

## Systematic Observation of the Formation Behavior of Helium Bubbles in Tungsten

タングステン中のヘリウムバブル形成挙動の系統的観察

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The formation of helium bubbles in tungsten was systematically investigated using the low energy ion irradiation device and the liner plasma simulator PISCES-A at various conditions. The size, density and depth profile of helium bubbles at near surface region were summarized as a function of ion energy, fluence and temperature. TEM observations after thinning exposed W samples with FIB revealed that the layer thickness (>30 nm) of He bubbles largely exceeds the ion implantation range of a few nm. The size of He bubbles was found to increase with an increase in the sample temperature: it was around 10 nm at 973 K, while only small He bubbles of 1-2 nm were observed at <773 K.

### 1. Introduction

In the ITER DT phase, one should take into account the effects of helium because it is well known that helium atoms have much stronger effects on material damage than hydrogen atoms. Recent experiments using tungsten (W) have shown that helium-related defects such as bubbles and nano-structures due to helium irradiation have great impacts on surface properties [1,2]. The formation and growth behavior of helium bubbles is an important key factor to determine the surface property in W. However, a systematic study on the formation and growth process of He bubbles at various conditions is not sufficient and the mechanism remains obscure. Therefore, in this study, the detailed information of He bubbles including size, density and depth profile were systematically investigated with cross-sectional microstructure observation.

### 2. Experimental

To simulate a divertor plasma condition, W samples were bombarded with low energy (~60 eV), high flux (~ $10^{22}$  m<sup>-2</sup>s<sup>-1</sup>) helium ion in the linear divertor plasma simulator PISCES-A [3] at several temperature (523-973 K). Pure-He plasma and He+D mixture plasma were used to obtain high and low helium ion flux, respectively. The fluence of helium ions,  $\Phi_{\text{He}}$ , was varied in the range of  $1 \times 10^{23}$  to  $1.5 \times 10^{26}$  m<sup>-2</sup>. In addition, to obtain information

on the initial formation behavior, in-situ observations during He ion irradiation with 3 keV and low flux (~ $5 \times 10^{17}$  m<sup>-2</sup>s<sup>-1</sup>) at temperature from R.T. to 1273 K were also performed using a transmission electron microscope (TEM) with a controlled ion gun.

Subsequent to the irradiation, the samples were thinned by Ion-slicer (EM-09100IS) and FIB, and was observed in TEM (JEOL-JEM2010).

### 3. Results

Fig. 1 shows SEM images and cross-sectional TEM images of the W samples exposed to He and He+D mixture plasmas at various conditions.

As shown in SEM images, dense step structures appear on the samples exposed to the plasmas at  $\Phi_{\text{He}} > 5 \times 10^{23}$  m<sup>-2</sup> and increase with an increase in the fluence. In addition to the step structures, small pores were observed on the sample surfaces exposed at  $T_s > 923$  K.

Cross-sectional TEM observations provided information closely related to the surface structures. High-density and fine helium bubbles are observed at the near surface region in the sample exposed to  $\Phi_{\text{He}} > 5 \times 10^{23}$  m<sup>-2</sup>. The damage layer with helium bubbles extends from the surface to several tens of nm in depth, which is much deeper than the helium ion range of about 2 nm for  $E_i \sim 50$  eV. At  $T_s > 923$  K, a decrease in the density and a rather wide size distribution, ranging from ~1 nm to more than 10

nm, were observed. The surface pores observed in the SEM images occur due probably to the migration of these large helium bubbles to the surface.

Fig. 2 shows the microstructural evolution of W under irradiation with 3 keV helium ions at constant temperatures of 293, 773, 1073, and 1273 K. With increasing the helium fluence, more helium bubbles tend to form at all irradiation temperature. The bubbles begin to be observed at lower fluence with increasing irradiation temperature. This may be due to easier nucleation by a higher mobility of vacancies and/or helium-vacancy complexes at higher temperatures. The bubble size for samples irradiated below 1073 K shows no rapid growth and remains in the range of 1-2 nm, even at high fluences. For these samples, the bubble densities also exhibit saturation, and are estimated to be  $1.4 \times 10^{17} \text{ m}^{-2}$  for samples irradiated at 293 and 773 K and to be  $3.0 \times 10^{16} \text{ m}^{-2}$  at 1073 K. However, the sample irradiated at 1273 K shows different behavior: a continuous increase in the size and a rollover in the density at the fluence of  $\sim 4 \times 10^{19} \text{ m}^{-2}$ . This is explained by observations of active migration of small helium bubbles and their coalescence.

As for the difference in temperature, while the grown bubbles are observed at  $T_s \sim 923 \text{ K}$  for the samples exposed to He plasma, they are rarely observed even at  $T_s \sim 1073 \text{ K}$  for the samples irradiated with 3 keV  $\text{He}^+$  ions. Since the damage region is very thin, it is unlikely that the temperature at the near-surface region locally reaches a higher temperature than the  $T_s$ . It could be inferred that the concentration of He in the near-surface region of the samples exposed to He plasmas would be much higher than that for the sample irradiated with the ion beam, and therefore

the bubble growth may be much faster at a given temperature for the sample exposed to the plasmas.

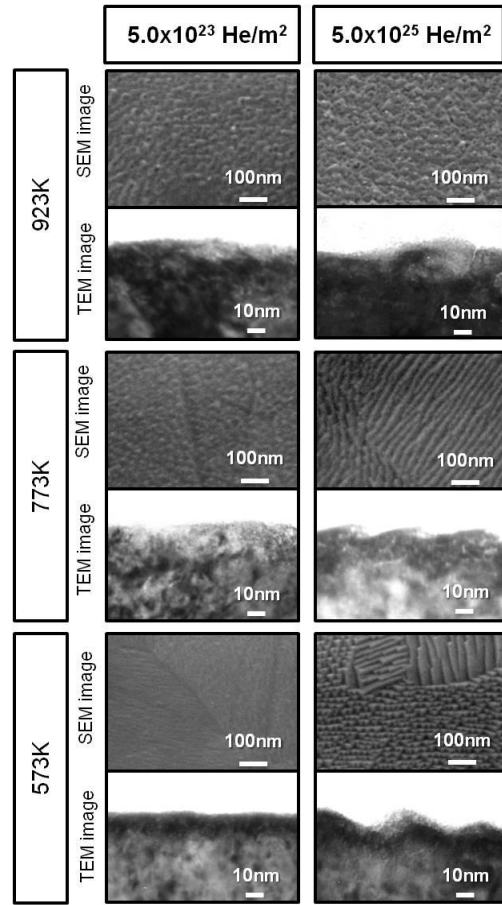


Fig.1. SEM images and cross-sectional TEM images of W samples exposed to plasmas in PISCES-A.

**References**

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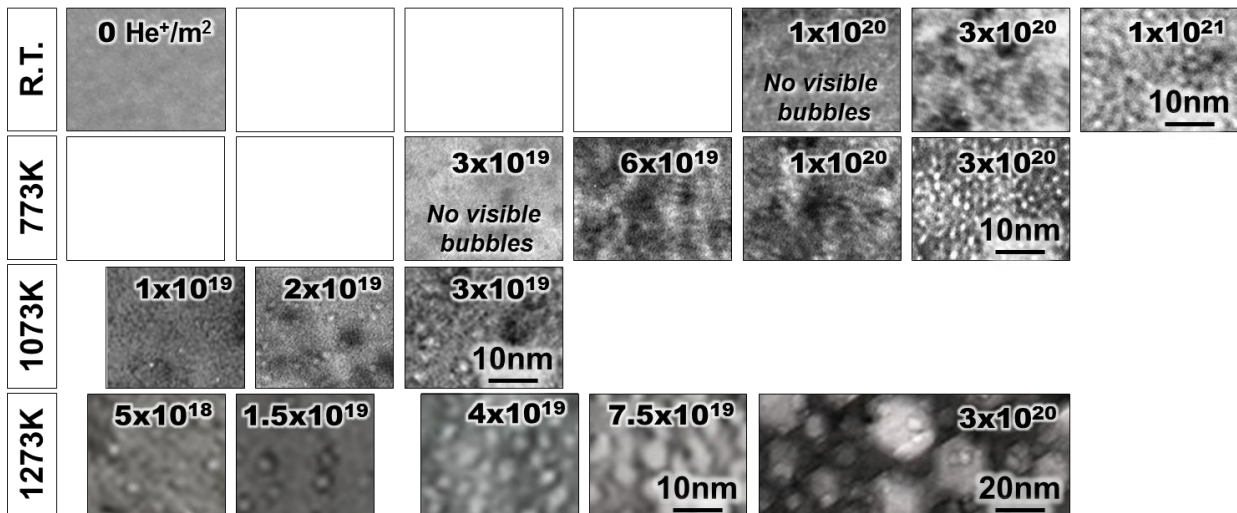


Fig.2. The microstructural evolution of W under 3 keV- $\text{He}^+$  irradiation at several constant temperatures.