

## Modifications of Tungsten Irradiated by Low Energy and High Flux Helium Plasma

YE Minyou\*, FUKUTA Shinya, OHNO Noriyasu, TAKAMURA Shuichi,  
TOKUNAGA Kazutoshi<sup>1</sup> and YOSHIDA Naoaki<sup>1</sup>

*Department of Energy Engineering and Science, Graduate School of Engineering  
Nagoya University, Nagoya 464-8603, Japan*

*<sup>1</sup>Research Institute for Applied Mechanics Kyushu University, Fukuoka 816-8580, Japan*

(Received: 18 January 2000 / Accepted: 11 May 2000)

### Abstract

Experiments were performed in a linear divertor simulator (NAGDIS-I) to study the change of properties of tungsten surfaces irradiated by a low ion energy ( $<50$  eV) and high ion flux ( $>10^{22}$  m<sup>-2</sup>s<sup>-1</sup>) helium plasma. The tungsten plate was irradiated by a steady state helium plasma at various substrate temperature. A surface micro-undulation was formed, and many holes of 0.1–1  $\mu$ m in diameter appeared on the tungsten surface. It was found that the microscopic structural changes of the tungsten surface are obviously dependent on the incident helium ion fluence and the temperature of the tungsten surface. In addition, The changes in light reflectivity of tungsten surface are discussed. It seems that the absorption of light will be enhanced due to the change of the microstructure of the tungsten surface.

### Keywords:

divertor plasma simulator, helium plasma, tungsten, bubble formation, microstructure, plasma-surface interaction

### 1. Introduction

High Z materials like tungsten have been considered to be a good candidate for the material in the divertor region of ITER [1-2]. Therefore, studies of the plasma-tungsten interactions become more and more important for the design and operation of ITER. However, systematic investigations on tungsten material have been lacking. Previous investigations of tungsten were focused on the use of energetic ion beams at a very high energy range of 1–300 keV and at room temperature. However, the low divertor plasma temperatures are currently envisioned for ITER and the bombarding ions will have a broad energy distribution down to a very low energy. Thus, there has an interest to study the change of properties of tungsten due to low ionic energy and high flux plasma irradiation [3-6].

Helium bubble and blister formation in tungsten was reported in high energy H and He ion beam studies [7,8]. We have already reported about microstructure changes, such as bubble and hole formation, on the tungsten surface irradiated by low ion energy ( $<100$  eV), high ion flux ( $>10^{22}$  m<sup>-2</sup>s<sup>-1</sup>) steady-state helium plasmas at high surface temperature [3]. The present study represents further experimental results on modifications of tungsten surface under low energy and high flux helium plasma irradiation at various target plate temperatures. It is reported that the bubble and hole structure of the tungsten surface has a clear dependence on the incident helium ion fluence and on the temperature of the tungsten surface. In addition, the optical properties of the tungsten surface are found to be

\*Corresponding author's e-mail: minyou@nuee.nagoya-u.ac.jp

changed. The light reflectivity is decreased with the increase of the ion fluence, which means that the light absorption is enhanced due to the change of the microstructure of the tungsten surface.

## 2. Experimental Setup

The experiments have been performed in the linear plasma device Nagoya University Divertor Simulator (NAGDIS-I), in which a high heat flux steady-state plasma is generated by Philips Ionization Gauge (PIG) discharge with helium, hydrogen and argon as working gases [9,10]. The plasma is confined by means of an axial magnetic field up to 0.15 T with a large diameter of 12 cm. The helium plasma density reaches  $5 \times 10^{18} \text{ m}^{-3}$  in steady-state. The electron temperature measured with a Langmuir probe was 5 to 15 eV, but the ion temperature is assumed to be much lower than the electron temperature. Therefore, the incident energy of ions is determined by the potential difference between the plasma and the target.

In each irradiation experiment, five pure tungsten samples ( $25 \times 25 \times 0.1 \text{ mm}$ ) were installed on a tantalum plate which was mounted on a movable stage and positioned normally to the magnetic field. The tungsten samples were treated by means of supersonic cleaning before mounting. The tungsten sample is about 2 m away from the plasma source. The diameter of the plasma was limited to 2.5 cm by a tungsten limiter at about 10 cm upstream from the tungsten sample. The target temperature was controlled by means of varying the plasma density. The incident fluences were changed by variation of the irradiation time with a constant ion flux. The surface temperature was monitored through a vacuum window with an infrared thermometer in the wavelength range of 0.8–1.1  $\mu\text{m}$ . Plasma parameters at one cross section were measured by a reciprocating fast scanning Langmuir probe set about 0.2 m upstream from the tungsten target. Surface analysis was made later by means of scanning electron microscopy (SEM).

## 3. Results and Discussions

### 3.1 Bubble and hole formation

In a previous publication [3] it has been reported about bubble and hole formation in tungsten under low energy and high flux helium plasma irradiation at temperature up to nearly 3200 K. In the present study we show bubble and hole formation in tungsten surfaces as functions of ion fluence and sample temperature.

### A. Dependence on incident fluence

We fixed the plasma parameters and the sample temperature (1020 K), and obtained the dependence of incident fluence by varying the irradiation time. The biasing voltage of the sample was  $-50 \text{ V}$  with respect to the vacuum chamber. Figure 1 shows SEM pictures of the tungsten surface after the helium plasma irradiation with two different incident fluences of  $6.8 \times 10^{25} \text{ m}^{-2}$  and  $1.4 \times 10^{26} \text{ m}^{-2}$ . The corresponding constant ion flux was  $1.4 \times 10^{22} \text{ m}^{-2}\text{s}^{-1}$ , and the ion energy  $\sim 50 \text{ eV}$ . No obvious changes of surface microstructure was observed at fluences below  $6.8 \times 10^{25} \text{ m}^{-2}$ , as shown in Fig. 1a. When the fluence was increased to  $1.4 \times 10^{26} \text{ m}^{-2}$ , bubbles started to grow, as shown in Fig. 1b. For a higher temperature (1770 K), the bubble formation started even below the fluence of  $6.8 \times 10^{25} \text{ m}^{-2}$ . This is shown in Fig. 2. The ion flux was kept at  $2.2 \times 10^{22} \text{ m}^{-2}\text{s}^{-1}$ , the ion energy at  $\sim 45 \text{ eV}$ . It is clearly to see that bubbles and holes were formed in the tungsten surface.

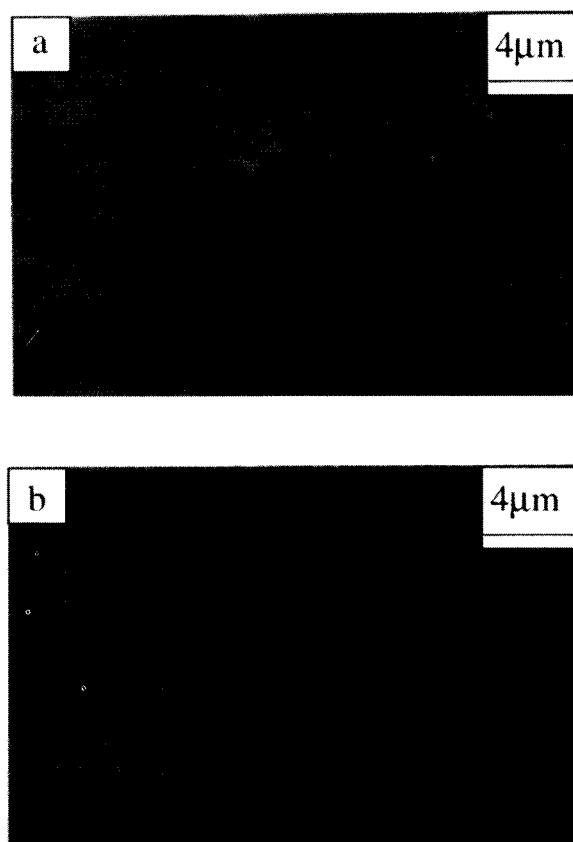


Fig. 1 SEM photographs of tungsten surfaces after the helium plasma irradiation at 1020 K for an incident fluence of (a)  $\sim 6.8 \times 10^{25} \text{ m}^{-2}$  and (b)  $\sim 1.4 \times 10^{26} \text{ m}^{-2}$

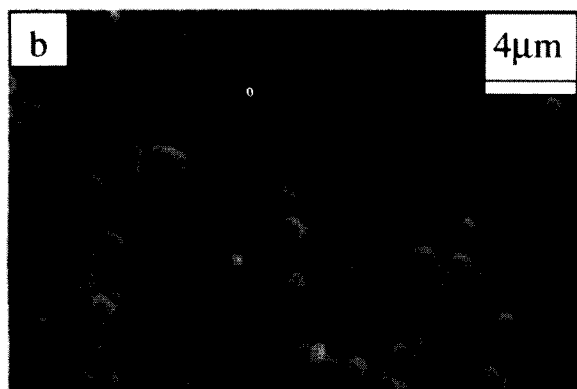


Fig. 2 SEM photographs of tungsten surfaces after the helium plasma irradiation at 1770 K for an incident fluence of (a)  $\sim 2.7 \times 10^{25} \text{ m}^{-2}$ ; (b)  $\sim 5.4 \times 10^{25} \text{ m}^{-2}$  and (c)  $\sim 1.1 \times 10^{26} \text{ m}^{-2}$ .

The size of bubbles and holes becomes larger with increasing fluence. The density of bubbles and holes are also increased. The dune-like relief exhibits many holes of about  $1 \mu\text{m}$  in diameter which appear in the tungsten surface as shown in Fig. 2c.

In the case of a higher surface temperature of 1770 K, the holes were observed on the surface at the fluence



Fig. 3 SEM photograph of tungsten surface after the helium plasma irradiation at 1470 K for an incident fluence of (a)  $\sim 1.5 \times 10^{26} \text{ m}^{-2}$ .

down to  $2.7 \times 10^{25} \text{ m}^{-2}$ , as shown in Fig. 2a. Such bubbles and holes formation on the tungsten surface has been found to have dependence of not only the ion fluence, but also the surface temperature.

#### B. Dependence of surface temperature

The dependence on the surface temperature can be seen by comparing Fig. 1b (1020 K), Fig. 3 (1470 K) and Fig. 2c (1770 K). The three samples were irradiated with almost the same ion fluence in the range of  $1.0 \times 10^{26}$ – $1.5 \times 10^{26} \text{ m}^{-2}$ . We can see that the sizes of bubble and hole in the high temperature are larger than that in the low temperature.

Helium ion sputtering is not responsible for such a structural change because of the negligible sputtering rate of tungsten ( $<10^{-4}$ ) by such a low energy helium ions. Although the mechanism in bubble formation on metal surfaces was discussed in early studies [11], the formation mechanism is not still yet fully understood. A possible reason for our experimental results is considered as follows, the implanted He ions will generally diffuse in all directions. Because of the low ion energy, no vacancies are produced by the ion bombardment. Helium ions are trapped dominantly at intrinsic vacancies as well as at grain boundaries. At low incident fluence corresponding to low concentrations of gas, atoms are trapped by vacancies and no major structural changes are observed. As the incident fluence increases up to a critical value, the helium gas starts to form stable bubbles near the surface. It seems that the critical incident fluence for bubble and hole formation has obviously a dependence of the target temperature. The target temperature is key parameter to determine the diffusion of He atoms in tungsten. The diffusivity of He

atoms in perfect crystals of tungsten has been studied employing the atom-probe field-ion microscope technique (FIM), and is given by the Arrhenius expression [12].

$$D(T) = \left( 5.4 \pm_{3.8}^{10.6} \right) \times 10^{-3} \exp(-0.28 \text{ eV}/kT) \text{ cm}^2\text{s}^{-1}.$$

Which indicates that the diffusivity is very sensitive to the target temperature.

At low temperatures, the diffusion rate is low and vacancies have no thermally mobile. Therefore, the critical incident fluence for bubble formation must be high. As the temperature increases, such a critical value of the incident fluence decreases since the diffusion is enhanced and vacancies are thermally mobile. Then, the size of bubbles becomes large. As to hole formation it is believed that the high gas pressure of bubbles at high surface temperature makes a sudden break-up of bubble surface.

In addition, impurity atoms in W may also act as trapping centers for He atoms. Many He atoms form bubbles by ejecting W atoms from their lattice site. Further He atom injection makes another ejection of W atoms to form a bigger bubble [6].

### 3.2 Change in light reflectivity

One of characteristic changes of tungsten surfaces irradiated by low energy and high flux helium plasma is a reduction of light reflectivity of the tungsten surface.

Figure 4 shows the relative light reflectivity from UV through visible range in the case of mirror reflection at incident angle of 45°. The solid line shows the reflectivity for a virgin tungsten sample, while the broken lines indicated by 1, 2 and 3 show the reflectivity, corresponding to the case of Fig. 2a, Fig. 2b and Fig. 2c, respectively. We can see that the light reflectivity in the case of Fig. 2c is about 2%. This is due to the change of microstructure of tungsten surface. In this case we can not measure a correct absolute light reflectivity including the diffusive scattering because of a problem of measurement. However, the decrease of light reflectivity means that the light absorption is enhanced. This was confirmed by examining an surface temperature increase due to a pulsed Ruby-Laser irradiation with an energy of 31 J, as shown in Fig. 5. The difference of the temperature increase between a virgin tungsten surface and a tungsten surface irradiated by helium plasma was found to be 200 K. Estimation of the light absorption is given by means of a comparison of calculation of the surface temperature with

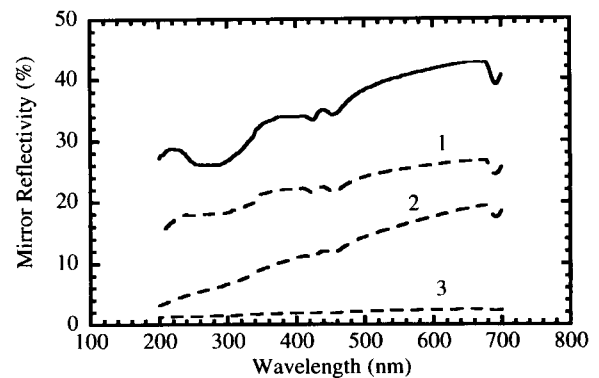


Fig. 4 The solid line shows the reflectivity for a virgin tungsten surface, while the dashed lines indicated by 1,2 and 3 show the reflectivity, corresponding to the case of Fig. 2a, Fig. 2b and Fig. 2c, respectively.

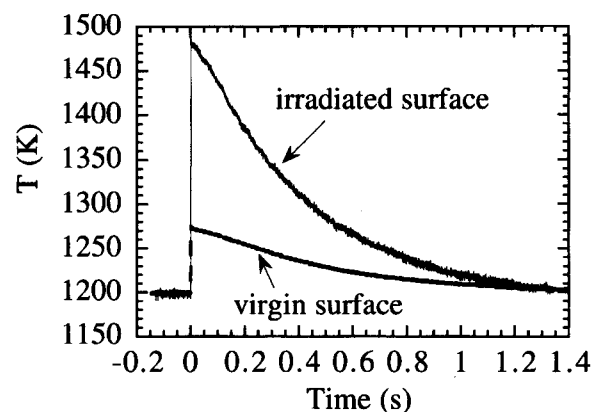


Fig. 5 Surface temperature increase by a pulsed Ruby-Laser irradiation with energy of 31 J on virgin tungsten surface and tungsten surface irradiated by helium plasma.

experimental results. It indicated that the difference of the temperature of 200 K corresponds to about three times change of the light absorption. The enhancement of the light absorption may be due to many cavities generated on the tungsten surfaces irradiated by helium plasma.

### 4. Summary

In the previous section we have described some characteristics changes of tungsten surface irradiated by the low energy and high flux helium plasma. We summarize the conclusions of this study as follows:

Bubble and hole formations are clearly observed in the pure tungsten surface under a low energy (<50 eV)

and high flux ( $>10^{22} \text{ m}^{-2}\text{s}^{-1}$ ) helium plasma irradiation. For a constant ion flux with a relative high surface temperature, the surface microstructures exhibit increased undulation, increased size of bubbles and holes and increased density of bubbles and holes as the fluence increases.

For a constant fluence, the size of bubbles and holes become large as the surface temperature increased. It seems that the surface temperature is a key parameter for hole formation in surface. The dune-like relief exhibits many holes of about  $1\mu\text{m}$  in diameter which appear on the tungsten surface at the fluence of  $1.1 \times 10^{26} \text{ m}^{-2}$  and the surface temperature of 1770 K.

The optical properties of the tungsten surface are found to be changed after helium plasma irradiation. The light absorption is enhanced due to the change of microstructure of the tungsten surface. This result may indicate the radiation coming from the plasma and impurities should be considered in divertor plate irradiated by helium contained plasmas in terms of surface temperature increase.

This studies indicate that such a bubble process due to low energy helium particles irradiation can also make the contribution to impurity introduction and wall erosion in D-T burning fusion devices and reactors. It also produces a great impact on the hydrogen retention. For a future work, we will study the tungsten surface

characteristics under low energy and high flux hydrogen plasma and hydrogen-helium mixing plasma irradiation.

### Acknowledgments

The authors would like to thank Dr. Y. Uesugi, Dr. K. Sato and Prof. N. Noda for their discussions, and M. Takagi for his technical assistance.

### References

- [1] G. Janeschitz, *J. Nucl. Mater.* **220-222**, 73 (1995).
- [2] N. Yoshida, *J. Nucl. Mater.* **266-269**, 197 (1999).
- [3] M.Y. Ye *et al.*, *J. Nucl. Mater.* **241-243**, 1243 (1997).
- [4] Fan C. Sze *et al.*, *J. Nucl. Mater.* **264**, 89 (1999).
- [5] Fan C. Sze *et al.*, *J. Nucl. Mater.* **266-269**, 1212 (1999).
- [6] H. Iwakiri *et al.*, to be published in *J. Nucl. Mater.*
- [7] S.T. Picraux *et al.*, *J. Nucl. Mater.* **53**(1), 246 (1974).
- [8] P.B. Johnson *et al.*, *J. Nucl. Mater.* **218**, 273 (1995).
- [9] S. Masuzaki *et al.*, *Jpn. J. Appl. Phys.* **29**, 2835 (1990).
- [10] S. Masuzaki *et al.*, *J. Nucl. Mater.* **223**, 286 (1995).
- [11] J.H. Evans, *J. Nucl. Mater.* **76&77**, 228 (1978) and references in this article.
- [12] Jun Amano *et al.*, *J. Appl. Phys.* **56**(4), 983 (1984).