Role of Carbon Impurities on the Surface Morphology Evolution of Tungsten under High Dose Helium Ion Irradiation

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Abstract

The effect of carbon impurities on the surface evolution (e.g., fuzz formation) of tungsten (W) surface during 300 eV He ions irradiation was studied. Several tungsten samples were irradiated by He ion beam with a various carbon ions percentage. The presence of minute carbon contamination within the He ion beam was found to be effective in preventing the fuzz formation. At higher carbon concentration, the W surface was found to be fully covered with a thick graphitic layer on the top of tungsten carbide (WC) layer that cover the sample surface. Lowering the ion beam carbon percentage was effective in a significant reduction in the thickness of the surface graphite layer. Under these conditions the W surface was also found to be immune for the fuzz formation. The effect of W fuzz prevention by the WC formation on the sample surface was more noticeable when the He ion beam had much lower carbon (C) ions content (0.01% C). In this case, the fuzz formation was prevented on the vast majority of the W sample surface, while W fuzz was found in limited and isolated areas. The W surface also shows good resistance to morphology evolution when bombarded by high flux of pure H ions at 900°C.

I. Introduction

Due to its higher melting point (3695K) and lower sputtering yield, tungsten (W) has been selected as a potential candidate for plasma-facing component (PFC) in the international thermonuclear experimental reactor (ITER)-divertor design during the D–T operational phase [1-4]. In addition, W is a plausible candidate for in-vessel mirror material because of its good optical reflectivity in a wide wavelength range. These extreme conditions promote a considerable erosion of the W PFC due to sputtering and thermal evaporation [1-2]. During reactor operation, these PFC materials will be exposed to high flux of energetic particles along with high heat loads [5]. Recently it has been noted that helium bombardment on W PFC can cause wide variety of microstructural evolution, such as dislocation loops, helium holes/bubbles [6] and fibreform nanostructure [7-8]. Note that surface morphology of PFC materials play vital role towards reliable steady state operation of Fusion devices. Formation of fine and brittle W nanostructure (Fuzz) on W surfaces after the exposure of low energy He^+ ions or plasmas relatively higher flux at elevated temperatures, have been found recently [8-11]. The presence of such fragile structure represents a serious threat for the durability of the confined plasma. The presence of such materials, due to some abnormal events, even very tiny amount, in the confined D-T plasma may lead to immediate plasma cooling (quenching) due to radiation energy losses [5, 12]. This scenario is expected if the extremely fragile W fuzz structure washed out into the confined plasma during transient events such as plasma instability [5, 12].

Recently, it has been recognized that carbon (C) contaminants on W surface may significantly reduce the formation of such brittle W nanostructure (Fuzz) [13-14]. The fuzz layer growth rate does not depend on whether the He is the dominant, or a minority, species

[13]. In fact, it can be inhibited if sufficient condensable impurities (in the case of ITER, beryllium (Be) or C) are contained within the incident particle flux to achieve a condition of net deposition and thereby allow an impurity surface layer to develop [13]. However, if the removal rate of impurities from the surface is greater than their arrival rate to the surface, then the fuzz layer may develop [13]. In the similar scenario a few studies were also performed on the understanding of plasma induced mixing on tungsten carbide (WC) [14, 15], WBe, and WBeC [16].

It has also been noted that the sputtering yield of W bombarded by C ions at room temperature (RT) was dramatically decreased with C dose at certain angles [17]. The presence of C on W surface reduced the weight loss of W due to sputtering [17]. The enhanced diffusion of C at high temperature played a significant role in modifying the results primarily observed in the case of RT bombardment [18]. The temperature has a strong effect on the range distribution of C in W. At higher temperature, C atoms are expected to penetrate more in depth in the W sample, as a result of enhanced diffusion of C in W [15, 18, 19]. Due to the reduction in C concentration near the W surface at elevated temperature, W erosion rate (weight loss) was higher. Although currently there is no intention to use the CFC (Carbon Fiber Composites) as a PFC in ITER (according to the updated ITER design) [20], still the existence of C contamination in any fusion device's plasma chamber is always a possible scenario. Studies by Ueda *et al.* [14, 15, 21] are noteworthy, where they have shown that H^+ ions irradiation (using 3×10^{24} ion/m² dose at 650°C) having even ~ 1% C can deteriorate the surface significantly and form WC on the W surface [15]. WC is a very hard and brittle material and hence, its presence on the W surface could lead to degradation in the mechanical and thermal properties of the W. Similar carbon depth distribution was also

obtained by simulation using ITMC-DYN models by us [22-23]. It was found that temperature has large influence on the chemical state of C in W substrate. In fact, the presence of C in the form of WC was favorable at higher temperature (above 700 °C) [24], and hence, unreacted C was found only near the surface, where the atomic concentration of C is higher than the stoichiometric ratio of WC (1:1) [15].

In the present study, we report on the effects of carbon impurities on the surface morphology evolution of W PFC under high dose He⁺ ion irradiation in extreme conditions. Although few studies have already reported perversely on the similar topic [13-15, 21-24]; the effects of intermixing of PFC materials, during plasma and/or ion exposure, are still not well understood. Our study shows the responses of the W as PFC surface during He⁺ ion irradiation as a function of C concentrations (tiny amount). Note that the C divertor is no longer part of the current ITER design. The temperature and ion energy spectrum in the ITER divertor are currently believed to fall outside the fuzz formation window in the expected detached plasma regime. Nevertheless, fuzz formation window in the hypothetical W first wall DEMO could still be significant as well as unexpected abnormal ITER operations. In these devices, small amounts of C seeding might be considered, not just for the benefits of radiative cooling, but also to inhibit fuzz. Additionally small amounts of C in the DEMO may not necessarily be a retention problem due to the high operational temperature.

II. Experimental details

The experiments have been performed in the IMPACT (Interaction of Materials with Particles and Components Testing) laboratory of center of materials under extreme condition (CMUXE) at Purdue University. Multiple pure (99.95%) W sheets (0.5 mm thick) were cut

in 10×10 mm and mechanically polished to a mirror like surface finish. After that, each sample was inserted into our irradiation chamber. Prior to He^+ ion irradiation, each sample was sputter cleaned by a high flux of 1 keV Ar^+ ions for around (0.5 to 1 min) at RT. After that temperature of the sample (hereafter target temperature) was increased to 900°C and then irradiated by a high flux (3.2×10^{20} ion/ m^2s at beam center) of 300 eV He^+ ions with various C concentrations for each sample. The total dose of ion irradiation for all samples were fixed at about 1.0×10^{25} ion/ m^2 . The variation of the ion beam composition was achieved by varying the concentration of the gas mixture (He and CH_4) fed into the irradiation ion gun. Both Ar^+ and He^+ ion irradiations were performed using our KRI-KDC-10 ion source gun. Base and working pressure of the UHV chamber were $\sim 2 \times 10^{-9}$ and $\sim 1 \times 10^{-3}$ torr, respectively. Five different cases of ion beam composition were considered in this investigation, viz., pure He^+ ion; 97.5% He^+ mixed with 0.5% C^+ and 2% of H^+ ions; 99.75% He^+ mixed with 0.05% C^+ and 0.2% H^+ ions; 99.95% He^+ mixed with 0.01% C^+ , and 0.04% H^+ ions, pure H^+ ions. The surface composition of each irradiated sample was observed by XPS. The XPS data analysis and chemical lines fitting were done by using CasaXPSTM program. All the XPS spectral line shapes were chosen to be asymmetrical Gaussian/Lorentzian sum formula, and a Shirley background subtraction was done. The surface morphology of the irradiated sample surfaces were evaluated by scanning electron microscopy. Cross section imaging was performed using FEI $\times$ T Nova NanoLab Dual Beam focused ion beam/scanning electron microscope (FIB/SEM).

III. Results and discussion

Several pure W samples were irradiated by He^+ ion beam with various carbon compositions. Five sets of experiment have been performed and the results are presented in the following sections.

III.A. Pure He irradiation

The first sample was irradiated by a pure He^+ ion beam (3.2×10^{20} ions/m²s flux at beam center) for ~ 9 hours. Surface darkening was noticed on the sample surface after the pure He irradiation. The sample surface tends to change to dark or black color in response to the change in the surface optical properties due to the formation of very fine nanostructure (W fuzz). The surface darkening in our He irradiated sample can be observed by comparing the sample before and after He^+ ion irradiation. The chemical composition of the sample surface after irradiation was measured by XPS. The XPS spectrum of the irradiated surface shows a typical pure W spectrum. The wide XPS survey (shown in Figure 1-a) shows mainly W peaks in addition to a weak oxygen peak. We believe that the presence of oxygen on the sample surface is due to water adsorption that might have occurred in the vacuum chamber after the He^+ ion irradiation process. This fact was confirmed by the absence of W oxide peaks in the high resolution XPS spectrum of the W4f region presented in figure 1-b. As it can be seen clearly in this figure, only three peaks represent the pure state of W were found. The W $4f_{7/2}$ and $4f_{5/2}$ were located at 31.4 and 33.58 eV, respectively. The shift between these two doublets was found to be exact at 2.18 eV. This value of shift and the position of the W $4f_{7/2}$ peak were in agreement with the most accepted peaks positions in literature that represent the pure state of W [26]. The third small peak on the higher binding energy side of the W 4f doublet is also representing the pure state of W5p level.

The surface structure of the irradiated sample was observed using SEM. As we mentioned earlier, W fuzz was formed on the sample surface after a dose (1.0×10^{25} ions/m²) of He irradiation. Figure 1-c shows SEM image of the irradiated surface. The image is taken at 52 degrees to help observe the depth of the structures. Very fine nano-rods of W were formed on the surface. This surface morphology evolution due to pure He⁺ ion irradiation at elevated temperature was expected and is in agreement with other similar studies [8, 11, 27-28]. The reason behind the formation of this nanostructure was justified by many different hypotheses [8, 11, 27, 29-31]. In summary, it is believed that the formation of He bubbles beneath the W target surface during the irradiation process plays a significant role in W fuzz formation. The growth and the coalescence of these He bubbles can significantly reshape the exposed surface and subsurface regions.

III. B. He⁺ ion irradiation with 0.5% C⁺ ions

In the next experiment we introduced 0.5% C⁺ ions into the He⁺ ion beam and study the W surface evolution after exposure to this mixed ions beam at 900°C of surface temperature. A new W sample was irradiated by the mixed ions beam for a total dose of 1.0×10^{25} ions/m². Shortly after the start of irradiation a visible C layer was developing on the sample surface. This C layer was noticed to be increasingly darkened over the course of bombardment. After the end of the irradiation process, the sample was cooled down and the surface composition was measured by XPS as shown in Figure 2 (a and b). The wide XPS survey scan shown in figure 2-a shows that the sample surface was fully covered with pure C. The C1s region was scanned by high resolution XPS (shown in figure 2-b) and shows a C 1s peak situated at 284.5 eV. This peak position emphasizes that the C layer was in a graphitic form. The main

conclusion of this experiment is the existence of thick pure C on the sample surface with no distinctive features. This full carbon coverage prevented the formation of W fuzz on the sample surface. Also we concluded that this percentage of C impurity within the He ion beam is very high. Our next step is to repeat the experiment with much lower C percentage.

III.C. He⁺ ion irradiation with 0.05% C⁺ ions

A newly prepared sample was irradiated at high temperature (900°C) by a mixed He⁺ ion beam composed of 99.75% He⁺, 0.2% H⁺, and 0.05% C⁺ ions. Similar to the previous irradiation cases, the ion beam flux at the beam center was 3.2×10^{20} ions/m²s and the irradiation was carried for 9 hours reaching a total ion dosage at the sample center of about 1.0×10^{25} ions/m². The main observation, after the completion of ion irradiation experiment, no significant change in sample surface by visible appearance. Unlike the cases of the pure He⁺ ion irradiation where the sample surface darkened by the formation of fuzzy layer and the other case of high C percentage irradiation where the surface was darkened by a formation of thick C film. The chemical composition of the sample surface after the irradiation was measured by XPS. A wide-scan XPS survey for the irradiated sample (not presented) shows a typical W peaks along with C peak without any other contaminants. The total C composition of the sample surface was found to be around 20%. More detailed look into the C1s and W4f of XPS spectral regions are presented in Figure 3-a and b. The C1s region shown in figure 3-a shows the presence of C on the surface. The C peak in this region was positioned at 283.2 eV. This peak position represents the most accepted peak position value for C in the form of WC. This finding emphasize that most C exist on the sample surface was actually in WC phase. Another shorter peak was needed to fit the higher binding energy side of the prominent WC peak as can be seen in Figure 3-a. This peak was found to

be situated at 284.5 which represent the graphitic form of C. Thus, the XPS data in the C1s region shows that a major part of the surface C (63%) exists in WC phase and the remaining part (37%) was still in pure C (graphitic) phase. On the other hand, the W 4f region (figure 3-b) shows the coexistence of the W in a pure phase as well as in the WC phase. Here, the presence of WC phase in the W 4f region confirm our finding in the C1s region.

The surface morphology of the irradiated sample was observed by SEM. As it can be seen in figure 3-c, the sample surface after irradiation was relatively smooth without any distinctive features (compared to fuzz structured surface). This finding also confirms our previous observation of no visible surface darkening was seen after irradiation. The reason behind the observed mitigation of the W fuzz formation could be due to the presence of WC on the sample surface.

III.D. He⁺ ion irradiation with 0.01% C⁺ ions

The next W sample was irradiated by He⁺ ion beam with even lower C content. The goal of performing this irradiation is to see how the surface responds to less C contamination. This sample was also irradiated at 900° C and by similar ion beam flux and similar ion fluence. The general appearance of the sample surface after irradiation shows no change in color similar to what we observed in the previous irradiation at higher C content. The sample surface composition after the irradiation process was measured by XPS. The wide XPS survey of the irradiated sample surface shows typical pure W peaks with relatively smaller C peak. The total surface C composition was found to be less than 10%. More careful look into the C1s and the W4f regions are presented in figure 4-a and b. The XPS C1s region presented in figure 4-a shows a minor presence of C in the form of WC with a peak positioned at 283.2

eV. This observation was also confirmed by the presence of WC peaks along with Pure W peaks in the W4f region as presented in figure 4-b.

The SEM images of the irradiated surface shows very interesting phenomenon as presented in figure 4. The vast majority of the irradiated surface shows very good immunity against fuzz formation where the surface was found to be smooth with no fuzz formation. Only the W fuzz appears to grow in very limited and isolated area on the sample surface (fuzz islands). These fuzz islands were randomly dispersed on the sample surface with large variation in size, shape, and fuzz thickness. Figure 4-c shows an example of small region on the surface where a thick W fuzz structure was found and surrounded by an unaffected sample surface. Similar W fuzz formation were widely reported to be formed on a wide variety of W material grades after high fluxes of He plasma exposure [11, 27, 30, 35]. It is also worthwhile to mention that not all of the fuzzy islands are having the same characteristics. Some of these islands are very thin and short (having short nano-fibers). Figure 5 shows some examples for different fuzzy islands. Cross-sectional SEM image was made for one of the thickest fuzzy islands to take a closer look on the surface beneath the fuzz (Figure 5-a). As it can be seen in this image, the surface beneath the fuzz is still flat and smooth while a the entire W fuzz structure appears to be developed above the surface without the aid of subsurface bubbles growth and coalescence mechanism. The SEM image in figure 4-b shows a region of the surface where W fuzz was barely able to grow. In such regions, the surface exhibit the formation of dispersed very short nano-rods of W that grow above the surface and also surrounded by smooth areas.

Beside all of the different surface phenomena that we mentioned in this case of low C content He ion beam irradiation, in some limited areas, the surface also exhibit the formation of very

long W nanoscopic fibers (nanowires-like) that tend to grow horizontally on the sample surface. These W nanowires were found to be always attached or very close to the fuzz covered areas. Most of these nanowires were branched into several nanowires, which altogether create what appears to be a network of nanowires covering the surface as shown in figure 5-c. The reason behind the formation of such nanowires under these conditions of He^+ ions irradiation is not well understood. We believe that these nanowires is most likely formed due to the same mechanism of fuzz formation. The differences could be the fuzz root density. In the case of pure He irradiation, large number of W nano-rods was found to evolved above the surface with a high areal density (i.e., large number of nano-rods per unit of surface area). Due to the high number of these growing features, the rate of W atoms accumulation is to be distributed equally on all these features. Therefore, the average height of these growing parts of the surface is expected to be low. On the other hand, having much lower number of growing surface spots could results in high rate of growth and very long nano-fibers formation. In fact, the areal density dependence of the nano-rods length can be observed easily by comparing areal density of the nano-rods in thick fuzz islands (seen in the x-sectional SEM image presented in figure 4-e and f) to the areal density of the nano-rods that was formed in the case of pure He irradiation (presented in Figure 1-c). We believe that the factor dictates the areal density and the distribution of the growing surface spots is still not well understood. A one main observation that can be drawn from this part of the experiment is that the process of W nanostructure formation in response to He ions or plasma irradiation, might be due to an active surface atomic transport mechanism rather than due to the formation, growth and coalescence of He bubbles. this later mechanisms cannot justify the formation of very long W nanowires that in some cases was found to be 20 to 30 μm long.

III.E. Pure H⁺ ion irradiation

The last sample was irradiated by a pure H⁺ ion beam at 900°C. The goal of performing this irradiation is to check whether the small percentage of the H⁺ in our previous irradiations will have any significance in terms of W surface morphology evolution. The H⁺ ion beam used in this sample irradiation experiment has a maximum flux at the beam center (sample center) of about 1.0×10^{21} ions/m²s. The irradiation was carried for 1×10^4 s giving a total H⁺ ion irradiation dose of about 1×10^{25} ions/m² at the sample center. The post-irradiation XPS measurement (not shown) shows nothing but a typical pure W sample with no contaminations. The sample also did not show any change in its physical appearance after the H⁺ ion irradiation process. The post-irradiated surface appeared to have its original bright metallic finish without any darkening or discoloration. SEM images of the irradiated surface at two different magnifications are shown in figure 6. As it can be seen clearly in figure 6-a, most of the sample surface was found to be clear with no distinctive features such as surface pinholes or blistering. Only in some very limited dispersed areas, the surface was found to form a kind of self-organized rippled structure as shown in figure 6-b. This distinctive structure was found to be very frequent in many areas on the sample surface. The formation of this distinctive structure is most likely a crystal orientation dependent.

IV. Conclusions

Several W samples were irradiated by He⁺ ion beam with various percentages of C⁺ ions. The W surface irradiated by pure He⁺ ion beam at elevated temperature exhibit the formation of wide spread W nano-fibers or fuzz structure. This phenomenon of He⁺ irradiation induced fuzz formation can be prevented in the presence of C contamination within the He ion beam.

We found that, a thick graphitic like C layer was formed on W surface after irradiation by He^+ ion beam mixed with 0.5% of C ions at 900°C . The formation of such layer prevented the formation of W fuzz on the sample surface. Lowering the C^+ ion % in the He^+ ion beam by an order of magnitude (0.05% C) was also effective in preventing W fuzz formation. In this case of lower C content ion beam irradiation, much lower C composition reside on the sample surface and therefore, the formation of WC on the surface was the effective mechanism for W fuzz mitigation. The effect of W fuzz prevention by WC formation on the sample surface was more noticeable when the sample was bombarded by He^+ ion beam with even lower C^+ ions content (0.01% C). In this case, the fuzz formation was prevented on the vast majority of the W sample surface. However, W fuzz in this case was formed in limited and isolated areas dispersed randomly on the sample surface (fuzzy islands). The W fuzz structure in such fuzz-covered areas was not uniform being thick in some areas and thin in others. We also observed the formation of very long W nano-fibers that appear to grow horizontally on the sample surface. The W surface shows no significant morphology evolution when bombarded by high flux of pure H^+ ions at 900°C . In this case, the vast majority of the bombarded surface shows no changes after irradiation while a formation of self-organized rippled structure appears to form in very small and dispersed areas on the sample surface. More analysis is needed to fully understand W surface evolution and fuzz formation during He^+ ion irradiation in the presence of minute C impurities.

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Figure 1. XPS of the sample irradiated by pure He⁺ ion beam. XPS full spectrum (a), and high resolution XPS spectrum for the W 4f region (b). Part (c) shows a SEM image of the nanostructure (Fuzz) covering on the W sample surface, the sample was irradiated by pure He⁺ ions (1.0×10^{25} ion/m²fluence) at 900°C.

Figure 2. XPS of the sample irradiated by 97.5% He^+ , 0.5% C^+ and 2% H^+ ion beam. XPS full spectrum of the C coat on the top of the original surface (a), high-resolution XPS spectrum for the C1s [region](#) (b).

Figure 3. XPS of the sample irradiated by 99.75% He^+ , 0.05% C^+ and 0.2% H^+ ion beam. High-resolution XPS spectrum for the C 1s region (a) and high-resolution XPS spectrum for the W 4f region (b). SEM images of the sample surface irradiated by 99.75% He, 0.05% C and 0.2% H ion beam (c).

Figure 4. XPS of the sample irradiated by 99.95% He^+ , 0.01% C^+ and 0.04% H^+ ion beam. High resolution XPS spectrum for the C1s region (a) and high resolution XPS spectrum for the W 4f region (b). SEM images of the sample surface irradiated by 99.95% He^+ , 0.01% C^+ and 0.04% H^+ ion beam. the vast majority of the sample surface was found to be smooth with no fuzz, isolated fuzz islands found to be dispersed randomly on the sample surface (c),

Figure 5. SEM images of the various W fuzz islands found on the surface after irradiation by 99.95% He^+ , 0.01% C^+ , and 0.04% H^+ ion beam at 900°C of surface temperature. Cross-sectional SEM image showing the sample surface beneath one of the thickest fuzz islands (a). SEM image of a barely fuzzy region (b). Long W nanowires that was found be attached to one of the fuzzy islands (c).

Figure 6. SEM images of the sample surface irradiated by pure H^+ ion beam at a surface temperature of 900°C (a) Lower magnification SEM image. (b) Higher magnification SEM image showing self-organized rippled structure.