the other hand, He is one of the product of D-T fusion reaction: D + T→ He + n. He irradiation results in the growth of He bubbles and surface modification in W, i.e. tungsten fuzz, which significantly changes hydrogen isotope retention behavior [10–13]. In order to clarify the He effect on hydrogen retention, a variety of irradiation experiments on W using plasma devices or ion beams were carried out. Sato et al. [14] found

* Corresponding author.
E-mail address: zhou.qilai@shizuoka.ac.jp (Q. Zhou).

https://doi.org/10.1016/j.jnucmat.2018.02.035
0022-3115/

that large He bubbles were formed near the surface for 3.0 keV He^+ irradiated W at 1173 K with the fluence of 1.0×10^{21} He^+ m^{-2}. It was also considered that the existence of He bubbles results in the decrease of W atom ratio on the surface region, which is responsible for the decease of hydrogen retention [15–17]. Recently, the research result from Baldwin et al. [18] indicated that apart from this effect, the He bubble layers also change the migration energy in W for hydrogen isotope.

To discuss the interaction of He with hydrogen in W, we need more information on He retention behavior. However, most experiments were related to the surface modification or D retention by He irradiation or He-D plasma exposure, systematical study on the He diffusion and retention is rather limited. As observed by Miyamoto et al. [19], that the depth distribution of He bubbles is deeper than the injected range of He ions in D-He plasma exposure. It is believed that He retention behavior would be quite different in the simultaneous irradiation due to the existence of hydrogen isotopes.

To understand the He retention behavior, in the earlier stage, Tokitani et al. [20] performed the 2.0 keV He^+ irradiation on 304SS steel with the flux of 1.0×10^{17} He^+ m^{-2} s^{-1} with various fluences and found that dense He bubbles were formed. Further irradiation in LHD and subsequent thermal desorption spectroscopy (TDS) measurement of SUS316L suggested that He desorption at different temperatures can be roughly divided into three stages according to different trapping sites and most of He desorbed at higher temperature above 1000 K was attributed to the He released from He bubbles [5]. However, with respect to W material, the experimental data of He retention behavior is scarce. Herein, simultaneous He^+-H_2^+ irradiation on W with the ion beam energy of 1.0 keV or 3.0 keV was performed. To study the H effect on He retention behavior at different dose, He fluence was set at 1.0×10^{21} He^+ m^{-2} and 1.0×10^{22} He m^{-2}, and H fluence was set at 1.0×10^{22} H^+ m^{-2}.

The objective of this study is to elucidate the He retention behavior in W, especially He retention behavior in simultaneous He^+-H_2^+ irradiation. The other aim is to reveal He and H interaction using different combination of ion energy, which we hope it could be helpful for the understanding of fuel recycling and plasma interaction with materials.

2. Experimental procedure

2.1. Simultaneous He^+-H_2^+ irradiation

Polycrystalline W (10 mm$^\varnothing$, 0.5mm^t, 99.99% purity from A.L.M.T. Corp. Ltd) samples were prepared from a rod of tungsten under stress-relieved conditions supplied by Allied Tungsten, whose grains were normal to the surface. The samples were polished mechanically to mirror-finish surfaces and cleaned by an ultrasonic bath with acetone, ethanol and de-ionized water for 5 min each. It was preheated at 1173 K for 30 min under ultrahigh vacuum ($<10^{-6}$ Pa) to release the stress introduced during polishing. Thereafter, simultaneous He^+-H_2^+ irradiation and single He^+ irradiation were performed by the triple ion beam device at Shizuoka University [21]. This device consists of a He gun, a D gun (H_2^+ or D_2^+), and a carbon gun, combined with a TDS. The energy of He^+ and H_2^+ beam can be set in the range of 1.0–3.0 keV, while the maximum flux can reach 5.0×10^{18} H^+ m^{-2} or 1.0×10^{18} He^+ m^{-2}. A Faraday Cup was attached beside the sample holder. Before each irradiation, the flux was confirmed by the Faraday Cup and then the sample holder was moved to irradiation position. The temperature was monitored by a thermal couple directly attached to the surface of the sample during irradiation. For the comparison of He retention behavior at different irradiation conditions, He^+ flux was fixed at 1.0×10^{17} He^+ m^{-2} s^{-1} with the fluence of 1.0×10^{21} He^+ m^{-2} during irradiation. Besides, in the case of reproducibility experiment, higher He flux of 1.0×10^{18} He^+ m^{-2} s^{-1} was used to achieve the fluence of 1.0×10^{22} He^+ m^{-2}.

To study the He transportation through or towards the hydrogen layer, different combination of He^+ and H_2^+ with the energy of 1.0 and 3.0 keV were used. Fig. 1 shows the results of SRIM calculation of He^+ and H_2^+ depth distribution in W with different ion energy. 3.0 keV He^+ and 3.0 keV H_2^+ show almost the same depths of about 40 nm. On the other hand, the depths of 1.0 keV He^+ and 1.0 keV H_2^+ are 20 nm. Therefore, the influence of hydrogen depth on He retention behavior can be investigated by using different combination of ion energy.

2.2. High-temperature thermal desorption spectroscopy (HT-TDS) measurement

The HT-TDS measurement was performed from room temperature (R.T.). to 1773 K with the ramping rate of 30 K/min at National Science Institute for Fusion Science (NIFS). Before each measurement, the quadruple mass spectrometer (QMS) and He mass position were calibrated by the standard He leak bottle with the leak rate of 1.4×10^{-7} Pa m^3 s^{-1} to ensure the accuracy of the data.

2.3. Transmission electron microscope (TEM) observation

Surface of the samples was observed by TEM at Kyushu University. This TEM device can heat the sample to different temperature during observation. Thus, isochronal heating for the irradiated samples before TDS measurement were performed at 573 K −1173 K to observe the microstructure changes during annealing.

2.4. Glow-discharge optical emission spectroscopy (GD-OES) measurement

He depth profile in the irradiated W was measured by GD-OES with Ne plasma at University of Toyama. To maximize the intensity of He emission, high pressure (1200 Pa) and high power (80 W) were used during the measurement. 7 mm diameter anode was used and the time interval was set at 0.05 s. The sputtering rate was determined by measuring the depth of sputtering crater using a surface profiler (Tokyo Seimitsu SURFCOM 1500DX). The detail of the measurement of described in Ref. [23].

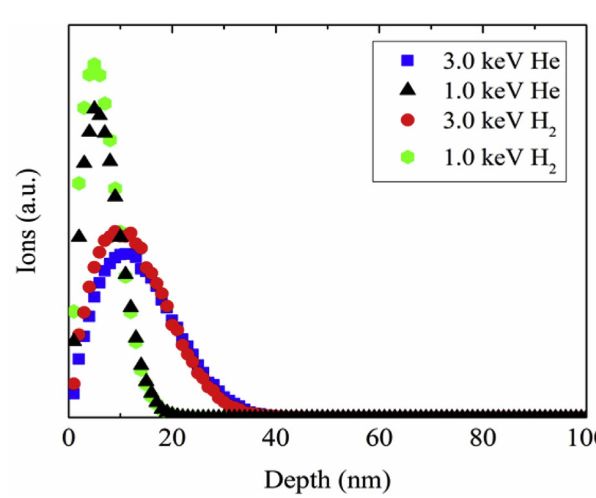

Fig. 1. Ion depth profile calculated by SRIM code [22].