NUCL SCI TECH (2016) 27:37
DOI 10.1007/s41365-016-0049-y

Erosion of tungsten surfaces in He and Ar/He plasma

Chao Li[1] · Bo Wang[1] · Zi-Long Zhang[1] · Li-Xia Zhang[2]

Received: 24 February 2015 / Revised: 17 June 2015 / Accepted: 20 August 2015 / Published online: 11 April 2016

Abstract Irradiation tests of tungsten surface were performed with He and He/Ar plasma generated by microwave electron cyclotron resonance. Thickness loss was used as the erosion rate of tungsten surface under the plasma irradiation. The results revealed that the thickness loss increased linearly with negative bias. SEM images proved that the addition of Ar apparently increased the plasma erosion. The thickness loss increased sharply with the Ar fraction of Ar/He mixture when it was <20 %, where the increasing slope of thickness loss lowered down gradually.

Keywords Plasma-facing material (PFM) · Erosion · ECR · Plasma irradiation · Tungsten

1 Introduction

With the advantages of high melting point, good thermal conductivity and low vapor pressure, tungsten is considered as the most promising plasma-facing material (PFM) for future fusion reactors [1, 2]. In the loading conditions of a fusion reactor, however, tungsten surfaces will suffer from high heat flux ($\sim$10 MW/m^2) and high irradiation flux (10^{22}–10^{24} m^{-2} s^{-1}) of plasma at low energies [3, 4]. The synergistic effect of high heat loading and plasma flux will result in severe irradiation effects, such as deuterium retention [5–8], nanostructures [9], bubbles and holes [5, 10], surface erosions [2] and morphology changes [11–13].

Among these irradiation effects, surface erosion of PFMs is an important factor which determines the lifetime of the plasma-faced components [2, 14, 15]. PFM erosion has been investigated extensively [16–18]. Both the irradiation conditions of steady state and transient loads can lead to PFM erosion. In a steady state, when the energy of impacting ions exceeds the sputtering threshold energy of tungsten [19], PFM sputtering is the main erosion mode [2].

PFM of diverters, the part to clean the plasma from helium and impurities, normally suffers from a stronger irradiation load than other parts do [20]. Usually, PFM surfaces of a diverter are irradiated by a plasma mixture. For the diverter of a burning plasma device, inert gases are considered as the injected impurities to cool down the excessive heat flux [21]. So, studying surface sputtering by plasma mixed with inert gas is of great interest.

In this paper, we study the erosion of tungsten irradiated by He plasma and Ar/He plasma mixture. The influence of plasma parameters on the thickness loss, which scales the erosion rate of tungsten surface, is discussed.

2 Experimental

2.1 The preparation of tungsten samples

Tungsten samples, of Φ20 mm × 1 mm, were cut from a plate of powder metallurgy tungsten (99.95 % purity). The samples were annealed for 1 h at 1273 K to eliminate the internal stress and were polished with the electrolytic polishing method in the solution of 2 % sodium hydroxide.

✉ Bo Wang
wangbo@bjut.edu.cn

[1] Department of Materials Science and Engineering, Beijing University of Technology, Beijing 100124, China

[2] The Third Laboratory, Research and Test Center, Hangtiankegong Corporation, Beijing 100854, China

2.2 Irradiation tests of plasma

The plasma irradiation was performed with an equipment of electron cyclotron resonance (ECR). Figure 1 shows the schematic diagram and a discharge picture of the ECR equipment. In the vacuum chamber, samples were placed on an electrode to control the bombardment energy, with a negative bias (50–275 V). A permanent magnet was placed under the samples to constrain the plasma converging.

The samples were irradiated at fluxes of 0.5×10^{21}, 1.0×10^{21} and 1.5×10^{21} m^{-2} s^{-1}, with changing Ar mass flow rate to helium (the Ar volume percentage in Ar/He mixture).

2.3 Measurements of samples

Thickness loss is used to scale the erosion rate of the irradiated tungsten samples, which were covered by a small piece of tungsten during the plasma irradiation. A surface profiler (SURFCOM 480 A) was employed to measure the step height which was regarded as the thickness loss. Every step was measured for five times at different site. The sample morphology was characterized by scanning electron microscopy (SEM) and atomic force microscope (AFM).

3 Results and discussion

3.1 Influences of the negative bias on the erosion of pure helium plasma

A series of tungsten samples were irradiated by pure He plasma of 1.0×10^{25} m^{-2} in fluence and 1×10^{21} m^{-2} s^{-1} in flux. The sample temperature was around 650 K. The samples were biased negatively at 50–275 V. As shown in Fig. 2, the thickness loss increased almost linearly with the irradiation energy which was controlled by the bias voltage.

Thickness loss was about 0.40 μm at 275 V. However, the surface micromorphology did not show an apparent variation before and after the pure He plasma irradiation (Fig. 3). This indicates that pure helium plasma at 50–275 eV can hardly make an observable damage to tungsten surface.

This is because that the energy transfer of helium ions to tungsten was low. It can be calculated by [22]:

$$U = 4M_1M_2E/(M_1 + M_2)^2, \tag{1}$$

where U is the energy transfer from an incident particle to a target atom, E is the energy of incident particle and M_1 and M_2 are weight of the incident particle and target atom, respectively. For He and W, $M_1 = 4$ and $M_2 = 183.84$, we have $U = 0.0834\ E$.

Thus, the transferred energy for a helium ion is only 23 eV when the negative bias is 275 V. The binding energy of tungsten is 8.90 eV, which means that a He ion of <106.7 eV cannot sputter a tungsten atom. A probable

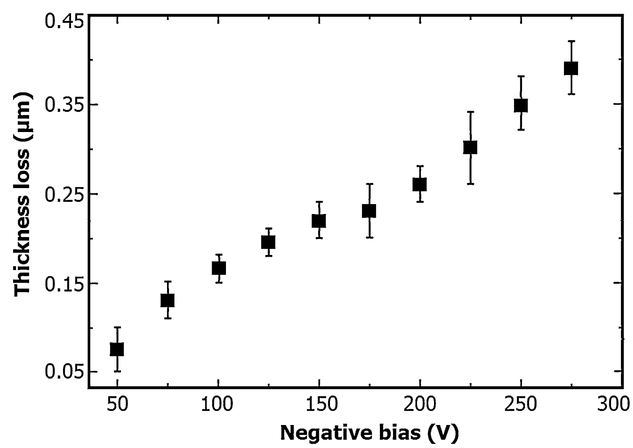

Fig. 2 Thickness loss of tungsten sample irradiated by pure He plasma, as function of the negative bias

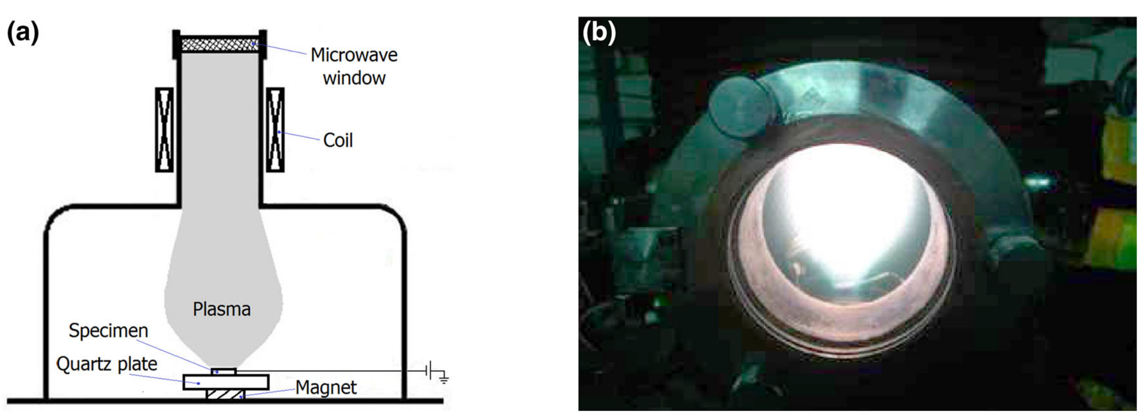

Fig. 1 Schematic diagram of the ECR equipment (**a**) and photo of the ECR plasma (**b**)

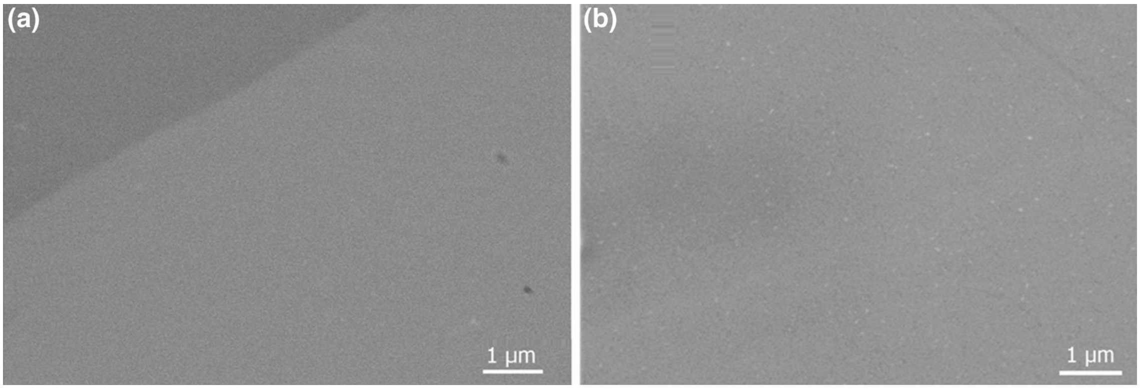

Fig. 3 SEM images of tungsten sample surfaces before (**a**) and after (**b**) irradiation by pure He plasma

reason for the slight thickness loss at 50 V might be due to the energy fluctuation effect, for which a few He and W atoms could obtain an energy much higher than the average value at 650 K. Additionally, at high fluxes, the mass of interstitial atoms of helium existing in the tungsten surface layer might be another possible influencing factor.

3.2 Influences of Ar in the plasma mixture

Inert gases were considered as cooling gases to add to diverters of a fusion reactor, and argon has been added into the plasma. Polished tungsten samples were irradiated by plasma mixed with argon in atomic percentages of 0, 4.8, 9.1, 13.0, 16.7, 33.3, 50.0, 66.7, 83.3, 92.3 and 100 %, at plasma flux of 1.0×10^{21} m^{-2} s^{-1}, fluence of $1.0 \times 10^{25}/m^2$ and bias of -200 V. The thickness losses are shown in Fig. 4. The results indicate that the addition of Ar aggravated greatly the thickness loss, which increased sharply with the Ar percentage, when it was <20 at.%, where the increasing rate of thickness loss began to lower down gradually.

The Ar addition effect can also be seen by SEM observations (Fig. 5). In Fig. 5b, ripple-like damages can be seen on the surface irradiated by the plasma mixed with Ar at 13 at.%. For comparison, we show also the morphology of the surface irradiated by plasma of pure He plasma (Fig. 5a) and pure Ar plasma (Fig. 5c). The latter made the most serious erosion on the surface, which was eroded to cobblestone-like morphology.

AFM images taken near the steps on the surfaces irradiated by the plasma mixed with Ar at 92.3 and 9.1 at.% are shown in Fig. 6. The scale marks of *Z*-axis were set as the symmetrical ranges from -1.9 μm depth to 1.9 μm height in Fig. 6a, and from -1.2 μm depth to 1.2 μm height in Fig. 6b. In each AFM image, the ridge-shaped topography can be seen along the step edge of the covered area, revealing that some sputtered particles could redeposited at the edge of the gap between the cover and the sample.

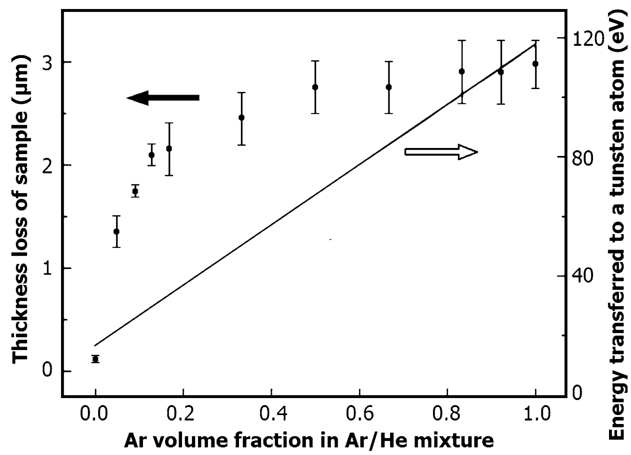

Fig. 4 Thickness loss vs fraction of Ar atoms in the mixed gas. (flux = $1.0 \times 10^{21}/m^2$ s, fluence = $1.0 \times 10^{25}/m^2$, bias = -200 V)

The energy transfer of Ar^+ to tungsten can be estimated as follows. If the secondary ionization was negligible and the ratio of Ar^+:He^+ in plasma is assumed the same as the atomic ratio of Ar:He in the gas mixture, the thickness loss shall be in proportion to the composite transferred energy:

$$\text{Thickness Loss} \sim U_{\text{com}} = U_{\text{Ar}}\, x + U_{\text{He}}(1-x), \tag{2}$$

where U_{com} is the composite transferred energy (transferred energy from composite to tungsten), which is a nominal variable; x is the fraction of Ar atoms in the mixed gas; and U_{Ar} and U_{He} are the transferred energy to tungsten atoms from an incident particle of Ar^+ and He^+, respectively. The mass of Ar atom is 39.95, so using Eqs. (1) and (2), we have:

$$U_{\text{com}} = (0.0834 + 0.5032x)E. \tag{3}$$

The composite transferred energy increases linearly with the fraction of Ar atoms. For the incident energy of 200 eV, the relationship of composite transferred energy can be drawn as shown in Fig. 4. However, it does not fit

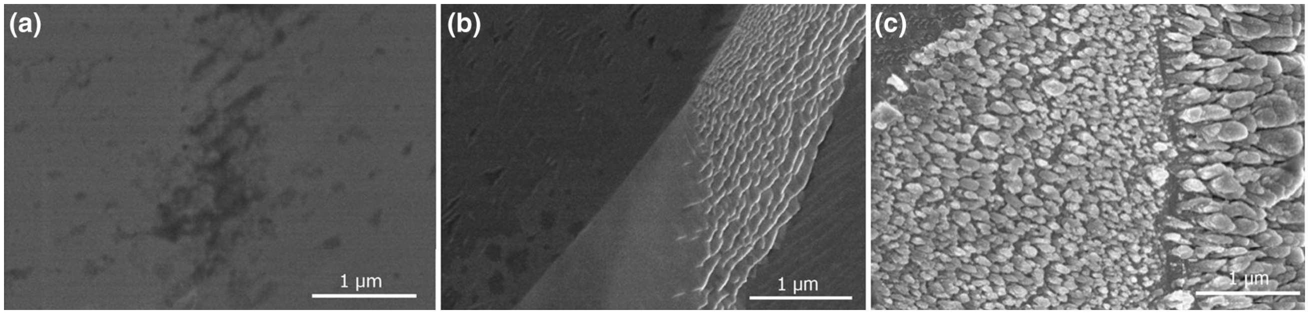

Fig. 5 SEM images of tungsten surfaces irradiated by plasmas of **a** pure He, **b** He–Ar mixture (Ar, 13.0 at.%) and **c** pure Ar

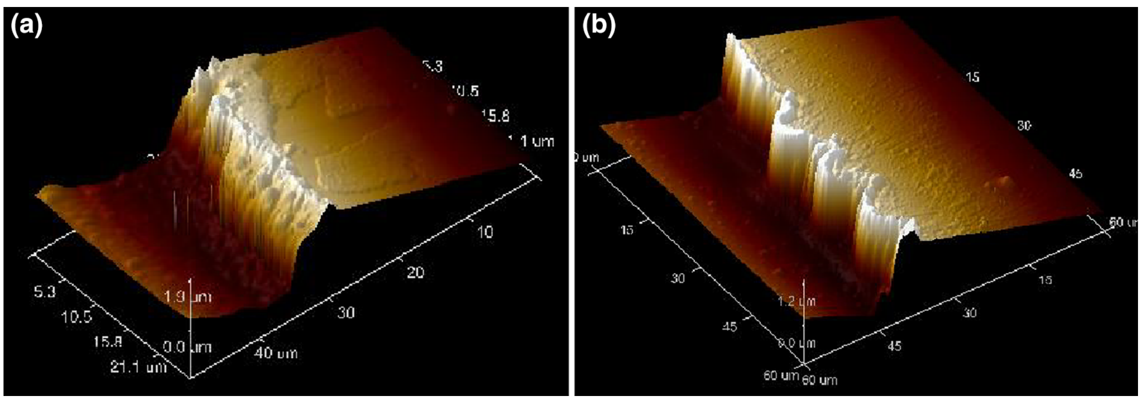

Fig. 6 AFM images of the surfaces irradiated by the plasma mixed with Ar at 92.3 % (**a**) and 9.1 % (**b**)

the experiment result of the relationship of thickness loss with Ar fraction. This is due to the above assumption which is too simple. Since the first ionization energy of Ar is lower than that of helium, the ratio of Ar ion and He ion in the plasma should be different from that of the neutral molecule. Further studies on the influences of Ar to the thickness loss will be carried out.

4 Conclusion

Tungsten surface was irradiated by the ECR plasma of helium or helium/argon mixture. The results revealed that the thickness loss of tungsten increased linearly with the negative bias, which can be explained as the increase in the bombardment energy. The addition of Ar could aggravate the plasma erosion. The plasma with a high fraction of Ar would make the tungsten surface damaged seriously. The thickness loss increased sharply with the Ar fraction below the Ar percentage of 20 at.%, where the increasing slope of thickness loss began to lower down gradually. The composite transferred energy does not fit the increase rate of the thickness loss measured with Ar-mixed plasma. The difference of the ionization rate of Ar and He should be taken into account.

Acknowledgments This work was supported by the National Magnetic Confinement Fusion Program (Grant No. 2013GB109003).

References

1. S. Wurster, N. Baluc, M. Battabyal et al., Recent progress in R&D on tungsten alloys for divertor structural and plasma facing materials. J. Nucl. Mater. **442**, S181–S189 (2013). doi:10.1016/j.jnucmat.2013.02.074
2. J. Roth, E. Tsitrone, A. Loarte et al., Recent analysis of key plasma wall interactions issues for ITER. J. Nucl. Mater. **390–391**, 1–9 (2009). doi:10.1016/j.jnucmat.2009.01.037
3. H. Bolt, V. Barabash, W. Krauss et al., Materials for the plasma-facing components of fusion reactors. J. Nucl. Mater. **329–333**, 66–73 (2004). doi:10.1016/j.jnucmat.2004.04.005
4. G.-N. Luo, W.M. Shu, H. Nakamura et al., Ion species control in high flux deuterium plasma beams produced by a linear plasma generator. Rev. Sci. Instrum. **75**, 4374–4378 (2004). doi:10.1063/1.1807006
5. W.M. Shu, M. Nakamichi, Alimov V. Kh et al., Deuterium retention, blistering and local melting at tungsten exposed to high-fluence deuterium plasma. J. Nucl. Mater. **390–391**, 1017–1021 (2009). doi:10.1016/j.jnucmat.2009.01.267
6. D. Nishijima, T. Sugimoto, H. Iwakiri et al., Characteristic changes of deuterium retention on tungsten surfaces due to low-energy helium plasma pre-exposure. J. Nucl. Mater. **337–339**, 927–931 (2005). doi:10.1016/j.jnucmat.2004.10.011
7. O.V. Ogorodnikova, J. Roth, M. Mayer, Ion-driven deuterium retention in tungsten. J. Appl. Phys. **103**, 034902 (2008). doi:10.1063/1.2828139

8. T. Tanabe, Review of hydrogen retention in tungsten. Phys. Scr. **T159**, 014044 (2014). doi:10.1088/0031-8949/2014/T159/014044
9. S. Kajita, W. Sakaguchi, N. Ohno et al., Formation process of tungsten nanostructure by the exposure to helium plasma under fusion relevant plasma conditions. Nucl. Fusion **49**, 095005 (2009). doi:10.1088/0029-5515/49/9/095005
10. D. Nishijima, M.Y. Ye, N. Ohno et al., Formation mechanism of bubbles and holes on tungsten surface with low-energy and high-flux helium plasma irradiation in NAGDIS-II. J. Nucl. Mater. **329–333**, 1029–1033 (2004). doi:10.1016/j.jnucmat.2004.04.129
11. S. Takamura, T. Miyamoto, Y. Tomida et al., Investigation on the effect of temperature excursion on the helium defects of tungsten surface by using compact plasma device. J. Nucl. Mater. **415**, S100–S103 (2011). doi:10.1016/j.jnucmat.2010.12.021
12. K. Tokunaga, S. Tamura, N. Yoshida et al., Synergistic effects of high heat loading and helium irradiation of tungsten. J. Nucl. Mater. **329–333**, 757–760 (2004). doi:10.1016/j.jnucmat.2004.04.178
13. T.Y. An, H.Y. Fan, Y. Wang et al., Influence of irradiation temperature on the surface damage of tungsten. Nuclear Techniques **37**(9), 090201 (2014). doi:10.11889/j.0253-3219.2014.hjs.37.090201
14. J.B. Lin, A.G. Li, S.M. He et al., Investigation on corrosion resistance of Hastelloy N alloy after He^+ ion irradiation. Nucl. Tech. **37**(5), 050601 (2014). **(in Chinese)**
15. Y. Wang, H. Fan, M.Y. LYU et al., Influence of low energy and high-flux He ion irradiation on the surface damage of Mo, Nuclear Techniques **37**(12), 120204 (2014). doi:10.11889/j.0253-3219.2014.hjs.37.120204
16. A. Kreter, D. Nishijima, M.J. Baldwin et al., Mitigation of carbon erosion in beryllium seeded deuterium plasma under bombardment by argon and helium ions in PISCES-B. J. Nucl. Mater. **417**, 651–654 (2011). doi:10.1016/j.jnucmat.2010.12.098
17. T. Sizyuk, A. Hassanein, Effect of surface segregation and mobility on erosion of plasma-facing materials in magnetic fusion systems. J. Nucl. Mater. **458**, 312–318 (2015). doi:10.1016/j.jnucmat.2014.12.105
18. B. Khripunov, V. Gureev, V. Koidan et al., Erosion and deuterium retention in ion-irradiated tungsten under plasma exposure. J. Nucl. Mater. (2015). doi:10.1016/j.jnucmat.2014.12.115
19. K. Krieger, H. Maier, the ASDEX Upgrade Team, Conclusions about the use of tungsten in the divertor of ASDEX Upgrade. J. Nucl. Mater. **266–269**, 207–216 (1999). doi:10.1016/S0022-3115(98)00890-3
20. M. Rieth, S.L. Dudarev, S.M. Gonzalez de Vicente et al., Recent progress in research on tungsten materials for nuclear fusion applications in Europe. J. Nucl. Mater. **432**, 482–500 (2013). doi:10.1016/j.jnucmat.2012.08.018
21. A. Kallenbach, M. Bernert, R. Dux et al., Impurity seeding for tokamak power exhaust from present devices via ITER to DEMO. Plasma Phys. Control Fusion **55**, 124041 (2013). doi:10.1088/0741-3335/55/12/124041
22. K. Katayama, K. Imaoka, T. Okamura et al., Helium and hydrogen trapping in tungsten deposition layers formed by helium plasma sputtering, Fusion Eng Des **82**, 1645–1650 (2007). doi:10.1016/j.fusengdes.2007.04.026