



# Incident ion energy dependence of bubble formation on tungsten surface with low energy and high flux helium plasma irradiation

D. Nishijima <sup>a,\*</sup>, M.Y. Ye <sup>b</sup>, N. Ohno <sup>a</sup>, S. Takamura <sup>a</sup>

<sup>a</sup> Department of Energy Engineering and Science, Graduate School of Engineering, Nagoya University, Nagoya 464-8603, Japan

<sup>b</sup> Max-Planck-Institut für Plasmaphysik, EURATOM Association, Boltzmannstr. 2, D-85748 Garching, Germany

## Abstract

Tungsten (W) specimens were irradiated by low energy (<65 eV) and high flux ( $\sim 10^{22-23} \text{ m}^{-2} \text{ s}^{-1}$ ) helium plasma to investigate the incident ion energy dependence of helium bubble formation. Experimental results indicate the existence of the incident ion energy threshold for the bubble formation. The threshold energy around 15 eV could associate with the surface potential for He ions entering to the W surface.

© 2003 Elsevier Science B.V. All rights reserved.

PACS: 52.40.Hf

Keywords: Tungsten; Helium; Bubble; High flux; NAGDIS-II

## 1. Introduction

Impurity generation and gas retention in plasma facing components are the most important issues in plasma–surface interactions in fusion devices. Tungsten (W) and its alloys have been considered to be candidates as the plasma facing material for next-generation fusion devices such as ITER.

In order to reduce the particle and heat flux onto the divertor plate in ITER, a partially detached plasma has been considered in the divertor region, in which the divertor will be operated under a ‘low temperature and high density’ plasma condition [1]. In such a plasma condition, it has been expected that there would be no erosion of the plasma-facing materials, especially for high *Z* materials. The incident energy of hydrogen and/or helium ions, which is determined by the sheath volt-

age, is certainly lower than the threshold energy of sputtering. However, it has been reported recently that a lot of bubbles were clearly observed on the W surface irradiated by He ions with a high flux ( $\sim 10^{22} \text{ m}^{-2} \text{ s}^{-1}$ ) and a high fluence ( $\sim 10^{26} \text{ m}^{-2}$ ), even at a low incident energy (<50 eV) [2,3]. The mechanism of the bubble formation has not been understood yet.

In this paper, we have investigated the incident ion energy dependence of the bubble formation on the incident He ion energy when the surface is irradiated by the helium plasma. Such an energy dependence has never been studied, especially the presence of threshold energy in such a low energy range. It will contribute to fundamental understanding on the mechanism of the bubble formation. Experimental results show that the threshold value of incident ion energy exists for the bubble formation.

## 2. Experimental setup

Specimens were powder metallurgy W disks (Nilaco Co.), and their purity are 99.95%, 3–5 mm in diameter,

\* Corresponding author. Tel.: +81-52 789 3145; fax: +81-52 789 3944.

E-mail address: [dai-nishijima@ees.nagoya-u.ac.jp](mailto:dai-nishijima@ees.nagoya-u.ac.jp) (D. Nishijima).

and 0.2 mm in thickness. We did not perform any heat treatment of the surface preparation such as annealing. Specimens were cleaned with ultrasonic conditioning.

Nine specimens were irradiated by the helium plasma using a linear divertor plasma simulator, Nagoya University Divertor Plasma Simulator (NAGDIS-II), which can generate a high density plasma ( $\geq 10^{19} \text{ m}^{-3}$ ) in steady state with the TP-D type discharge [4]. Fig. 1 shows a schematic illustration of experimental setup. The W disk was mounted at the head of the movable probe system, and was electrically biased with respect to the vacuum chamber to control the incident ion energy.

The single probe measurement was carried out with the W disk to obtain the  $I$ - $V$  characteristic. Ion saturation current  $I_{\text{is}}$  to the W disk, electron temperature  $T_e$  and floating potential  $V_f$  can be obtained from the  $I$ - $V$

characteristic. The incident ion flux  $\Gamma$  was estimated from the following equation:

$$I_{\text{is}} = e\Gamma S,$$

where  $I_{\text{is}}$  is the measured ion saturation current,  $e$  is the electric charge,  $S$  is the surface area of the specimen. Plasma potential  $V_p$  was estimated from  $T_e$  and  $V_f$  by using the equation,  $V_p = 4.0T_e + V_f$  [5].

The incident ion energy  $E_{\text{in}}$  during the plasma irradiation is determined from the difference between the plasma potential  $V_p$  and the biasing potential of the W disk  $V_b$ . It should be noted that the probe measurement was made within a very short time before the irradiation. The W disk was heated by the incident plasma heat flux. The surface temperature  $T_s$  was measured through the Pyrex window with the radiation thermometer as shown in Fig. 1. Table 1 shows the plasma parameters in the experiments. After the irradiation, scanning electron microscopy (SEM) analysis was performed on each specimen.

### 3. Experimental results and discussion

Fig. 2 shows SEM micrographs for eight specimens designated as A–H. The incident ion energy  $E_{\text{in}}$  is controlled from 64 to 1 eV by changing the biasing voltage. The surface temperature  $T_s$  determined by plasma heat flux onto the specimen is varied from 1850 to 2850 K. These  $T_s$  are higher than re-crystallized temperature for W ( $\sim 1500$  K). The fluence range from  $0.5 \times 10^{26} \text{ m}^{-2}$  to  $2.1 \times 10^{26} \text{ m}^{-2}$  as shown in Table 1. Many bubbles and holes can be seen in the specimens A, B and C. On the contrary, no bubble is observed in the specimens E, F, G and H, but grain boundaries are clearly seen. The specimen D seems to be the intermediate state between the former and the latter. Furthermore, the traces of the arc discharge were seen partially in the specimens D, E, H. Though SEM micrograph of specimen I is not shown in Fig. 2, the surface condition is similar to those of G

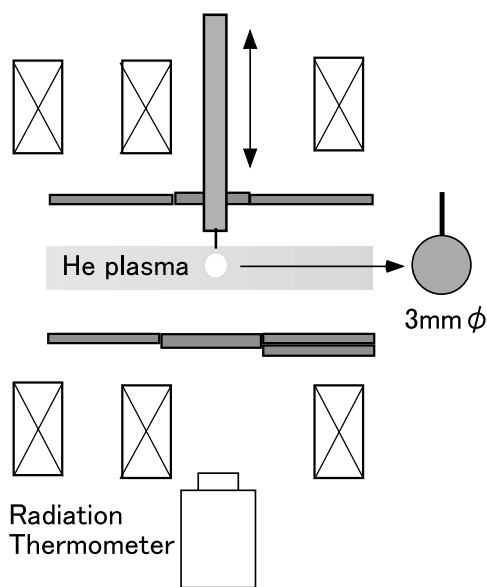


Fig. 1. Experimental setup.

Table 1  
Specimen designation and parameters used in the experiments

Specimen name	Incident ion energy (eV)	Surface temperature (K)	Fluence ( $10^{26} \text{ m}^{-2}$ )	Flux ( $10^{23} \text{ m}^{-2} \text{ s}^{-1}$ )	Irradiation time (s)
A	64	2050	0.5	0.6	720
B	60	2300	1.5	2.6	600
C	26	1950	1.7	2.6	660
D	18	2250	1.3	2.2	600
E	12	2300	2.1	3.5	600
F	9	1850	0.5	0.9	600
G	7	2850	1.4	1.2	1200
H	6	2250	2.0	0.5	4200
I	1	1850	0.6	0.1	4200

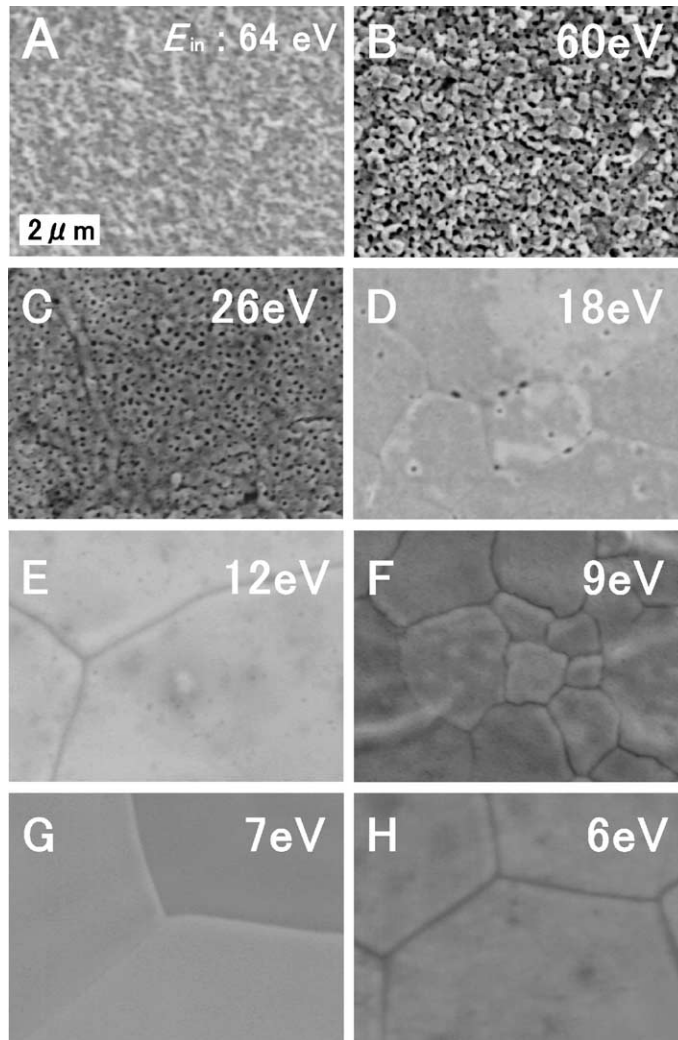


Fig. 2. SEM micrographs of specimen A–H. All pictures have same magnification.

and H. Bubbles and holes are created with the incident ion energy above 18 eV, but are not formed with energy below 12 eV. It seems that these experimental results indicate that there exists a threshold value of the incident ion energy for the bubble formation around 15 eV. Fig. 3 shows experimental conditions with and without the bubble formation. Closed circles represent the experimental parameters where the bubbles are formed. Since the floating potential of the W disk  $V_f$  was fluctuated in time with a peak-to-peak amplitude of  $\sim 5$  V,  $E_{in}$  could also fluctuate. The plasma potential  $V_p$  was estimated from the floating potential  $V_f$  and the electron temperature  $T_e$  as mentioned before, the  $E_{in}$  determined by the  $V_p$  and the biased voltage of the W disk  $V_b$  has the error around  $\pm 5$  eV.

It is obvious from Fig. 3(a) and (b) that the bubble formation arises in the parameter region bordering on

$E_{in}$  of 12–18 eV. It should be noted that there also exist the threshold values of the fluence and the surface temperature for the bubble formation as found in the previous experiment [2]. The bubbles were observed at the larger fluence than  $10^{25}$   $m^{-2}$  with  $E_{in} \sim 50$  eV and  $T_s \sim 1800$  K. On the other hand, no bubble was formed at  $T_s$  below 1500 K with  $E_{in}$  of 50 eV and the fluence of  $1.5 \times 10^{26}$   $m^{-2}$ . In addition to these thresholds, our experimental results indicate that no bubble is created below the incident ion energy even at sufficient fluences and sufficiently high surface temperature  $T_s$  mentioned in the previous experiment. The surface temperature and the fluence could also be important parameters for the bubble formation. However, there is no clear dependence of these two parameters for the bubble formation in the parameter range concerned in the present experiment.

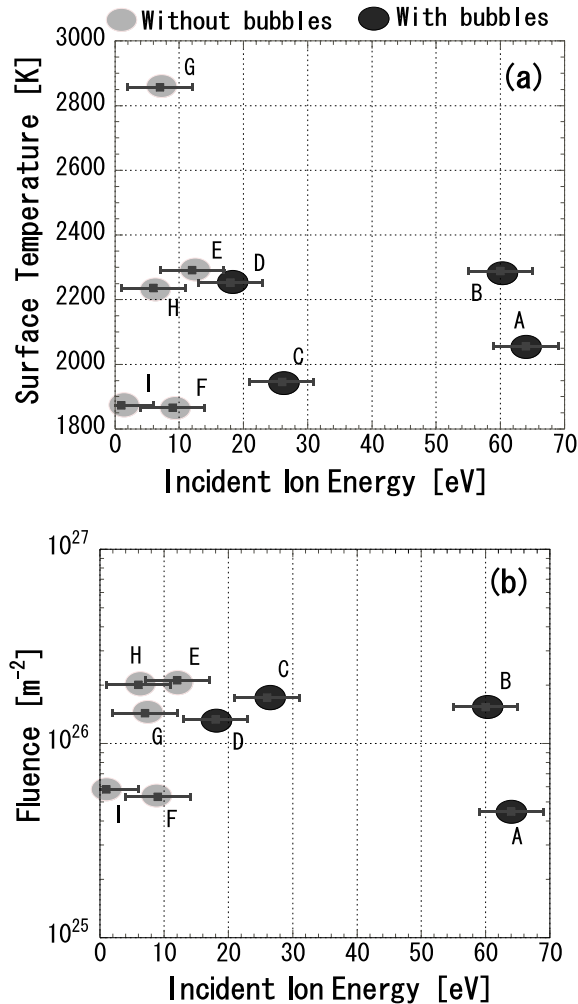


Fig. 3. Summary of experimental conditions with and without bubble formation in the parameter spaces of (a) incident ion energy  $E_{\text{in}}$  and surface temperature  $T_s$ , (b) incident ion energy and fluence.

Fig. 4 shows a cross section of the specimen B irradiated by 60 eV He ions with a dose of  $1.5 \times 10^{26} \text{ m}^{-2}$  at 2300 K. Bubbles are clearly seen within the depth of 1–2  $\mu\text{m}$  from the top surface while the bulk seems to be recrystallized (Fig. 4(a)). An average size of diameter of the visible bubbles is about 0.5  $\mu\text{m}$ . However, He ions with the  $E_{\text{in}}$  of below 100 eV cannot penetrate into micrometer-order depth from the W surface, because the projected range of the He ions is estimated to be below 10  $\text{\AA}$ . It seems that the He contained in the bubbles come from the surface region by a diffusion process. This means that the mobility of He in the W is one of the most important factors for the bubble formation as well as He intrusion in the surface.

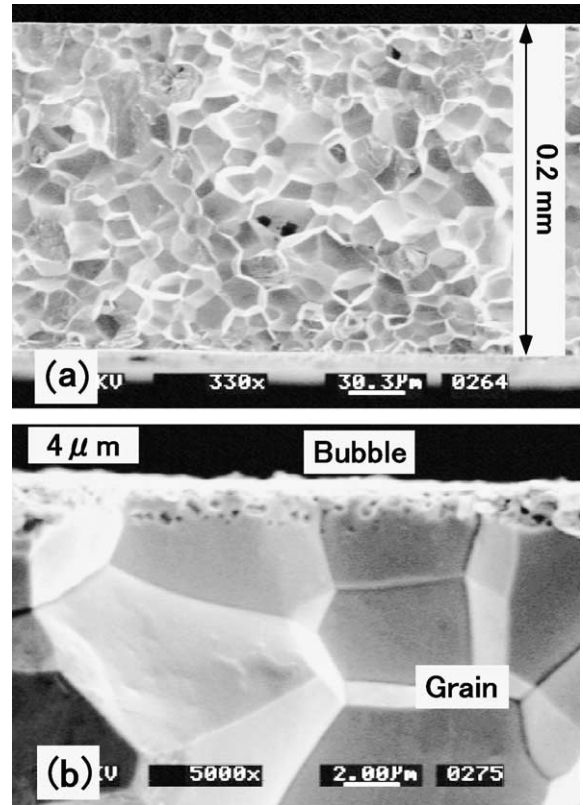


Fig. 4. SEM micrographs of the cross section of specimen B.

The mechanism of the bubble formation in this experiment has not been well understood yet, but we try to discuss the mechanism of the bubble formation from our experimental results.

If there are vacancies as trapped sites in W, many He atoms which diffuse in W can be trapped in the vacancy and become nano-scale bubble because the potential well of the trapped site is very deep for He [6]. It is easier for He atoms in the W to enter into bubble or void rather than to escape from them. Then, many He atoms can be absorbed in the bubbles or voids. An increase in the internal pressure of these trapping sites result in mutation of crystal lattice, leading to an expansion of the bubble. Of course the collision between the small bubbles would also form bigger bubbles or blisters in W.

Three important factors should be considered in the bubble formation: the vacancy concentration, the He concentration and the mobility of the vacancies and He atoms in W.

Origins of the vacancies in this experiment come from the intrinsic defects (vacancy) and thermal vacancy. The defects made by incident helium ions collision with W atoms in solid could not be generated in this experiment because the incident energy of He ions is too low to make displacement defects.

The intrinsic defects exist commonly in all specimens, but its concentration is unclear. Furthermore, many thermal vacancies are born in W at high  $T_s$ . For example, the concentration of the thermal vacancy can be estimated to be  $10^{-9}$  fraction to total W atoms at 2000 K, and  $10^{-6}$  at 3000 K. Though intrinsic defects would disappear due to the effect of annealing at temperature higher than 1500 K (re-crystallized temperature), the irradiation time is too short to disappear intrinsic defects in specimens except for H. Then it can be supposed that there are a lot of vacancies existed in W at high  $T_s$ 's in our experimental condition. Such a high  $T_s$  also provides larger mobility of vacancies and He atoms in W.

From the above discussion, the reason why no bubble forms below a certain  $E_{in}$  even at the huge fluence and high  $T_s$  could be associated with the He concentration in W. We suppose that He ions below the threshold  $E_{in}$  cannot penetrate into the W surface because there is a surface potential barrier on the W for He ions.

There have been few studies on the evaluation of surface potential for He entering into W. However, one theoretical calculation gives 5.5–5.9 eV as a solution energy for He into the W interstitial site [6]. To investigate the discrepancy between our experimental value and this reference value is future work.

#### 4. Conclusion

We have investigated the threshold value of incident ion energy for the bubble formation on powder metallurgy W specimens (purity: 99.95%) irradiated by low energy and high flux helium ions. Bubbles and holes were observed in the specimens irradiated by He ions, whose incident energy is above 18 eV. On the contrary, no bubble was formed at an incident energy below 12

eV. Three important factors should be considered in the bubble formation: the vacancy concentration, the He concentration and mobility of the vacancies and He atoms in W. There are a lot of vacancies existed in the W at high  $T_s$ 's in our experimental condition. But the He concentration would be too low in the specimens irradiated by He ion whose incident energy is below 12 eV. As a result, no bubble was observed in these specimens. The speculation is that He ions below the threshold  $E_{in}$  cannot penetrate from the W surface into the bulk because there is the surface potential barrier on the W for incident He ions. The barrier energy would range between 12 and 18 eV.

#### Acknowledgement

One of authors (D.N.) deeply acknowledges Professor K. Morita of Nagoya University for his fruitful discussion.

#### References

- [1] ITER Physics Basis, Nucl. Fusion 39 (1999) 2137.
- [2] M.Y. Ye, S. Fukuta, N. Ohno, S. Takamura, J. Plasma Fusion Res. Series 3 (2000) 265.
- [3] K. Moriguchi, S. Fukuta, M.Y. Ye, N. Ohno, S. Takamura, Proc. XXV Int. Conf. Phenom. Ionized Gases 1 (2001) 83.
- [4] N. Ohno, D. Nishijima, S. Takamura, Y. Uesugi, M. Motoyama, N. Hattori, H. Arakawa, N. Ezumi, S. Krashennnikov, A. Pigarov, U. Wenzel, Nucl. Fusion 1 (2001) 1055.
- [5] M.A. Lieberman, A.J. Lichtengerg, Principles of Plasma Discharges and Materials Processing, Wiley, New York, 1994.
- [6] H. Ullmaier, Nucl. Fusion 24 (1984) 1039.