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Surface morphology and helium retention on tungsten exposed to low energy and high flux helium plasma

K. Tokunaga [a,*], R.P. Doerner [b], R. Seraydarian [b], N. Noda [c], Y. Kubota [c], N. Yoshida [a], T. Sogabe [d], T. Kato [e], B. Schedler [f]

[a] *Research Institute for Applied Mechanics, Kyushu University, 6-1 Kasuga-Koen, Kasuga-shi, Fukuoka 816-8580, Japan*
[b] *University of California, San Diego, Fusion Energy Research Program, 9500 Gilman Drive., La Jolla, CA 92093-0417, USA*
[c] *National Institute for Fusion Science, Toki, Gifu 509-5292, Japan*
[d] *Toyo Tanso Co., Ltd., Ohnohara-cho, Mitoyo-gun, Kagawa 769-1614, Japan*
[e] *Nippon Plansee K.K., Chiyoda-ku, Tokyo 102-0083, Japan*
[f] *Plansee Aktiengesellschaft, A-6600 Reutte, Austria*

Abstract

Surface modification and helium retention on powder metallurgy tungsten and plasma sprayed tungsten coated CFC exposed by low energy (100 eV) high flux (3.83×10^{21}–1.20×10^{22} $m^{-2}\,s^{-1}$) helium plasma have been investigated. Fluence is about 10^{26} He m^{-2}. It is found that fine surface morphology change occurs by the helium exposure. There is little difference of the surface modification between the powder metallurgy tungsten and the plasma sprayed tungsten. In the case that the surface morphology do not changed, two helium desorption peak appeared. The amount of desorption of helium is larger than that of deuterium from tungsten under the same exposure condition by heating up to 1273 K by TDS. In the case that fine surface morphology change occurs, desorption peak at high temperature newly appears. Surface modification such as blister is not formed on the surface exposed to helium under the same exposure condition which the blister is formed by deuterium exposure.

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1. Introduction

Recently, tungsten became one of the candidate plasma facing materials for fusion devices due to its low sputtering yield and good thermal properties. The plasma facing materials will be subjected to high-flux low energy particle bombardment including fuel hydrogen isotope and helium ash. In recent studies, surface modification and hydrogen retention on tungsten exposed to hydrogen isotope ion with energies (a few hundred eV) below that required to generate elastic displacement have been investigated [1–5]. The studies have demonstrated that hydrogen isotope implantation into tungsten can produce blustering. The blister formation affects the trapping behavior of hydrogen isotopes and erosion resulting from surface flaking.

On the other hand, the accumulation of helium is much more harmful to metal than hydrogen isotope because of its strong interaction with lattice defects. Many experiments on the material damage due to helium irradiation have carried out [6–10]. The results show that helium enhances the formation of bubbles drastically due to the strong bonding to vacancies and their clusters. As a results, blistering, flaking, surface swelling, gas reemission and degradation of mechanical properties of bulk materials take place. However, these experiments have been performed at low flux

* Corresponding author. Tel.: +81-92 583 7986; fax:+81-92 583 7690.
E-mail address: tokunaga@riam.kyushu-u.ac.jp (K. Tokunaga).

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$<10^{20}$ $m^{-2}\,s^{-1}$ and high energy greater than a few keV, which only for energies high enough to cause elastic damage. Therefore, further experimental investigations at high flux low energy relevant to the fusion devices are required for more accurate assessments on tungsten [11]. Ye et al. reported that holes are formed on the thin powder metallurgy tungsten (PM-W) irradiated with 50 eV He with the fluence of 10^{25}–10^{26} He m^{-2} at 1770 K [12]. In the present study, surface modification and helium retention of thick PM-W, plasma sprayed tungsten exposed to low energy helium have been investigated.

2. Experimental

Carbon/carbon composite (CFC) CX-2002U made by Toyo Tanso was coated using vacuum plasma spraying (VPS) [13–16]. Heat treatments were performed to stabilize the microstructure of the sample. The thickness of the tungsten coating layer was 0.5 and 1.0 mm. The density of VPS tungsten (VPS-W) was 92.5% of that of theoretical density. They are simply denoted as VPS-W/CX-2002U. Details of the fabrication method were described in the previous papers [13]. The sizes of VPS-W/CX-2002U were 20 mm length, 14.5 mm width and 2.0 mm thickness. Pure tungsten fabricated by powder metallurgy (PM-W) supplied by Tokyo Tungsten Co., Ltd. was also used to compared with the results of the VPS-W/CX-2002U. Purity of PM-W was 99.99 wt%. A disk of PM-W has a size of 24.5 mm diameter and 2 mm thickness. The surface of the PM-W was electrically polished. All samples were degassed by heating up to 1273 K in high vacuum before installation to a plasma facility.

The facility used in the experiments was the PISCES-B which is a liner plasma simulator device at the University of California, San Diego. Details of the PISCES-B were described in Ref. [17]. In the present experiments, helium was used as working gas. The ion flux (He^+) to the sample was varied from 3.83×10^{21} to 1.20×10^{22} $m^{-2}\,s^{-1}$ corresponding to the plasma exposures. The energy of the impinging ions was 100 eV. Plasma parameters remained constant throughout the plasma exposure. The sample was clamped to a water or air cooled sample holder with a Tantalum cap. The temperature was measured at the center of the sample on the side opposite to the plasma with a thermocouple. The samples were heated by the plasma and reached their final steady state temperatures within about 1 min. The sample temperatures were in a range from 705 to 933 K depending on the ion fluxes.

After the plasma exposures, surface composition of the samples was measured with an Auger electron spectroscope (AES) equipped with the main chamber of the PISCES-B. The sample was removed from the PISCES-B facility and microstructure was studied by scanning electron microscopy (SEM). Weight loss before and after the exposure was also measured. In addition, retention of helium after the exposure was examined by thermal desorption spectroscopy (TDS). The sample was linearly ramped up to 1273 K for 65 min (0.25 K/s) and remained at 1273 K for 20 min, and was cooled down linearly to 298 K for 90 min (0.18 K/s).

3. Results

3.1. Surface modification

Fig. 1 shows SEM images of the surface of PM-W before (a) and after ((b),(c)) the helium irradiation. PM-W(b) and PM-W(c) were exposed by helium ion at 933 K with the fluence of 1.11×10^{26} He m^{-2} and at 837 K with a the fluence of 6.00×10^{25} He m^{-2}, respectively. In the case of PM-W(b), it seems that crystal grain growth

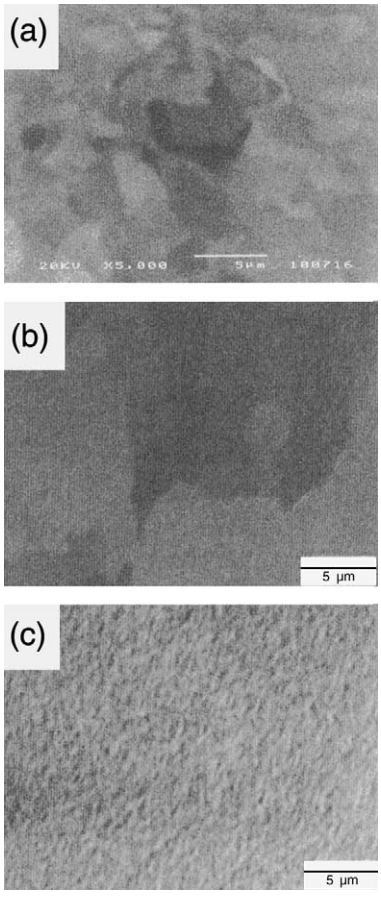

Fig. 1. SEM image before and after plasma exposure (a) PM-W before exposure. (b) PM-W(b) after exposure to helium ion (1.11×10^{26} He m^{-2} at 933 K). (c) PM-W(c) after exposure to helium ion (6.00×10^{25} He m^{-2} at 873 K).

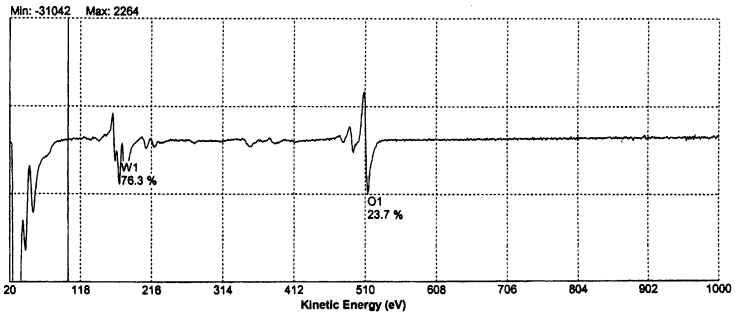

Fig. 2. AES spectrum of PM-W(c) after exposure to helium ion (6.00×10^{25} He m^{-2} at 873 K).

occurs but surface morphology change is not observed. On the other hand, in the case of PM-W(c), very fine morphology change occurs and surface is modified to be island structure. The color of this sample (PM-W(c)) becomes to be black from metallic sliver color. Fig. 2 shows an AES spectrum of the PM-W(c) after the exposure. This result indicates that surface consists of W (76.2 at%) and O (23.7 at%).

Fig. 3 shows SEM images of the surface of VPS-W/CX-2002U before (a) and after ((b),(c)) the helium irradiation. VPS-W/CX-2002U(b) and VPS-W/CX-2002U(c) were exposed by helium ion at 705 K with the fluence of 3.45×10^{25} He m^{-2} and at 908 K with the fluence of 4.15×10^{25} He m^{-2}, respectively. Before irradiation, it can be seem that spherical particles are

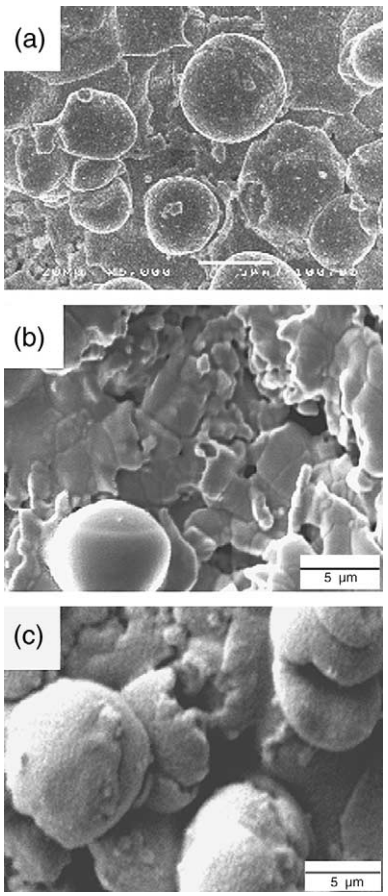

Fig. 3. SEM image before and after plasma exposure. (a) VPS-W/CX-2002U before exposure. (b) VPS-W/CX-2002U(b) after exposure to helium ion (3.45×10^{25} He m^{-2} at 705 K). (c) VPS-W/CX-2002U(c) after exposure to helium ion (4.15×10^{25} He m^{-2} at 908 K).

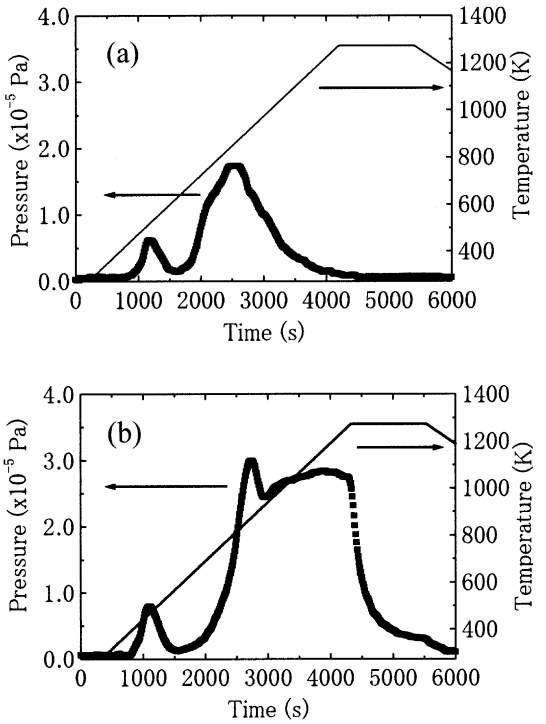

Fig. 4. Thermal desorption spectrum of He. (a) PM-W(b) after exposure to helium ion (1.11×10^{26} He m^{-2} at 933 K). (b) PM-W(c) after exposure to helium ion (6.00×10^{25} He m^{-2} at 873 K).

melted or partially melted and joined each other and pores are formed in the tungsten coatings. In addition, very small fragments (~0.1 μm) are observed on the surface of VPS-W/CX-2002U as shown in Fig. 3(a). After the irradiation, they disappeared but no large morphology changes are observed as shown in Fig. 3(b) in the case of PM-W(b). On the other hand, in the case of PM-W(c) as shown in Fig. 3(c), surface morphology and color were changed in the similar behavior of the results of the PM-W(c).

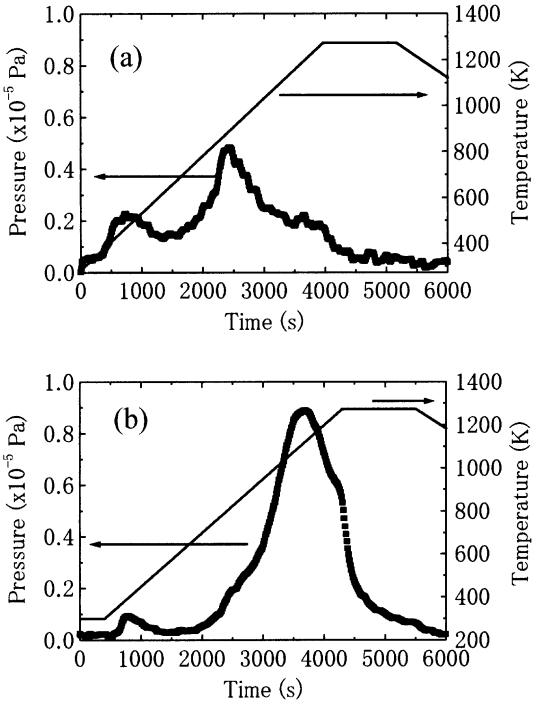

Fig. 5. Thermal desorption spectrum of He. (a) VPS-W/CX-2002U(b) after exposure to helium ion (3.45×10^{25} He m^{-2} at 708 K). (b) PVS-W/CX-2002U(c) after exposure to helium ion (4.15×10^{25} He m^{-2} at 908 K).

Table 1
Incident fluence and total desorption of He

	Sample irradiation temperature (K)	Fluence (m^{-2})	Total desorption (He m^{-2})
PM-W(b)	933	1.11×10^{26}	4.25×10^{20}
PM-W(c)	873	6.00×10^{25}	1.29×10^{21}
VPS-W/CX-2002U(b)	705	3.45×10^{25}	2.47×10^{20}
VPS-W/CX-2002U(c)	908	4.15×10^{25}	4.69×10^{20}

3.2. Retention property

Fig. 4(a) and (b) show thermal desorption spectrum of helium from PM-W(b) and PM-W(c), respectively. The major peaks appears at around 523 and 873 K for PM-W(b). In the case of PM-W(c), as shown in Fig. 4(b), a large amount of He desorption continues at the higher temperature.

Fig. 5(a) and (b) show thermal desorption spectrum of helium from VPS-W/CX-2002U(b) and VPS-W/CX-2002U(c), respectively. Peaks are observed at 473 and 873 K for VPS-W/CX-2002U(b) as shown in Fig. 5(a), and a tail of desorption appears at the higher temperature as shown in Fig. 5(a). On the other hand, in the case of VPS-W/CX-2002U(c), two peaks appears at 393 and 1093 K with a small shoulder at about 873 K.

Table 1 shows the fluence and the total amount of desorption. This indicated that the retained amount of helium increases when surface morphology change occurred.

4. Discussion

In the case of deuterium exposure under the approximately same experimental condition, blisters with a diameter of a few tens μm to 0.1 μm were formed on the surface of PM-W [5]. On the other hand, in the present helium exposure experiment, such kind of blister was not observed. Helium implanted into tungsten dose not travel for away from deposited because binding energy of helium with defects in tungsten is very high and therefore helium would be trapped near surface. This is expected to be a reason why the blister was not formed in the present helium exposure experiment. At higher temperature, the traps no longer are able to hold helium and therefore the helium will diffuse to deeper into material. In this case, surface modification such as the blister formation may occur. Indeed, it was reported that small holes are formed on tungsten surface irradiated with 50 eV helium at the fluence of 10^{25}–10^{26} He m^{-2} at 1770 K [12].

Very fine morphology change was observed on the surface of PM-W(c) and VPS-W/CX-2002U(c). One of the reasons of the surface modification may be due to formation of re-deposited layer. The 100 eV helium exposure causes sputtering on tungsten [18]. In fact, in these cases, electron densities in plasmas and helium flux on the surface of samples during the exposure were slightly higher than that of the case which morphology change was not observed for PM-W and VPS-W/CX-2002U, respectively. However, it is not clear that re-deposition layer was formed or not. In addition, accumulation of helium in the very near surface region may create such surface modification. Further experiments will be required to reveal the phenomena.

Helium has low solubility in tungsten but amount of desorption on PM-W was about a few times larger than that of deuterium under the nearly same exposure condition [5]. This suggests that most of helium in tungsten traps in some kinds of defects. Helium desorption started at temperatures below the exposure temperature. This is expected that as the sample cooled after the plasma exposure, the diffusing helium atoms already in the sample may become trapped in sites that remain unoccupied during the higher temperature during plasma exposure. Higher desorpion peaks were observed from the samples which their surface morphology changed. This indicates that trapping sites which has higher bonding energy were newly formed.

5. Conclusion

In the present work, surface morphology and helium retention on two kind of tungsten exposed by low energy high flux helium have been investigated. Results of observation on tungsten surface after the exposure has indicated that surface modification occurs even if at low energy irradiation. Retention of helium in the tungsten increases when the surface modification occurs. The amount of retention of helium is relatively high. Such kind of helium implanted may also eventually result in material degradation not only surface but also deeper region by diffusion at higher temperature.

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References

[1] A.A. Haasz, M. Poon, J.W. Davis, J. Nucl. Mater. 266–269 (1999) 520.
[2] F.C. Sze, R.P. Doerner, S. Luckhardt, J. Nucl. Mater. 264 (1999) 89.
[3] T. Venhaus, R. Causey, R. Doerner, T. Abeln, J. Nucl. Mater. 290–293 (2001) 505.
[4] W. Wang, J. Roth, S. Lindig, C.H. Wu, J. Nucl. Mater. 299 (2001) 124.
[5] K. Tokunaga, R.P. Doerner, R. Seraydarian, N. Noda, N. Yoshida, T. Sogabe, T. Kato, B. Schedler, J. Nucl. Mater. 307–311 (2002) 126.
[6] M. Kaminsky (Ed.), Radiation Effects on Solid Surface, American Chemical Society, 1976, p. 112.
[7] H. Ullmaier, Radiat. Eff. 78 (1983) 1.
[8] B. Emmoth, Radiat. Eff. 78 (1983) 365.
[9] R. Behrisch, B.M.U. Scherzer, Radiat. Eff. 78 (1983) 393.
[10] V.N. Chernikov, Ju.V. Lakhotkin, H. Ullmaier, H. Trinkaus, P. Jung, H.J. Bierfeld, J. Nucl. Mater. 212–215 (1994) 375.
[11] M.Y. Ye, S. Takamura, N. Ohno, J. Nucl. Mater. 241–243 (1997) 1243.
[12] M. Ye, S. Fukuda, N. Ohno, S. Takamura, K. Tokunaga, N. Yoshida, J. Plasma Fusion Res. SERIES 3 (2000) 265.
[13] K. Tokunaga, N. Yoshida, N. Noda, T. Sogabe, T. Kato, J. Nucl. Mater. 258–263 (1998) 998.
[14] K. Tokunaga, N. Yoshida, N. Noda, Y. Kubota, S. Inagaki, R. Sakamoto, T. Sogabe, L. Plochl, J. Nucl. Mater. 266–269 (1999) 1224.
[15] K. Tokunaga, N. Yoshida, Y. Kubota, N. Noda, Y. Imamura, T. Oku, A. Kurumada, T. Sogabe, T. Kato, L. Plochl, Fusion Eng. Des. 49&50 (2000) 371.
[16] K. Tokunaga, T. Matsubara, Y. Miyamoto, Y. Takao, N. Yoshida, N. Noda, Y. Kubota, T. Sogabe, T. Kato, L. Plochl, J. Nucl. Mater. 283–287 (2000) 1121.
[17] Y. Hirooka, R.W. Conn, T. Sketchley, W.K. Leung, G. Chevalier, R. Doerner, J. Elverum, D.M. Goebel, G. Gunner, M. Khandagle, B. Labombard, R. Lehmer, P. Luong, Y. Ra, L. Schmitz, G. Tynan, J. Vac. Sci. Technol. A 8 (1990) 1790.
[18] R. Behrisch (Ed.), Sputtering by Particle Bombardment I – Physical Sputtering of Single Element Solids, Springer-Verlag, 1981, p. 185.