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Formation mechanism of bubbles and holes on tungsten surface with low-energy and high-flux helium plasma irradiation in NAGDIS-II

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Abstract

A systematic study on the formation mechanism of micron-sized He bubbles and holes in powder metallurgy tungsten due to helium ion irradiation with an ion energy below 30 eV and a particle flux above 10^{22} $m^{-2}\,s^{-1}$ has been performed in the linear divertor plasma simulator NAGDIS-II. Holes are formed with incident helium ion energy above 5 eV, which could be related to the surface barrier potential energy for He penetrating into tungsten. Tungsten surface temperature strongly influences the number and size of hole. Above 1600 K, bubbles and/or holes with several hundreds nano-meter diameter appear on the tungsten surface. Single crystal tungsten, which has much fewer intrinsic defects than powder metallurgy tungsten, was also irradiated by He plasmas. There is no qualitative difference in the hole formation between the two grades of tungsten. Bubble and hole formation mechanisms are discussed based on the experimental results.

1. Introduction

Tungsten (W) is one of the most important candidate materials for the divertor of ITER [1]. It is desirable to achieve high edge density with low temperature in the divertor region which facilitates power removal by employing partial plasma detachment as an operational regime. In detached plasmas of next generation fusion devices with ignited plasma, like ITER, divertor materials are exposed to high density helium (He) plasma with low temperature as well as hydrogen isotopes (H, D, T) plasmas. Under such conditions, high-Z material like tungsten is considered as a favorable material, due to their low sputtering yield and their high sputtering threshold energy, which may lead to no erosion by He ion irradiation with an ion energy below 100 eV [2]. However, recent experiments have shown that even at such a low energy He ion irradiation causes micron-sized He bubble and hole formations on W surfaces [3–6]. He bubble or hole formation in variety of materials had been observed and studied systematically in the high-energy (KeV–MeV) beam irradiation where the irradiation-induced defects play an essential role [2,7–9]. However, the formation mechanism of hole and He bubble on W surface under low energy (1–100 eV) plasma irradiation without irradiation-induced defects [9] is not yet clearly understood.

In order to understand hole formation mechanism, we have done a systematic experiment to reveal key parameters related to holes in powder metallurgy tungsten in the divertor plasma simulator, NAGDIS-II. In

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this paper, we will report incident He ion energy and tungsten surface temperature dependence of the hole formation. Holes were generated for incident He ion energy above 5 eV. It is also found that higher tungsten surface temperature leads to an increase in the number and the size of hole. Above 1600 K, many holes and bubbles with a several hundreds nano-meter diameter are observed on the tungsten surface. We also studied hole formation on single crystal tungsten, which has much fewer intrinsic defects than powder metallurgy tungsten. There is no qualitative difference in hole formation between them. From these experimental results, hole formation mechanism, including a source of the nucleation, is discussed.

2. Experimental setup

Seven powder metallurgy tungsten (PM-W) samples and one single crystal tungsten (SC-W) sample were irradiated by He plasmas in the NAGDIS-II generating a high density He plasma ($\sim 10^{19}$ m^{-3}) in steady state with the TP-D type discharge [10]. The purity of PM-W and SC-W is 99.95% and their thickness are 0.1–0.2 mm and 1mm, respectively. Samples were cleaned with ultrasonic conditioning before setting up. The SC-W sample was electro-polished prior to the ultrasonic cleaning. In order to control the incident ion energy to the samples, the samples were electrically biased with respect to the vacuum chamber. Detailed configuration of the sample set-up is described in Ref. [5]. The surface temperature (T_s) on the W was measured by a pyrometer through a quartz window. After the plasma irradiation, the samples were analyzed by a scanning electron microscopy (SEM) and a transmission electron microscopy (TEM).

3. Results and discussion

3.1. Dependence of hole formation on the incident ion energy

Fig. 1 shows SEM micrographs of the surface and the cross section of four W samples designated as W1–W4. The size of hole becomes smaller as the incident ion energy E_{in} decreases. It is found that at E_{in} ~5 eV (W3) the number and the size of the hole are drastically reduced compared with those in the W1 and W2. No hole is observed on the sample W4 at E_{in} of around 1 eV. These experimental observations indicate that there is a threshold energy of E_{in} for generation of hole. The theoretical calculation gives about 6 eV as a surface barrier potential energy for He penetrating into the W interstitial site [11], which would be strongly related to the observed threshold value because incident ions with an energy below the surface barrier potential energy can not penetrate into the W. As a result, there are no holes formed. In the sample W4, the incident energy of He ions was so low that no He ions could enter W interstitial sites. However, one may argue that in the case of W4, almost all incident He atoms are promptly released from the W surface due to the high surface temperature of 2950 K, resulting in no hole formation. In the pre-

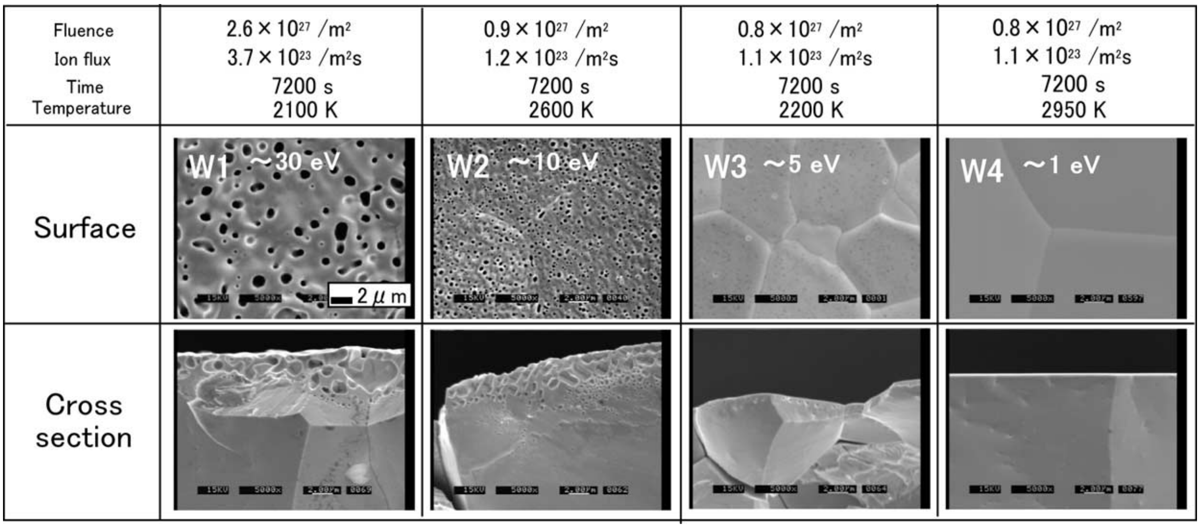

Fig. 1. The dependence of incident ion energy E_{in} for bubble and hole formation on the PM-W surface irradiated by low energy and high-flux He plasma. Heavy hole formations are seen on the surface as well as the cross section of the samples W1(~30 eV) and W2(~10 eV). Smaller holes and bubbles are seen in the sample W3(~5 eV) and no hole and bubble formation is observed in the sample W4(~1 eV). Magnification of all eight photos is same(×5000).

vious experiment, however, no hole formation was also observed in a sample at E_{in} of 1 eV [5] and a surface temperature around 1850 K with an ion flux of 1.0×10^{22} $m^{-2}\,s^{-1}$ and fluence of 6.0×10^{25} m^{-2}. Surface temperature and fluence of this sample are enough for hole formation if the E_{in} is high (ex: 10 eV). Although there is some difference in the flux, the fluence and the surface temperature between the two samples, it could be concluded that there is some boundary value of E_{in} for hole formation on the W surface. Finally, we should mention an uncertainty in determining E_{in} The value of E_{in} was determined by difference between time-averaged plasma potential and the sample target potential, which is fixed by a biasing voltage with respect to the vacuum chamber. The plasma potential could fluctuate in time in a range of ±4 eV, which was estimated by temporal measurement of the floating potential [5]. Then, the sample W3 with averaged value E_{in} of 5 eV would be irradiated by He ions with an incident ion energy ranging form 1 to 9 eV. Thus it is very difficult to determine the exact threshold value E_{in} for hole formation in our experiments. Small holes observed on the sample W3 could be attributed to He ions with ion energy above the surface barrier potential energy although averaged E_{in} is below the surface barrier potential energy.

3.2. Hole formation in single crystal tungsten

It is one of the most important subjects in the He bubble and hole formation induced by low energy and high particle flux He plasma irradiation to understand dominating trap sites of He atoms for nucleation of He bubbles. In high-energy ion implantations, above the sputtering threshold energy, defects such as vacancy induced by radiation displacement play an essential role in the nucleation of bubble formation. However, no defects are expected to be produced under such low energy ion implantation in our experimental condition. Intrinsic defects in the PM-W sample and thermal vacancy are considered as possible trap sites for He atoms. The number of thermal vacancy increases with the material temperature. We have investigated the hole formation on single crystal tungsten (SC-W), which has much fewer intrinsic defects than powder metallurgy tungsten. Experimental conditions are as follows: surface temperature $T_s \sim 1650$ K, $E_{in} \sim 25$ eV and fluence $\sim 9.0\times10^{25}$ m^{-2}. These parameters are high enough to produce holes on PM-W. Fig. 2 shows a SEM micrograph on the surface of the SC-W after irradiated by He plasma. A lot of holes are seen on the W surface, and there is no qualitative difference between the SC-W and the PM-W samples. This result indicates that intrinsic defects in the PM-W sample do not play a major role in the bubble and hole formations, and the thermal vacancies, which is determined by material temperature, should be dominating trap sites for He atoms in the material.

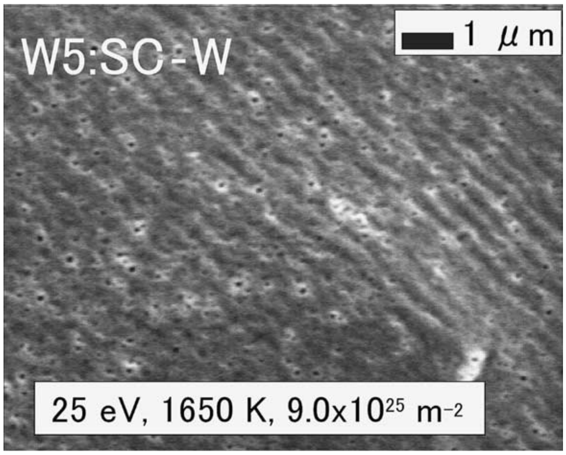

Fig. 2. Hole formation on single crystal W (SC-W). The orientation is [1 1 0] plane. Irradiation parameters are as follows: $E_{in} \sim 25$ eV, $T_s \sim 1650$ K, flux $\sim 4.8\times10^{22}$ $m^{-2}\,s^{-1}$, irradiation time ~1800 s, fluence $\sim 9.0\times10^{25}$ m^{-2}.

3.3. Surface temperature dependence of the bubble and hole growth

TEM analysis was performed on three PM-W samples (W6-W8) irradiated by He plasma at E_{in} of 20–25 eV with sample temperatures of 1300 K (W6), 1650 K (W7), 1950 K (W8) as shown in Fig. 3. It is found that the size of He bubbles d_B depends drastically on the sample temperature. The bubble size d_B of the sample W6(d_B < 5 nm) is much smaller than that of the sample W8(d_B < 500 nm), even when the He ion fluence of the sample W6 is about twice as large as that of the sample W8. It is found in the SEM microphotograph that no visible holes appear at a sample temperature of 1300 K. This result also indicates that thermal vacancies play an essential role in the bubble growth because the thermal vacancy density drops rapidly as decreasing the sample temperature. The thermal vacancy density $C_v = N_v/N_w =\sim \exp(-E_v/kT)$ in the sample W6 is estimated to be $\sim 10^{-15}$ by using the vacancy formation energy E_v of W 3.7 eV [11], which is much smaller than $\sim 10^{-12}$ in the sample W8. N_v and N_w are number density of vacancies and W atoms in the matrix, respectively. Another possible reason to explain the difference of the bubble size between the samples W6 and W8 is a temperature dependence of diffusion of small bubbles as well as vacancies in the interstitial site. Marochov et al. [12] indicated the importance of coalescence of small bubbles as the bubble growth mechanism. Fig. 4 shows another cross section of the sample W1. The parts indicated by white circles are speculated to be an early stage of bubble coalescence. Holes or pass to the neighbor

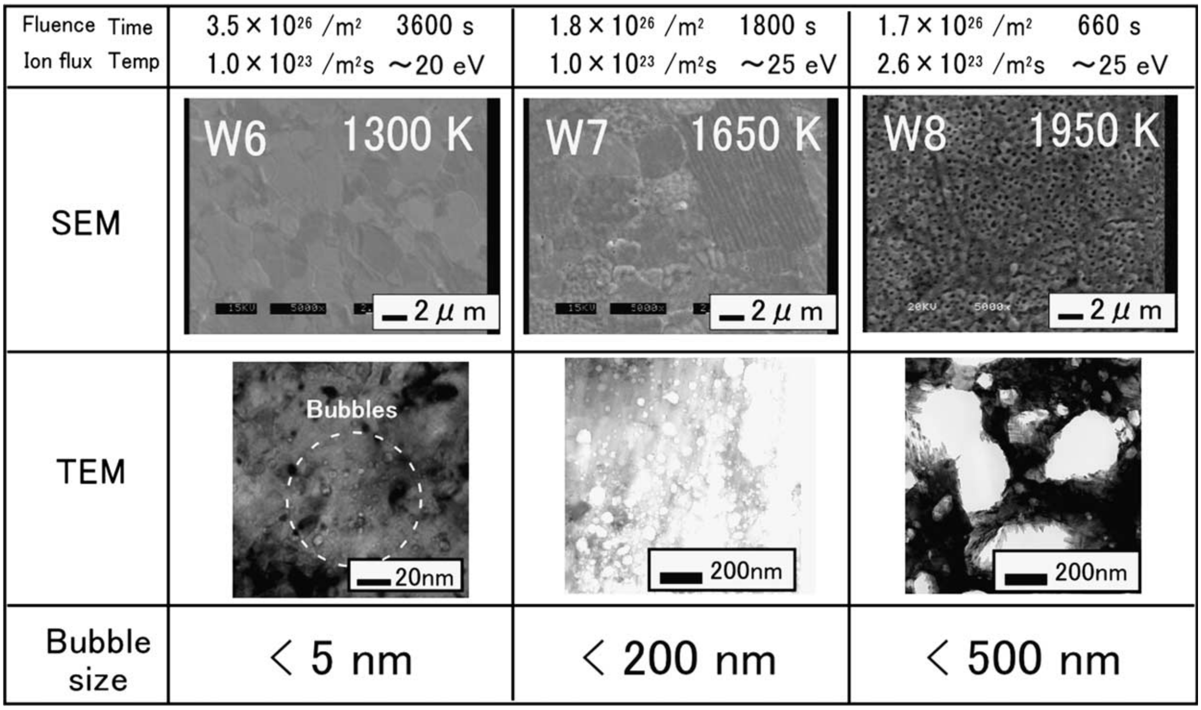

Fig. 3. Temperature dependence of bubble and hole formations on PM-W surfaces at different temperatures; W6: 1300 K, W7: 1650 K, W8: 1950 K. Bubbles or holes clearly appear, and the size of bubble becomes drastically larger with the temperature above the recrystallization temperature of W (1400–1500 K).

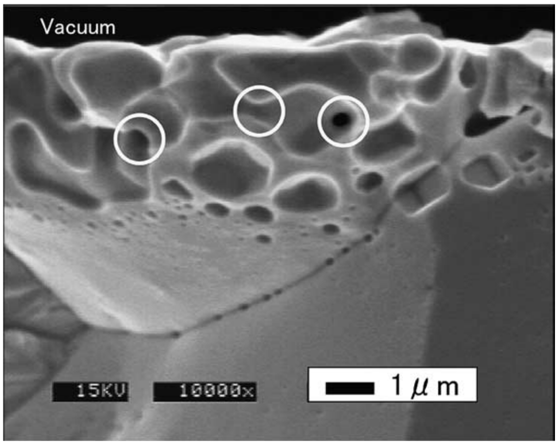

Fig. 4. Cross section of the sample W1 which shows the hole or pass to neighbor bubble at the bubble wall.

bubble are formed on the bubble interior wall. Above the re-crystallization temperature of the material (W: 1400–1500 K), migration and coalescence of bubbles tend to occur easily because the relaxation of W's atomic bond makes He bubbles and vacancies more mobile and bubble itself can expand easily. On the other hand, when the temperature of the sample is below the re-crystallization temperature, both reduction of the thermal vacancy density and poor mobility of small bubbles and vacancies due to low sample temperature could suppress the bubble growth and expansion.

4. Conclusion

We have done a systematic study to investigate bubble and hole formation on W surfaces irradiated by a low-energy and high-flux steady state He plasma. Large micron-sized He bubbles and holes can be formed on the W surface under the low energy plasma irradiation when the experimental condition satisfies the following conditions: that (1) incident He ion energy exceeds about 5 eV, and (2) tungsten surface temperature is higher than approximately 1600 K. Condition (1) corresponds to the penetration of He atoms into the W bulk, which is associated with the surface barrier potential energy of W for He. Condition (2) is necessary for the generation of thermal vacancies which could be dominating trap sites for He atoms for the bubble nucleation. Condition (2) also relates to high mobility and coalescence of He bubbles and vacancies, which contributes to the growth of the He bubbles. If the surface temperature is high enough, especially above the re-crystallization temperature, He atoms and vacancies meet more frequently and expansion and coalescence of He bubbles are accelerated.

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