Temperature-Dependent Surface Modification of Ta due to High-Flux, Low-Energy He⁺ Ion Irradiation

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Abstract:

This work examines the response of Tantalum (Ta) as a potential candidate for plasma-facing components (PFCs) in future nuclear fusion reactors. Tantalum samples were exposed to high-flux, low-energy He⁺ ion irradiation at different temperatures in the range of 823 to 1223 K. The samples were irradiated at normal incidence with 100 eV He⁺ ions at constant flux of 1.2×10^{21} ions m⁻² s⁻¹ to a total fluence of 4.3×10^{24} ions m⁻². An additional Ta sample was also irradiated at 1023 K using a higher ion fluence of 1.7×10^{25} ions m⁻² (at the same flux of 1.2×10^{21} ions m⁻² s⁻¹), to confirm the possibility of fuzz formation at higher fluence. This higher fluence was chosen to roughly correspond to the lower fluence threshold of fuzz formation in Tungsten (W). Surface morphology was characterized with a combination of field-emission scanning electron microscopy (FE-SEM) and atomic force microscopy (AFM). These results demonstrate that the main mode of surface damage is pinholes with an average size of ~ 70 nm² for all temperatures. However, significantly larger pinholes are observed at elevated temperatures (1123 and 1223 K) resulting from the agglomeration of smaller pinholes. *Ex situ* X-ray photoelectron spectroscopy (XPS) provides information about the oxidation characteristics of irradiated

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surfaces, showing minimal exfoliation of the irradiated Ta surface. Additionally, [optical reflectivity](#) measurements are performed to further characterize radiation damage on Ta samples, showing gradual reductions in the [optical reflectivity](#) as a function of temperature.

Keywords: Plasma facing materials, Tantalum, Fuzz formation, ion irradiation, Atomic force microscopy, X-ray photoelectron spectroscopy, optical reflectivity

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I. Introduction:

Over the past several decades, there has been an increasing interest in developing and testing a wide range of materials for use as plasma facing components (PFCs) in nuclear fusion reactors. In particular, high-Z materials (namely, refractory metals) have been strongly considered due to their high resistance to sputtering, good thermal conductivity, and moderately low tritium uptake[1]. While Tungsten (W) is currently considered the primary material for the International Thermonuclear Experimental Reactor (ITER) [2], material choices for [future tokamaks](#) and other fusion devices [remain](#) open-ended [problems](#). For W, however, under substantial heat loads and He^+ ion particle fluence the surface may grow fine nano-tendrils commonly referred to as “fuzz” [3,4]. It is still unknown whether the creation of fuzzy structures on W surfaces will ultimately be detrimental to the fusion plasma since these fuzzy surfaces could exhibit larger mass losses due to pulsed plasma irradiation [5], but also may exhibit substantially lower physical sputtering [6]. If the operating conditions of future fusion devices are determined to be conducive to the growth of W fuzzy structures, it may be advantageous to consider other refractory metals as a substitute. While W is currently considered to be

the leading material for PFC's, recent studies [7] demonstrate that most other refractory metals may be almost as effective in withstanding fusion plasmas. Despite many of the bulk properties (such as the melting temperature, thermal conductivity, and density) of the refractory metals being well known, it is not known how these materials will respond to high fluences of H isotopes and He ions expected from fusion reactor operation. The present study aims to elucidate the effects of high-fluence, low-energy He^+ ion irradiation on Ta to investigate the performance of alternate refractory metals in extreme, fusion-relevant environments. The present study suggests that Ta may not form fuzzy structures under similar He^+ ion fluences and temperature ranges for which W fuzz is observed.

Robust PFC materials also need to be made of materials that are economically viable. While tungsten has a higher melting temperature and better thermal conductivity than Ta, it is also far less ductile at room temperature [8]. Traditional fabrication of tungsten or tungsten-coated PFCs may be an expensive option compared to Ta components [9]. However, it should be mentioned that recent advancements in W-component fabrication technology may render W to be a more cost-effective option. Additionally, Ta occludes hydrogen exothermically whereas W does so endothermically [1] suggesting that the hydrogen retention properties in these two materials will be different. However, further investigations into thermal desorption mechanisms of Ta and W need to be performed to resolve the complex behavior of temperature-dependent hydrogen release. If Ta is used as PFCs in future fusion devices, it is important to understand how it responds to simultaneous H isotopes and He^+ irradiation with high heat loads. While some reports [10,11] have previously suggested the use of Ta or Ta coatings

as PFCs, there has not yet been any study on the effect of low-energy He^+ irradiation on Ta surfaces.

The purpose of the present study is twofold. First, we aim to demonstrate the response of Ta surfaces to low-energy He^+ irradiation to replicate how the material might respond in the extreme environment of a nuclear fusion reactor and determine how the characteristics of the observed radiation damage may affect the integrity of the material. Second, the data collected here will aid in future modeling efforts to elucidate physical driving mechanisms involved in micro- and nanostructure evolutions. By characterizing the radiation damage observed in Ta, and comparing it to the damage observed in W and other refractory metals under similar conditions, we may empirically infer trends between material characteristics (i.e., melting temperature, ductility, thermal conductivity, etc.) and the resulting microstructural features.

II. Experimental Conditions

Annealed and rolled Ta samples with a thickness of 0.5 mm and apurity of 99.95% were cut from a large foil into 10×10 mm samples and mechanically polished to a mirror-like finish. The observed average surface roughness using atomic force microscopy (AFM) was ~ 2 nm, indicating for a reasonably smooth surface. He^+ ion irradiation experiments were performed in ultra-high vacuum (UHV) environment (base pressure of $\sim 10^{-7}$ Torr) with simultaneous sample heating via sample holder with resistive heating element. The heating element is equipped with a thermocouple feedback mechanism. The 100 eV He^+ ions were produced by a gridless end-hall type broad-beam ion source with a constant

flux of 1.2×10^{21} ions $\text{m}^{-2} \text{s}^{-1}$ at operating pressure of 2.0×10^{-4} Torr. The duration of irradiation for all Ta samples was constant (one hour), corresponding to a total fluence of 4.3×10^{24} ions m^{-2} . Samples were irradiated at several temperatures, ranging from 823 to 1223 K to demonstrate the effect of temperature on the Ta surface microstructure during He^+ ion irradiation. In addition, one extra sample has been irradiated to a total fluence of 1.7×10^{25} ions m^{-2} (using same ion flux of 1.2×10^{21} ions $\text{m}^{-2} \text{s}^{-1}$) at 1023 K target temperature. This additional experiment was performed to determine the response of Ta surface under an ion fluence where W fuzz is known to occur [12]. The irradiated Ta surfaces were analyzed by an assortment of *ex situ* characterization techniques. Field emission scanning electron microscopy (FE-SEM) was employed to record microstructural changes due to ion irradiation. High-resolution AFM in tapping-mode was performed to resolve the generated nanostructures and to measure the aspect ratio of surface features. Furthermore, high-resolution X-ray photoelectron spectroscopy (XPS) with an Mg-K_α source (1253.6 eV energy) was utilized not only to confirm the absence of surface impurities that may have been introduced in the irradiation process, but also to measure the extent of surface oxidation in post-irradiated samples. XPS data was fitted and analyzed using commercial CasaXPS software [13]. In addition, specular optical reflectivity measurements of the samples were performed to determine optical characteristics of the Ta surface and further characterize He^+ irradiation damage. Optical reflectivity measurements were performed over a spectrum of incident light (using a combination of halogen and deuterium light and a beam diameter of ~ 1 mm) ranging from 200 to 1120 nm wavelengths.

III. Results and Discussion:

III. (A) Scanning Electron- (SEM) and Atomic Force (AFM) –Microscopy Studies:

Figure 1 depicts FE-SEM surface micrographs of virgin and irradiated (via 100 eV He⁺ ion having a constant fluence of 4.3×10^{24} ions m⁻², i.e. 1.2×10^{21} ions m⁻² s⁻¹ flux for 1 hour duration) Ta samples as a function of target temperature in the range of 923 to 1223 K. All ion irradiated micrographs (Figs. 1(b)-(e)) are referenced against Figure 1(a), showing SEM micrograph of the virgin, mirror-polished Ta sample. Scanning electron microscopy on the virgin sample demonstrates a reasonably flat and defect-free surface. As evidenced in Fig. 1, He⁺ ion irradiation modifies the Ta surface significantly even at 923K target temperature. As a result, the appearances of homogeneously populated pinholes of 15 – 200 nm² in surface area were seen. Note that two types of pores, “small” and “large” were observed during the SEM studies. “Small” pores are defined as the pores having a total exposed surface area ≤ 150 nm². This cut off value (150 nm²) of pore surface area was chosen because pores larger than 150 nm² start losing their circularity (Fig. 1). A sequential reduction in the pore density of smaller pores, i.e., 651 (923K) to 576 (1023K) to 355 (1123K) to 116 pores μm^{-2} (1223K) was observed. On the other hand, a simultaneous sequential enhancement in the large pore density, i.e., 6.1 (923K) to 8.2 (1023K) to 61.2 (1123K) to 73.5 pores μm^{-2} (1223K) was also observed (Fig 1). A summary of both small and large pore density variation as a function of target temperature is shown in Figure 1(f). This data suggests that, at higher temperatures, a pronounced agglomeration of smaller pores results in observed pore percolations.

Figure 2 depicts high-resolution FE-SEM micrographs of 100 eV He⁺ ion irradiated Ta samples at 1023, 1123, and 1223K target temperatures. Most notably, the agglomeration

of smaller pores can clearly be seen in the 1223K sample (Fig. 2(e) and (f)), as evidenced by the appearance of small pores within the bounds of larger pores. Overall, the surface deterioration mechanism (as a function of target temperature) seems to be similar for all irradiated Ta samples. This observation is interesting as compared to the other refractory high-Z metals such as W [14] and Mo [15-17] where clear temperature windows and lower ion fluence thresholds for nanoscopic filaments and/or fibers, commonly known as fuzz growth, were observed at similar fluxes and fluences of low-energy He^+ ion radiation. Therefore, it seems that under high-flux, low-energy He^+ irradiation conditions studied in this work, Ta is free from the formation of fine nano-tendrils structures that are observed in Mo irradiated under similar conditions [15-16]. However, since fuzz formation in W may not be fully realized until higher He^+ ion fluences [12], an additional He^+ ion irradiation of Ta was also performed to a total fluence of 1.7×10^{25} ions m^{-2} (at the same flux of 1.2×10^{21} ions $\text{m}^{-2} \text{s}^{-1}$). The resulting surface morphology is shown in Figure 3. Irradiation at this higher fluence produces features that may resemble early-stage formation of nanotendrils (fuzz) on Ta surface, but the features are overall relatively less pronounced than those observed in W [12,14]. While the thermal conductivity of bulk Ta is initially lower than the other two materials, the relatively less pronounced appearance and lower aspect ratio of these fine nanostructures may suggest that this property is less likely to degrade in Ta under large particle fluxes. However, further investigation using even higher He^+ ion fluences is required to fully assess the behavior of Ta in extreme reactor environments.

In the case of W, these fuzzy nanostructures exhibit reduced shear strength [18] and demonstrate significant mass loss under pulsed power deposition [5]. The degradation of

these properties should be less substantial for Ta due to the lack of high aspect ratio nano-structuring. However this still needs to be confirmed with mechanical testing, modeling, and pulsed power deposition experiments. The pore surface area distribution histograms as function of target temperature (for Ta samples irradiated to a fluence of 4.3×10^{24} ions m^{-2}) have been shown in Fig. 4. The surface area calculations were performed on a number of the highest-fidelity images to generate these histograms. Note that the most common pinhole size ($\sim 70 \text{ nm}^2$) remains the same for the samples irradiated at 923 to 1123K, and only increases slightly for 1223K target temperature. This demonstrates that $\sim 70 \text{ nm}^2$ pinholes are the dominant damage mechanism.

Figure 5 shows the surface porosity (i.e., percentage of open pore surface areas) behavior of the Ta as function of target temperature for Ta irradiated to a fluence of 4.3×10^{24} ions m^{-2} . The surface porosity was calculated by adding up the total area of all pores in each individual micrograph, and then dividing it by the total surface area imaged in the corresponding micrograph. This figure demonstrates that the surface porosity increases exponentially with target temperature. This can be understood in the realm of the mobility of trapped He clusters under the Ta surface. These He clusters will increase with increasing target temperature, leading to a higher probability of He cluster agglomeration. However, observing the relatively small difference in surface porosity between 923 (surface porosity = 0.036) and 1223K (surface porosity = 0.041) suggests that the desorption of He from the surface is roughly the same order of magnitude for all target temperatures. This corroborates with the fact that all samples are irradiated at the same total fluence (the only variant parameter was the sample temperature), and further

demonstrates that the larger radiation-induced surface structures result from enhanced mobility and agglomeration of He clusters. To further elucidate the physical mechanisms responsible for the observed deterioration in irradiated Ta samples, tapping-mode AFM was employed to generate high-resolution images of the ion irradiation damaged Ta surface. Figure 6 depicts AFM micrographs of He⁺ ion irradiated Ta samples at 923 ((a)-(c)) and 1223 K ((d)-(f)) target temperature. Figure 6(a) shows the smaller pore and/or pinhole having an average diameter of ~20 nm (line profile in Fig. 6(b)). The pore size increases significantly for 1223K (Fig. 6(d)) as compared to 923K. In addition to pore diameter, pore depth also increases significantly with target temperature viz. 7 nm (923K) to 35 nm (1223K).

III. (B)XPS Results

To determine the chemical composition of the irradiated surfaces, *ex-situ* core-level XPS has been performed on the irradiated samples. The obtained XPS spectra from these samples were not only compared to against each other to understand how sample temperature during irradiation plays a role in chemical composition, but the spectra were also compared against a virgin sample (both before and after a sputter cleaning process) to compare the chemical composition to pristine and oxidized Ta surfaces. The sputter-cleaned sample was prepared via 1 keV Ar⁺ ion sputtering to remove the initial oxide layer. Figure 7 shows the survey spectra of selective irradiated samples and the virgin sample. Since every sample (with the exception of the sputter-cleaned sample) was exposed to atmospheric conditions for some time, it is reasonable to expect the appearance of both oxygen and carbon peaks in these spectra. However, the spectra are

free from any other peaks, confirming the absence of any other surface impurities. Figure 8 shows the high-resolution region spectra surrounding the 4f doublet peak observed in Ta. Each individual spectrum is fitted with components demonstrating a separation between the pristine Ta 4f doublet peaks and the 4f doublet peaks resulting from oxidized Ta in the form of Ta₂O₅. These respective components resulted from a deconvolution of the XPS data by a mixed Gaussian-Lorentzian (GL, m=30, where m=0 is pure Gaussian and m=100 is pure Lorentzian) line-shape using commercial CasaXPS analysis software [13]. From these region spectra, it is seen that there is some slight deviation in the extent of oxidation between all irradiated samples. The total amount of oxidation for all irradiated samples are marginally higher than the virgin (not sputter cleaned) sample with total concentrations of oxidized vs. pristine Ta in the range of 74 – 82%, as summarized in Table 1. In Fig. 8(e), component fitting of the sputter-cleaned Ta sample reveals that a small additional peak is present next to the pristine Ta 4f peaks. This peak is attributed to some mixing (with oxygen or possibly other surface contaminations) from the 1keV Ar⁺ beam. Table 2 shows the position and full-width half-maximum (FWHM) for each individual fitted component for all samples. It should be noted, that the binding energies at which the pristine Ta 4f peaks appear are slightly lower (by less than 1 eV) than those found in the oxidized samples. Some variation in the FWHM of Ta-oxide peaks indicates the possibility of additional Ta-oxide states (minor). Note that the extent of oxidation on the sample surfaces measured by XPS can give some indication of how pronounced the surface nanostructures are. A very rough and structured surface would exhibit more surface area, effectively creating more sites for oxidation. The only slight deviation in oxidized concentration between the virgin (not sputter-cleaned) sample and the irradiated

sample serves to confirm relatively small amounts of surface roughening. This result attests to the structural stability (small aspect ratio nanostructuring) of Ta under He^+ ion irradiation.

III. (C) Optical Reflectivity Results

Optical reflectivity was performed on selected irradiated Ta samples and a virgin, mirror-polished Ta sample to demonstrate the effect of surface morphology changes on the material's optical properties. Figure 9 shows the relative reflectivity of samples irradiated at 823, 1023, and 1223 K (fluence of $4.3 \times 10^{24} \text{ ions m}^{-2}$) over a spectrum of light (using a combination of halogen and deuterium light and a beam diameter of $\sim 1 \text{ mm}$) ranging from 200 – 1120 nm. Looking at the overall trend of these spectra, it is clear that an increase in temperature during irradiation leads to a decrease in reflectivity from the initial value of $\sim 30\text{-}40\%$ as seen from the virgin Ta spectra. The initial reflectivity of the virgin sample is consistent with other mirror-polished virgin Ta samples in other experiments involving Ta optical reflectivity measurements [19]. The overall temperature-dependent trend is consistent with the extent of radiation damage in the samples observed with FE-SEM (more surface damage implies lower reflectivity). At the highest temperature (1223 K), the He^+ irradiated Ta sample demonstrated optical-reflectivity $\sim 10\%$ for $\lambda = 800 \text{ nm}$. Although the reflectivity is substantially reduced from the virgin sample, it is still much higher than the very low reflectivity of low-energy, high-flux He^+ ion irradiated fuzzy W surfaces [20]. This data suggests that the irradiated Ta surface may be less coarsened or “fuzzy” than irradiated W surfaces in similar conditions.

Conclusions:

Low energy of 100 eV He^+ ion irradiation on Ta samples was performed. The Ta samples were irradiated at a constant flux of 1.2×10^{21} ions $\text{m}^{-2} \text{s}^{-1}$ over one hour duration to give a total fluence of 4.3×10^{24} ions m^{-2} . Sample temperature was varied from 823 to 1223K to investigate the effect of sample temperature on the resulting surface morphology of irradiated samples. An additional He^+ ion irradiation using an ion fluence of 1.7×10^{25} ions m^{-2} at 1023 K was also performed, suggesting the possibility of fuzz formation at higher fluence (in the range of the lower fluence threshold for W). Through a combination of FE-SEM and AFM, the surface morphology was characterized revealing small pinhole structures in the material surface. While the average size of surface pinholes increased with increasing sample temperature, the most common pinhole size remained approximately the same ($\sim 70 \text{ nm}^2$). This suggests that the larger pinholes are forming as a result of agglomeration of smaller pores as the temperature increases. High-resolution XPS spectra reveal little deviation in percent oxidation from a virgin, mirror-polished sample. This data suggests that the observed structures have a relatively low aspect ratio (confirmed by AFM). Optical reflectivity measurements show a decrease in total reflectivity with increasing sample temperature during irradiation. Notably, there was no observed nano-tendrill structure (fuzz) formation in the entire range of temperatures used in this study (823 to 1223K) for the lower fluence (4.3×10^{24} ions m^{-2}), which may give Ta an advantage over W and Mo as potential candidate for PFC in future fusion devices since these fuzzy structures may compromise the integrity of the material and contribute to plasma contamination. However, the Ta sample irradiated at a higher fluence shows

what may resemble the early stages of fuzz formation; though the fuzz formation features are relatively less pronounced than those observed in W [12,14]. Further investigations using even higher He^+ ion fluences are required to determine the threshold fluence (if it exists) for fuzz formation in Ta.

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Table 1: Atomic concentrations of the individual components of the region spectra for each component.

	Atomic Concentration (%)				Totals	
Sample	Ta 4f 7/2	Ta 4f 5/2	Ta₂O₅ 4f 7/2	Ta₂O₅ 4f 5/2	Ta	Ta₂O₅
Virgin, Sputter-cleaned	34.97	65.03	0	0	100	0
Virgin	2.99	16.32	37.1	43.59	19.31	80.69
823 K	6.26	19.75	34.36	39.69	26.01	74.05
923 K	4.81	14.77	35.87	44.55	19.58	80.42
1023 K	4.61	14.58	35.93	44.88	19.19	80.81
1123 K	4.43	14.11	37.29	44.17	18.54	81.46
1223 K	5.46	16.4	33.61	44.53	21.86	78.14

Table 2: Summary of the binding energies and full-width half-maximum (FWHM) values for the fitted components of the XPS region spectra.

Sample	BE (eV)	FWHM (eV)	BE (eV)	FWHM (eV)	BE (eV)	FWHM (eV)	BE (eV)	FWHM (eV)
Virgin, Sputter- cleaned	22.14	1.47	23.98	2.22				
Virgin	22.61	1.44	24.56	2.95	27.43	1.74	29.31	2.15
823 K	22.21	1.36	24.11	2.55	27.05	1.77	28.93	2.16
923 K	22.29	1.38	24.19	2.50	26.98	1.72	28.85	2.18
1023 K	22.24	1.37	24.13	2.52	26.94	1.70	28.81	2.17
1123 K	22.2	1.34	24.11	2.52	27.08	1.73	28.94	2.14
1223 K	22.23	1.31	24.1	2.48	26.73	1.68	28.6	2.22

Figure 1: (a) Virgin polished Ta surface. (b)-(e) Ta irradiated with 100 eV He⁺ ions at 923, 1023, 1123, and 1223 K, respectively. (f) Areal density of small and large pores for each micrograph.

Figure 2: High-resolution SEM micrographs for Ta samples irradiated at 923 (a)-(b), 1123 (c)-(d), and 1223 K (e)-(f), respectively.

Figure 3: FE-SEM micrograph of a Ta surface irradiated at 1023 K to a total fluence of 1.7×10^{25} ions m^{-2} .

Figure 4: Normalized histograms of measured pore areas for Ta samples irradiated at 923, 1023, 1123, and 1223 K, respectively.

Figure 5: Surface porosity of irradiated Ta as a function of sample temperature. Porosity here is defined as the total (summed) area of all pores in an SEM micrograph, divided by the total imaged area of the micrograph.

Figure 6: High resolution atomic force microscopy (AFM) imaging of 100 eV He^+ ion irradiation, having a fluence of 4.3×10^{24} ions m^{-2} , induced surface modification in mirror finished polished Ta surfaces at 923 ((a)-(c)) and 1223 K ((d)-(f)) target temperatures. Line profiles of pinholes and/or pores (showing depth and diameter) from the marked circular regions are shown in figures (b) (923K) and (e) (1223K). All the AFM images in three dimension (3D) are in the scan area of $500 \times 500 \text{ nm}^2$.

Figure 7: *Ex situ* XPS survey spectra of Ta samples irradiated at several temperatures, as well as a virgin Ta sample and a sputter-cleaned (*in situ*) Ta sample for reference.

Figure 8: *Ex-situ* XPS region spectra for irradiated and virgin Ta samples. (a) – (c) depict the survey spectra for samples irradiated at 823, 1023, and 1223 K, respectively. (d) shows the region spectrum for a polished, virgin Ta sample and (e) shows the region spectrum for a sputter-cleaned Ta sample (*in-situ*).

Figure 9: Optical-reflectivity measurements of various irradiated Ta samples relative to a virgin, mirror-polished Ta sample.