Low energy helium ion irradiation induced nanostructure formation on tungsten surface

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Abstract

We report on the low energy helium ion irradiation induced surface morphology changes on tungsten (W) surfaces under extreme conditions. Surface morphology changes on W surfaces were monitored as a function of helium ion energy (140 – 300 eV), fluence (2.3×10^{24} - 1.6×10^{25} ions m⁻²), and flux (2.0×10^{20} - 5.5×10^{20} ion m⁻² s⁻¹). All the experiments were performed at 900° C. Our study shows significant effect of all the three ion irradiation parameters (ion flux, fluence, and energy) on the surface morphology. However, the effect of ion flux is more pronounced. Variation of helium ion fluence allows to capture the very early stages of fuzz growth. The observed fuzz growth and morphology changes were understood in the realm of various possible phenomena. The study has relevance and important impact in the current and future nuclear fusion applications.

1. Introduction

Tungsten (W) is currently the most promising candidate materials to serve as a plasma-facing component (PFC) in future fusion devices [1]. In addition to its higher melting point, W has good thermal conductivity and lower sputtering yield when bombarded by energetic light ions such as Helium (He) and Hydrogen (H) isotopes [1, 2]. Moreover, W has shown low H isotopes

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retention characteristics compared to other PFC candidate materials[3]. On the other hand, W being a high-Z element is not favorable to be mixed with the DT plasma. Contamination of high-Z materials in the plasma can enhance the plasma radiative heat loss considerably, leading to a significant reduction in confinement time or even plasma quenching at certain contamination levels [4]. The importance of studying the behavior of W-radiation interaction rises from this important point. Many research groups around the world have performed a wide variety of experiments on W response to conditions similar to fusion reactor environments. Most of these studies have investigated the response of W exposed to high fluxes of He, deuterium (D) or He/D plasmas [2, 5-10]. In fusion reactors, including ITER (International Thermonuclear Experimental Reactor) and DEMO (DEMONstration Power Station), PFCs including W are exposed to high fluences of low-energy He^+ ions [6, 11, 12], which motivates performing these experiments.

A common conclusion among all recent studies is the fact, that W surface, under high fluxes of He plasma exposure, can form very fine nanostructures (nano-fibers) or “fuzz” [2, 6, 9, 11]. W fuzz can grow to high thicknesses (as thick as few μm) on the top of the original W surface [6]. W-fuzz is a very fragile structure that can be detached from W surface during sudden stress or high-heat loads [13, 14]. The fuzz formation is unfavorable scenario where W fuzz may develop during the early stages of normal operations of future fusion devices [8, 13] and could severely affect the PFC performance. This specially formed structure was found to occur in most of the different grades of W and W-alloys that are being considered in future devices [6]. Fuzzy W surface can be a significant source of W (high-Z) dust particles which can pose a serious threat for the sustainability of a stable fusion reaction [15, 16]. Therefore, minimizing the possibility of formation of high-Z dust is a priority in PFC material selection [16]. In fact, many transient events due to plasma instability such as ELMs and disruptions can create enough stresses and thermal loads to break or easily remove the W fuzz [17]. Therefore, contamination of plasma due

to the presence of fuzzy W surfaces is always a serious concern [8, 13, 18]. Additionally, the presence of W fuzz on a W surface may alter and significantly degrade the W thermal properties [17].

To the date, researchers have concluded that the major factors significantly influence the W fuzz growth are surface temperature, incident He ion-energy, -flux, and -fluence[18]. The lower energy threshold for fuzz growth is 12 to 30 eV[2, 19, 20] depending up on sample grade, plasma conditions, and surface temperature used during the experiments. The fuzz growth also has the lower ($\sim 900\text{K}$) and upper ($\sim 2000\text{K}$) temperature thresholds, referred as “temperature window”. In general, below 900K pits and blisters appearance were observed. The studies performed by Wright *et al.* is also relevant in where they have observed W nano-tendrils in the divertor region of a high-power density tokamak experiment [21]. In addition to this, a series of experiments have been performed extensively on high-Z and high-melting point materials in tokamak environment and/or in laboratory plasma accelerator systems, using comparatively low energy H and He ions[5, 22-27]. However, the physical mechanisms responsible for such growth are not yet very clear [5]. Although most of the reported fuzz formation cases happened when the surface is bombarded by He^+ ions having lower energy than the energy required for displacing the target atoms [5, 6, 28], a considerable surface modification was not seen. Many researchers believe that the reason behind the fuzz formation is mainly due to the role of He bubbles that form just beneath the surface [2, 29-31]. The formation and the growth of these bubbles were widely reported in many TEM studies [32-34]. He bubbles formation and growth in W due to He^+ ion irradiation occurs via two main scenarios. The first is when the He^+ ion energy is greater than the energy required to displace the W atoms from their lattice sites. In this case, along with the high percentage of solute He atoms in the W matrix, a considerable amount of point defects in the original W lattice, is formed. These point defects include short lived self-interstitials and

relatively long-lived vacancies. A fraction of these point defects survives long enough to migrate and agglomerate to create larger two dimensional (2D) defects (such as a dislocation loops, caused by the agglomeration of interstitials) and 3D defects (such as voids, caused by the agglomeration of vacancies). The presence of voids will allow the He solutes to migrate freely into these voids. As a result of He accumulation in these voids, an internal He pressure can build up, leading to vacancies absorption in the process of void growth [35]. In this scenario the growth rate will depend solely on the rate of vacancies migration as well as on the internal He pressure inside these bubbles [35]. The second scenario is expected to occur in the case of low energy He^+ ions (lower than the energy required to cause atomic displacement). It is also the case for most of the recent experimental observations where the W-fuzz was reported after He plasma exposure [5, 6, 28, 36]. In these cases, nano-scale He bubbles can be formed due to the migration of He atoms (solute) into preferred sites in the W lattice (such as dislocations, grain boundaries, and vacancies, etc.) [30, 37, 38]. The accumulation of He atoms in these site can build up an internal He pressure that is enough to make the newly nucleated bubbles to grow by emitting self-interstitials and dislocation loops [38, 39]. The formation of He bubbles in this scenario is much more difficult than in the first one. In fact, one condition required for the growth of these He bubbles is that the internal He pressure inside these bubbles to be greater than the strain free energy associated with squeezing self-interstitials into the bubble's surrounding lattice. Moreover, the rate of self-interstitials emission determines the rate of the volumetric growth of the He bubbles. The mechanical strength of the surrounding W matrix is another factor which need to be considered where He bubbles are situated near the surface. Blistering occurs when the He bubble internal pressure is greater than the materials' mechanical strength that separates the He bubbles from the outer surface (bubble cap). Therefore, deformation, cracking, or even complete breakage of the cap materials are common blistering features [7, 40-42].

On the other hand, the increased He internal pressure inside these newly formed bubbles also promotes the backward migration of the He atoms from the bubbles to the W lattice [35]. Due to this fact, the internal pressure of He bubbles is expected to be maintained at a fixed value (at equilibrium) during the bubble growth process. The internal He equilibrium pressure is largely depends on the He concentration in bubble's surrounding lattice. Therefore, only appreciable He concentration in the matrix surrounding the He bubble will guarantee a forward migration of the He atoms into the bubble even at high bubble pressure. The maximum achievable He pressure will always be dictated by the He concentration in the bubble's surroundings and therefore the bubbles growth rate is also He concentration dependent. On the other hand, the flux of He ions during the bombardment governs the He concentration in the sample matrix. Also, the He concentration is expected to vary with depth. For these reasons the bubble formation is always preferred to occur near the irradiated surface [29] where the He concentration is maximum. The temperature of the sample is another important factor because it governs the energy of formation of self-interstitials in the W lattice and the rate of interstitial diffusion. Self-interstitial formation rate will be higher for a higher lattice temperature.

In this study, we focus on the evolutions and changes in the W surface morphology due to He^+ ion irradiations having ion fluence (2.3×10^{24} - 1.6×10^{25} ions m^{-2}), ion flux (2.0×10^{20} - 5.5×10^{20} ion $\text{m}^{-2} \text{s}^{-1}$) and ion energy (140 -300 eV). Although a few studies have been performed on the similar topic with similar ion flux range, the uniqueness of our investigation is predicting and demonstrating the very early stage of the W-fuzz structure formation, due to low energy He^+ ion beam irradiation at elevated temperature.

2. Experimental methods

The experiments were performed in the IMPACT (Interaction of Materials with Particles and Components Testing experimental) facility at the center for materials under extreme environment (CMUXE), Purdue University [43]. Several W samples of 10 mm $\times$ 10 mm were cut from cold-rolled W sheet having thickness of 0.5 mm with pure (99.95% W) samples. All W samples were mechanically polished to a mirror- finish. Each sample was sputter cleaned by 1 keV Ar⁺ ion beam (current density ~ 10 mA cm⁻²) for 2 minutes at room temperature (RT). After that, each sample was heated to 900^o C and irradiated at normal incidence. Ion irradiation experiments were performed in three different cases: as a function of (a) ion fluence (2.3×10^{24} - 1.6×10^{25} ions m⁻²) at constant ion flux (5.5×10^{20} ion m⁻² s⁻¹) and energy (300 eV), (b) ion flux (2.0×10^{20} - 5.5×10^{20} ion m⁻² s⁻¹) at constant ion fluence (1.0×10^{25} ions m⁻²) and energy (300 eV), and (c) ion energy (140 -300 eV) at constant ion flux (5.5×10^{20} ion m⁻² s⁻¹) and fluence (1.0×10^{25} ions m⁻²). Both Ar (for surface cleaning) and He ions irradiations were performed using KRI-KDC-10 ion source that is attached to the irradiation chamber in IMPACT facility. The base pressure of the UHV chamber was $\sim 3.0 \times 10^{-9}$ torr, however the working pressure (during He⁺ ion irradiation) was 1.0×10^{-3} torr, which was kept constant for all experiments. In-situ X-ray photoelectron spectroscopy (XPS) was performed after the irradiation process to assure the purity of the ion irradiation processes. The XPS data analysis and chemical lines fitting were done using commercial CasaXPS software. All the XPS spectral line shapes were chosen to be asymmetrical Gaussian/Lorentzian sum formula and a Shirley background subtraction were performed. The effects of He⁺ ion incident particle energy on the surface morphology evolution were studied by irradiating positively biased samples at various bias voltages. The surface morphology of the irradiated sample surfaces was evaluated using scanning electron microscopy “Hitachi S-4800 Field Emission SEM”.

3. Results and discussions

a. Effect of He⁺ ion fluence

The impact of the total He⁺ ion fluence on the fuzz evolutions were studied by irradiating several W samples at a fixed ions flux (5.5×10^{20} ion m⁻² s⁻¹) by varying irradiation time duration. All samples were heated during irradiation to a constant temperature of 900° C. The first sample was irradiated for 70-minute achieving total fluence of 2.3×10^{24} ions m⁻². Figure 1(a) shows SEM image of the irradiated surface. All the SEM images shown in Fig. 1 were taken after tilting the sample at 52°. At this stage of irradiation tiny nano-features started to grow on the sample surface. It seems this stage is the very early stage of “fuzz formation” [2]. The second samples were irradiated at the same conditions for longer time duration (166 minute) for achieving the total fluence of 5.5×10^{24} ions m⁻². The irradiated surface is shown in Fig. 1(b). As it can be seen in this SEM image, the small nano-features have grown bigger and they started to have more irregular shapes. After that, another sample was irradiated with a higher He⁺ ion fluence (9.2×10^{24} ions m⁻²) and the resulted surface morphology is shown in Fig. 1(c). The SEM images presented in this figure show a considerable vertical growth of previously noticed nano-features. The figure shows that the ion fluence enhancement causes denser and tangled fuzz evolutions. Furthermore, higher degree of vertical fuzz growth structure (Fig. 1 (d)) was observed on increasing ion irradiation time duration (for achieving He fluence of 1.6×10^{25} ions m⁻²). Although the information shown in Fig. 1 is vital to demonstrate the vertical fuzz evolution of the observed nanostructure, a clear understanding of the evolution mechanism is yet to come. In another approach, we recorded the SEM images at 0° sample tilt, for monitoring the changes in lateral evolution.

Figure 2 shows a comparison of the surface nanostructure appearances, due to 52° sample tilting and without sample tilting (0°). The figure shows, the initial stage of W fuzz formation, for ion fluence of 2.3×10^{24} ions m⁻² He⁺ ion irradiation. In fact, Fig. 2(a) is a high magnification image

from the region in Fig. 1(a), which shows nanostructure appearance on the surface. On the other hand, the 0° case (without sample tilt) reveals the presence of a network of surface holes in between the nano-tendrils (Fig. 2(b)).

Figure 3 shows the SEM images from the same samples (as above, in Fig. 1 (a)) but with 0° sample tilt and at higher magnifications. This part of the experiment was performed to show the lateral surface evolutions due to He^+ ion irradiations in extreme conditions, which may not appear in the case of titled (52°) samples. Figure 3 clearly demonstrates the appearance of nanoscopic hole (pinholes) networks between the surface nano-structures (which evolved to nano-tendrils at higher fluences), which we could not see in the sample tilted (52°) case (Fig. 1). Figure 3 also shows an enhancement in the hole size broadening with increasing the He^+ ion fluence. The enhancement in the hole size may be understood in the realm of coalescence between the adjacent holes. Such coalescence is evidenced by hole shape transformation from smaller (Fig. 3(a)) to bigger and irregular shape (Figs. 3(b) - (d)). At the same time, thinning of the walls (that separate these holes) is also noticeable. Considering the “surface nano-feature vertical growth” between these nanoscopic holes, as observed in the sample tilting case (Fig. 1), it is clear now that the “excess materials,” which results from the “growth” and the “coalescence” of the surface holes, migrate upward and cause the out-of-plane nanostructure growth.

Similar nanoscopic holes after high fluence He plasma exposures on W surfaces were also previously reported by several researchers [32, 44, 45]. The hypothesis suggested by Kajita *et al.* [2] is more relevant for explaining the pinhole formation in our study. In their hypothesis, they rely on the localized temperature gradient that might be formed within the W sub surface region due to the presence of progressively growing He bubbles. Our results show similar surface

pinhole formation phenomena. The role of He bubbles formation and growth, within the lattice of irradiated W, may be another anticipated factor, for such surface morphology evolutions on W surfaces due to low energy He^+ ion irradiations [5]. Further, in the evolutions of such ultra-fine W-fuzz structures, the surface energy has a significant role over the other energies due to the large surface to volume ratio. This explains the unfeasibility to find naturally grown fuzz structure for any metallic system. Another possibility for such structural transformation, disregarding the surface energy constraints, may be the recrystallization phenomena.

The final stage that can lead to formation of the nano-structure is the surface diffusion and the redistribution of the newly arrived surface atoms (*i.e.*, the self-interstitials). These atoms may arrive and set singly on the top surface. In this situation, these surface atoms will have a minimal binding energy and they are susceptible to diffuse freely on the top surface. The experimental observation confirms the progressive growth of only small part of the surface to form the nano-tendrils structure. We assume that the tip of these nano-tendrils is the preferred destination for the surface W diffused atoms. In an intense surface diffusion situation, any local increase of the surface binding energy in some surface spots will create a net-flow of atoms toward these spots and allow them to grow.

The chemical composition of irradiated sample has also been monitored from XPS, in *in-situ* condition. As shown in Fig. 4, the XPS spectrum shows a pure W peaks with no contamination. We only noticed a minor oxygen peak [46] at 530 eV as can be seen in Fig. 4(a). This minor oxygen presence in the surface is most likely due to some water adsorption on the target surface occurred after the irradiation process [47]. High resolution XPS of the W-4f region (Fig. 4(b)) confirms the absence of W- oxide and supports our conclusion that the presence of oxygen was due to water adsorption. In this region only peaks of pure state of W were found. The W 4f $_{7/2}$

was found to be situated at 31.4 eV, while the W 4f_{5/2} was positioned at 33.57 eV. These values were found to be in agreement with most accepted peaks values for pure state of W [46].

b. Effect of He⁺ ion flux

Three additional W samples were prepared in similar condition and irradiated by 300 eV He⁺ ion beam using 2.0×10^{20} , 3.2×10^{20} , 5.5×10^{20} ions m⁻² s⁻¹ fluxes. Other irradiation parameters such as sample temperature (900° C) and He⁺ ion fluence (1×10^{25} ions m⁻²) were kept constant for all the three samples. As evidenced, in SEM images (Fig. 5), and also in the physical appearance of the irradiated samples, W nano-tendrils growth increases with increasing He⁺ flux. Despite the long irradiation time for the sample that was bombarded by low ion flux (2.0×10^{20} ions m⁻² s⁻¹) the sample surface after irradiation shows very fade darkening (almost no change). Moreover, the nanostructure of the irradiated surface was found to be still in the very early stages of fuzz formation as shown in Fig. 5 (a).

Increasing the He⁺ ion flux to (3.2×10^{20} ions m⁻² s⁻¹) cause a noticeable darkening in the sample fuzz region. The surface SEM images confirm the growth of W fuzz in this region as shown in Fig. 5(b). The third sample confirms the trend of flux dependent W fuzz growth rate. This sample was irradiated at higher flux (5.5×10^{20} ions m⁻² s⁻¹) for shorter period. The irradiated sample surface was darker than the previous two samples. Moreover, the W fuzz was found to be more advanced and much finer than the previous cases as shown in Fig. 5(c).

In addition to the He⁺ ion fluence dependency of the W fuzz evolution phenomenon, the He⁺ ions flux found to have an important role. Higher He⁺ ion flux results more fuzz growth and vice versa. We believe that helium flux governs the rate of nano-tendrils' growth by two main roles; the first one can be understood in the realm of the fact that He⁺ ion flux is the most important

factor that determines the He concentration in the W subsurface lattice during irradiation. This concentration as we believe should be much higher than the equilibrium solubility limit of the W lattice. Thus, nucleation and growth of helium bubbles is the expected response to reduce Helium concentration from W lattice. Hence, Ion flux is the parameter that governs the internal He pressure in the He bubbles. Therefore, the He bubbles internal pressure dictates the rate of self-interstitials emission which in turn determines the rate of fuzz growth.

The second role can be understood from the role of high helium lattice concentration in slowing down or jamming the W self-diffusion process. We believe that the presence of high concentration of He within the W lattice will jam and freeze the W atoms self-diffusion process. The self-diffusion process mostly occurs when a lattice atom jumps into a vacant site. Therefore, it is hard to imagine the presence of vacant lattice sites within a supersaturated W structure. Instead, all the vacant sites will be filled with multiple He atoms (He-vacancies complexes). This He occupation will restrict the jumping of the W atoms into the occupied vacant site. Thus, self-diffusion process will be largely parallelized in high flux Helium ions irradiation situation. This hypothesis of jammed W self-diffusion is necessary to explain the stability of the surface elongated features (with large interfacial energy) such as the W nano-tendrils during the He^+ ion bombardment. Otherwise, a backward bulk diffusion process is expected to initiate an internal backward flow of W atoms from the tip of nano-fiber toward its root to minimize the increasing surface energy of the system. This trend of reversible flow was confirmed by annealing of the fuzzy W surface to a high temperature in absence of Helium irradiation. Hence, obvious reduction in the W-fuzz height was reported[48-50].

The observation of reduction in W- fuzz thickness after annealing is very useful as it leads us to the fact that, W-fuzz structure is a thermodynamically unstable structure. Therefore, it has the

ability (upon being thermally activated to enhance the self-diffusion process) to reverse itself to lower its total free energy. The unstable pure metallic structures appear either when the transformation is too fast, or when the self-diffusion process is too slow to maintain the possible minimum free energy. In fact, the transformation of the W sample from a smooth to fuzzy surface is a slow process, and is developed over relatively long time. Therefore, W self-diffusion process must be significantly slow during the He irradiation process. This agrees with our hypothesis of the role He concentration within the W-lattice in mitigating the W self-diffusion process during irradiation. This He concentration that we believe, it is being dictated by the helium ion flux.

It is worthwhile to mention that, the ion flux used in the present study is nearly 0.5 times smaller than the typically used ($\sim 1 \times 10^{21}$ ions $\text{m}^{-2} \text{s}^{-1}$), to grow the fuzz structure surface modifications and pinholes because of He bubble formation[50]. However, in ITER, the ion flux can be of the order of $\sim 1 \times 10^{25}$ ions $\text{m}^{-2} \text{s}^{-1}$ [50].

c. Effect of He^+ ion energy

To understand the effect of variation of ion beam flux profile (due to ion beam energy changes) on surface morphology, incident ion beam energy (at constant ion flux, fluence, and temperature) was varied and corresponding surface morphology changes was monitored, using SEM. The results were summarized in Fig. 6. For changing the incident ion beam energy, samples were positively biased using external power supply. Such sample biasing will generate an electrical field (in proportion to applied external voltage) which will decelerate the incident He^+ ions. The first sample was biased by +160 V and therefore, the ions are expected to hit the surface by an incident energy equal to 140 eV. Although the samples were irradiated at high flux for 5 hours, the surface structure (nano-tendrils) (Fig. 7(a)) was found to be much shorter than what we

usually see at the same irradiation fluence and flux when we bombard the surface by 300 eV He^+ ions (Fig. 7(c)). The surface structures, after 200 eV ion irradiations, grew to an intermediate stage that is somewhat in between the 300 eV and the 140 eV cases (Fig. 6(b)).

4. Conclusion

A series of pure W samples were irradiated by high fluxes of low energy He^+ ions for high fluences at 900° C. The phenomenon of He^+ ion induced fuzz formation was observed in most of the irradiation cases. Several attempts were made to understand such fuzz formation by varying several irradiation parameters, viz. fluence, flux, and energy of incidence He^+ ions. The role of He^+ ion irradiation fluence was studied by varying the irradiation time duration for each sample. Surface pinholes and nano-tendrils structure (fuzz) appearance, at a very early stage, were observed. By increasing He^+ ion fluence, these surface features evolved progressively to form what it appears to be an expanded surface holes and taller nano-tendrils before its further evolution to a W fuzz structure. Our study shows that W fuzz growth rate depends strongly on ion flux. This finding was confirmed in another irradiation experiments, where W samples were irradiated for the same ion fluence but with different ion fluxes. In these experiments, the W fuzz found to grow faster at higher He ion fluxes. Understanding various mechanisms responsible for surface morphology changes as well as possible mitigation techniques are very important for the reliability and performance of plasma-facing components in future fusion devices.

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Fig. 1. SEM images of nanostructure formed in the samples dark regions, all samples were bombarded by 300 eV of He ions (flux = 5.5×10^{20} ions $\text{m}^{-2} \text{s}^{-1}$ at the sample center) for different irradiation fluence per each sample, the SEM images taken at angle of view of 52° . The irradiation fluences that had been received for each sample are: 2.3×10^{24} ions m^{-2} (a), 5.5×10^{24} ions m^{-2} (b), 9.2×10^{24} ions m^{-2} (c), and 1.6×10^{25} ions m^{-2} (d)

Fig. 2. SEM images at various angles of views showing the main differences in a nanostructure appearance. , the sample was bombarded by 300 eV of He ions (flux = 5.5×10^{20} ions m⁻² s⁻¹) for a fluence of 2.3×10^{24} ions m⁻². The SEM images were taken at 52° (a), and at 0° (top view) (b).

Fig. 3. SEM images of nanostructure formed in the samples dark regions (fuzzy structured regions) all the samples were bombarded by 300 eV of He ions (flux = 5.5×10^{20} ions $\text{m}^{-2} \text{s}^{-1}$) for different irradiation fluence per each sample, the SEM images taken at angle of view of 0° . The irradiation fluences that had been received by each sample are: 2.3×10^{24} ions m^{-2} (a), 5.5×10^{24} ions m^{-2} (b), 9.2×10^{24} ions m^{-2} (c), and 1.6×10^{25} ions m^{-2} (d)

Fig. 4. XPS full spectrum (a), and high resolution XPS spectrum for the W 4f region (b).

Fig. 5. SEM images of nanostructure formed in the samples. The samples were bombarded by 300 eV of He ions (fluence = 1×10^{25} ions $\text{m}^{-2} \text{s}^{-1}$) at a samples temperature of 900° C. The ion beam flux was varied for each sample as well as the irradiation times. The ion beam fluxes that was selected for each sample are: 2.0×10^{20} ions $\text{m}^{-2} \text{s}^{-1}$ (a), 3.2×10^{20} ions $\text{m}^{-2} \text{s}^{-1}$ (b), and 5.5×10^{20} ions $\text{m}^{-2} \text{s}^{-1}$ (c)

Fig. 6. SEM images of nanostructure formed in the samples. The samples were bombarded by He ions (fluence = 1×10^{25} ions m^{-2} s^{-1} and flux = 5.5×10^{20} ions m^{-2} s^{-1}). Samples temperature during irradiation was fixed at 900° C. He⁺ ion incident energy was varied for each sample by applying positive bias voltage. He⁺ ion incident energies that were selected are: 140 eV (a), 200 eV (b) and 300 eV (c). The SEM images were taken at 52° sample tilt.