Temperature-Dependent Surface Porosity of Nb₂O₅ under High-Flux, Low-Energy He⁺ Ion Irradiation

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Abstract

The present study reports on high-flux, low-energy He⁺ ion irradiation as a novel method of enhancing the surface porosity and surface area of naturally oxidized niobium (Nb). Our study shows that ion-irradiation-induced Nb surface micro- and nano-structures are highly tunable by varying the target temperature during ion bombardment. Mirror-polished Nb samples were irradiated with 100 eV He⁺ ions at a flux of 1.2×10^{21} ions m⁻² s⁻¹ to a total fluence of 4.3×10^{24} ions m⁻² with simultaneous sample annealing in the temperature range of 773-1223 K to demonstrate the influence of sample temperature on the resulting Nb surface morphology. This surface morphology was primarily characterized using field-emission scanning electron microscopy (FE-SEM) and atomic force microscopy (AFM). Below 923 K, Nb surfaces form nano-scale tendrils and exhibit significant increases in surface porosity. Above 923 K, homogeneously populated nano-pores with an average diameter of ~60 nm are observed in addition to a smaller population of sub-micron sized pores (up to ~230 nm in diameter). Our analysis shows a significant reduction in surface pore number density and surface porosity with increasing sample temperature. High-resolution *ex-situ* X-ray photoelectron spectroscopy (XPS) shows Nb₂O₅ phase in all of the ion-irradiated samples. To further demonstrate the length scales in which radiation-induced surface roughening occurs,

optical reflectivity was performed over a spectrum of light between 200-1100 nm, showing a recovery of nano-scale surface damage at high sample temperatures.

I. Introduction

Over the past several years niobium (Nb) oxide micro- and nano-structures, particularly Nb₂O₅ structures, have been studied and tested for a wide range of applications including solar energy conversion [1-3], chemical catalysts [4, 5], supercapacitors [6], and bio-applications [7, 8]. While this is certainly a wide range of applications, every study placed a common emphasis on enhancing Nb₂O₅ surface area in a controlled manner. Particularly for bio-applications, it is important to be able to control both the surface porosity and aspect ratio of metal-oxide nanostructures [9-11]. Recently, several efforts have been made by research groups all around the world to develop highly porous Nb₂O₅ surfaces using several techniques such as electrosynthesis and anodization [12-14], phase transformation [15], thermal oxidation [16], and sol-gel processes [17,18]. In this study, we present a novel one-step ion irradiation processing technique for generating similar nanostructured Nb₂O₅ surfaces with relatively large surface area and surface porosity. This top-down approach guarantees good contact between the different crystallites and avoids electrical conductivity limitations (if required). [Similar He⁺ plasma-processing techniques have been used to produce nanostructures on oxidized W surfaces \[19\] and Fe surfaces \[20\] for solar water splitting applications. While W is the most widely studied material in this field \(due to the relevance of He⁺ ion irradiation damage to fusion applications\), high-flux, low-energy He⁺ ion irradiation has been known to produce various types of nanostructures in other materials such as nano-pillars and pinholes in Ti](#)

[21-22], nano-tendrils in Mo [23-24], voids and pinholes in Ta [25], and other void and nanostructure formations in Cu and Al [22].

We present He^+ ion irradiation as a novel processing technique; however, the effects of He^+ ion irradiation on Nb surfaces have previously been studied in the context of nuclear structural materials to assess Nb as a potential plasma-facing component (PFC) for nuclear fusion reactors [26-30]. In these studies, He bubble formation [26-28], surface blistering [29], and tendril-like structures [30] are observed as a result of He^+ ion induced surface modification. However, almost all of these studies use relatively higher He^+ ion energies (up to several keV) where sputtering also plays an important role. Furthermore, these studies are also primarily concerned with the reduction of Nb surface damage under high-flux ion irradiation instead of trying to optimize and finely tune the surface porosity and surface area. While it is understood that He^+ ion irradiation may cause certain morphological changes in Nb surfaces, none of these studies assessed the modified material's surface chemical composition or oxidation characteristics to demonstrate its relevance to applications other than nuclear structural materials. The present study will demonstrate that the generation of Nb_2O_5 micro- and nano-structures are possible with a low-energy (100 eV) He^+ irradiation process and the resulting feature sizes are tunable by tailoring the Nb temperature during ion irradiation. This may open a new pathway for solar power concentration technology, chemical catalysts, and bio-applications. In addition, surface analysis of irradiated Nb surfaces may provide some further understanding of nanostructure formation on high-Z refractory metals for nuclear applications.

II. Experimental Methods

The experiments were performed at the UHFI (ultra-high flux irradiation) and IMPACT (interaction of materials with particles and components testing) surface characterization laboratories [23-25, 31] at the center for materials under extreme environment (CMUXE), Purdue University. 0.5 mm-thick Nb foils (99.8% purity, annealed) were cut into 10×10 mm samples and mechanically polished to a mirror-like finish. Ion irradiation experiments were performed using 100 eV He⁺ ions generated from the EH-400LE, a broad-beam End-Hall (EH) gridless ion source manufactured by Kaufman & Robinson, Inc. The Nb samples were irradiated with a constant flux of 1.2×10^{21} ions m⁻² s⁻¹ for one-hour duration, leading to a total He⁺ fluence of 4.3×10^{24} ions m⁻². All irradiation experiments were performed inside an ultra-high vacuum (UHV) chamber with a base pressure of $\sim 10^{-7}$ Torr and a working pressure of 2.0×10^{-4} Torr. During irradiation, the Nb samples were simultaneously annealed at a constant temperature throughout each experiment at a temperature in the range 773-1223 K. For a precise temperature measurement, thermocouples were used instead of radiation thermometers; a thermal-feedback mechanism in the sample heater corrected for ion-induced heating of the Nb samples during the experiment by altering the current supplied to the heating element based on the difference between current and desired sample temperatures. Post-irradiation sample surface morphology was characterized *ex-situ* by field emission scanning electron microscopy (FE-SEM). Atomic force microscopy (AFM) studies using Bruker Innova atomic force microscope were also performed on selected samples to resolve finer details of the ion-induced nanoscopic changes. To characterize changes in surface oxidation and chemical composition, X-ray photoelectron spectroscopy (XPS)

was performed on a virgin and all post-irradiated Nb samples. X-rays for XPS were generated with an Mg-K α source and electrons were detected using an Omicron Argus hemispherical analyzer with round aperture of 6.3 mm. No sample charging was observed. All XPS spectra were analyzed using commercial CasaXPS software [32]. XPS spectra were also taken from a virgin, mirror-polished sample both before and after a sputter-cleaning process to compare XPS peaks from the He⁺ ion-irradiated samples to both pure and naturally oxidized Nb. The sputter-cleaning process consisted of irradiating (*in-situ*) the Nb sample in a UHV environment with 1 keV Ar⁺, at a flux of $\sim 10^{21}$ ions m⁻² s⁻¹, for several minutes to remove any native Nb-oxide layers. Additionally, optical reflectivity measurements were taken for post-irradiated Nb samples to further characterize surface alterations and assess the material's relevance to solar applications. These optical reflectivity measurements were performed using a PerkinElmer 950 spectrometer with an integrating sphere of 1100 nm [33]. A combination of halogen and deuterium light was used to study a wide range of incident light wavelengths ranging from 200 to 1120 nm.

III. Results and Discussion

III. (A) Scanning electron microscopy (SEM) and atomic force microscopy (AFM) studies

Figure 1(a) depicts an FE-SEM micrograph of a virgin mirror-polished Nb surface showing relatively flat and defect-free Nb surface. Figures 1(b)-(g) show FE-SEM micrographs of Nb surfaces after He⁺ ion irradiation with a fluence of 4.3×10^{24} ions m⁻² as function of temperature in the range of 773-1223 K. As evidenced in figure 1(b),

irradiations at temperatures as low as 773 K significantly alter the Nb surface. This leads to the generation of homogeneously populated porous structures on the entire surface. Similar porous structures are also observed at 823 K. Note that these structures seem to resemble the early-stage formation of nano-scale tendrils or “fuzz” observed by our group in the case of Mo [23, 24] and by others in the case of W [34, 35], under similar He^+ irradiation conditions. The fuzzy structures are ultimately a result of sub-surface He clusters formed from the trapping of penetrating He^+ ion irradiation, which then form pressurized bubbles and erupt through the surface leaving behind dense tendril structures. In the case of Mo, fuzz is observed under similar irradiation conditions when the sample temperature is kept between 873 and 1073 K [23]. Beyond this upper temperature limit, we observe the appearance of pillar-like nanostructures. In W, this temperature window for fuzz formation is even higher (~1000-2000K) [36]. For the present case of Nb surfaces, however, we observe large separation between nano-tendrils at temperatures above 923 K due to the growth and collapse of surface pores. In fact, around 1023 K, the main mode of surface deterioration is the appearance of a significant amount of surface pores of various sizes (Figure 1(e)) instead of nano-scale tendrils or “fuzz” formation (Figure 1(b)-(d)). The average sizes of these pores increase with temperature, as evidenced in Figures 1(f) and (g). Figure 1(h) plots the surface pore number density vs temperature for the three highest target temperatures. The three lower temperatures (773, 823, and 923 K) were excluded from this analysis since the surface morphologies observed at those target temperatures were significantly different (nano-scale tendrils as opposed to surface pores). Note that while the average surface pore size increases with increasing temperature, the pore number density decreases. Detailed analysis of surface

pore size was performed on several of the highest fidelity FE-SEM micrographs to generate pore diameter histograms shown Figure 2. All histograms were fitted using deconvoluted Gaussian distributions to guide the eye and show existing correlations between surface pore sizes and sample temperature. For the two samples irradiated at a higher temperature (1123 and 1223 K), the histograms appear to have two separate distributions. For all three samples, there is a dominant distribution of smaller surface pores (~60 nm in diameter) as well as a second, larger distribution that has an increasing diameter with increasing temperature. The histograms reveal a sequential increase in the average surface pore diameter with target temperature for the second distribution from ~100 nm (1123 K) to ~150 nm (1223 K) while the first distribution (~60 nm) only changes slightly. At the highest temperature (1223 K), the largest observed pore size was ~230 nm in diameter. Simultaneous reduction in the surface pore number density along with an increase in average pore size suggests that the smaller pores may be agglomerating into larger surface pores. Higher temperatures during irradiation lead to enhanced mobility of He clusters trapped beneath the surface, effectively allowing for the formation of larger clusters that manifest themselves as larger pores upon surface reemission. The simultaneous exponential decrease in surface pore number density and increase in average pore size may also suggest that the growth of surface pores can be modeled with percolation theory [37], and the results presented here are reminiscent of previous ion irradiation experiments where similar percolation phenomena were identified [31]. Additionally, several high fidelity FE-SEM micrographs were used to measure the projected two-dimensional surface area of pores. For each sample, the total projected areas for all surface pores were added up and then divided by the total Nb

surface area used in analysis to calculate a “surface porosity.” The surface porosity gives some quantitative information about how porous the irradiated Nb surface is; a surface porosity of 0% would indicate a pore-free surface, whereas a surface porosity of 50% would mean that 50% of the area in an SEM image is populated by surface pores. This concept of surface porosity is not unlike a two-dimensional void fraction. Surface porosity results have been summarized in Figure 3. This figure clearly shows that the surface porosity decreases with increasing target temperature even though the average pore size increases.

While similar fuzzy and porous nano- and micro-structures are well documented in the nuclear fusion community for W, there is still some debate over the exact driving mechanism for such structural formation. A viscoelastic-based model shows that helium penetrating the W surface agglomerates into bubbles, which then are “pushed out” from the surface due to internal stresses [38]. The resulting structure is then formed by sub-surface accumulation of these He bubbles and a subsequent temperature-dependent creep and elastic deformation in the near-surface W. Previous modeling coupled with experimental results also evidences internal stress gradients forcing He bubbles to the surface, resulting in a highly porous surface due to high-flux, low-energy He^+ irradiation [39]. On the other hand, there are also theories that suggest fuzz formation in W may be caused by a collection of knocked-out adatoms [36] or by kinetic roughening from He bubble rupture [40]. However, most of the proposed models agree that the observed surface damage results from He^+ penetrating the sample surface, becoming trapped and agglomerating into pressurized sub-surface He bubbles, and then erupting through the outer sample surface, effectively roughening that surface. The damaged surface observed

in the Nb samples presented here may also result from similar processes; however, additional fluence-dependent studies will be needed to further elucidate an exact driving mechanism for structure formation.

To further characterize the fine details of Nb nanostructure formation, high-resolution AFM studies were also performed (Figures 4 and 5). Figure 4 clearly shows the presence of fine tendril-like (fuzzy) nanostructures even at 1023 K. The fact that these structures are not observed in the corresponding FE-SEM image is not surprising since the AFM has superior resolution. The figure also shows evidence of two different populations of surface pores; a large population of small (~60 nm) pores is scattered between a sparser population of large pores. An arrow in Figure 4(a) marks one of such smaller pores. The line profile of the same pore has been shown in the inset of Figure 4(a); the pore's diameter from this line profile is similar to those observed from FE-SEM results. Additionally, Figure 4(c) and (e) show 3D AFM contour plots to further resolve surface features. The corresponding line profiles of the highlighted features in Figures 4(c) and (e) are shown in Figures 4(b) and (d), respectively. Figure 4(c) shows the interspacing of two neighbor submicron size grains. On the other hand, Figure 4(e) shows the submicron size grain and one of the bigger-sized pores. Figure 5 shows similar AFM results for a sample irradiated at 1223 K. In contrast to the observations in Figure 4, Figure 5(a) shows complete depletion of the fine nano-scale tendril-like (fuzz) structure and grainy structure having larger pores, two typical pores and their line profiles are shown in the respective figures, Figure 5(b)-(e).

III. (B) X-ray Photoelectron Spectroscopy (XPS) Studies

Figure 6 shows the survey XPS spectra over binding energy values of 650 to 0 eV for several samples. The top three spectra correspond to Nb samples that were irradiated at constant target temperatures of 1223, 1023, and 823 K, respectively. These spectra are referenced against similar XPS spectra for a virgin, mirror-polished Nb sample (second from the bottom) and an Ar⁺ sputter-cleaned Nb sample (bottom). Each spectrum shows Nb 3s, 3d, and 4p XPS peaks. With the exception of the sputter-cleaned virgin sample, each sample also produces O1s and C1s XPS peaks. Appearance of O1s and C1s are expected since XPS studies were performed *ex-situ*. The bottom-most spectra corresponding to the virgin sample after the 1keV Ar⁺ ion sputter-cleaning process shows no XPS peaks from oxygen or carbon. This spectrum was then used to resolve the true position of the pure Nb XPS peaks. To determine the oxidation characteristics of the ion irradiated Nb samples compared to the virgin samples, high-resolution XPS spectra were taken over a range of binding energies from 215-200 eV. Figure 7 displays these high-resolution spectra for Nb 3d doublet peaks. All the spectra were deconvoluted into their respective components by a mixed Gaussian-Lorentzian (GL, m=30) line-shape (where m=0 is pure Gaussian and m=100 is pure Lorentzian shape) using commercial CasaXPS peak fitting software [32]. Nb 3d peaks have been resolved into four individual peaks: two corresponding to the virgin Nb 3d doublet and two corresponding to the oxidized Nb 3d doublet, recognized by a slight shift in this doublet peak to a higher binding energy. Table 1 displays the binding energy (BE) and FWHM for each of the four peaks (corresponding to Nb 3d doublets) in each sample. Note that there are only two peaks observed for the sputter-cleaned virgin Nb sample, since the naturally oxidized Nb layer has been sputtered off by 1keV Ar⁺ ion irradiation. The separation in binding energy

between the peak positions of virgin Nb 3d and oxidized Nb 3d doublets, ~5 eV, matches well with previously reported binding energy shifts observed for Nb₂O₅ [41]. Irradiated and virgin (not sputter-cleaned) Nb samples demonstrate a significantly larger area under the higher-energy XPS peaks, suggesting that the vast majority of Nb on these surfaces is in an oxidized state, namely Nb₂O₅. Table 2 summarizes the atomic concentrations for each doublet, giving the total percent oxidation for each sample. It should be noted here that the oxidation state of each irradiated sample is only marginally different from the virgin, naturally oxidized Nb sample.

III. (C) Optical Reflectivity Studies

To further characterize the resulting Nb surface nano- and micro-structures after high-flux, low-energy He⁺ ion irradiation, optical reflectivity measurements were performed. Figure 8 shows optical reflectivity spectra in the wavelength range of 200-1100 nm for irradiated and virgin mirror-polished samples. For a qualitative analysis of these reflectivity measurement results, two reflected light wavelengths, 200 and 1100 nm, will be considered. The selections of these two wavelengths are based on the minimum and maximum wavelength limits of these optical reflectivity measurements. At 1100 nm wavelength, there is an appreciable reduction in the reflectivity value with increasing target temperature. The reflectivity for the mirror-polished virgin sample at this wavelength was just over 27%. After the He⁺ ion irradiation to a total fluence of 4.3×10^{24} ions m⁻² at 823 K, the reflectivity value reduces to 16%. For the sample irradiated at the highest target temperature (1223K), the reflectivity value for 1100 nm wavelength reduces even more to only ~3%. These results indicate that, on micron scale,

the surface roughness increases with increasing target temperature and effectively reflects less light from the surface. At the lower light wavelength, 200 nm, the reflectivity begins to decrease with increasing sample temperature from 11% (virgin) to 2% (823 K) to 1.3% (923 and 1123 K). However, the reflectivity at this wavelength for the sample irradiated at highest target temperature (1223 K) improves somewhat to over 2%. This demonstrates that the highest-temperature (1223 K) sample has a surface with lower roughness than samples at a lower temperature at this length scale of a couple hundred nanometers. This indicates that the area between the oversized surface pores observed in Figure 1(g) is actually somewhat smoother than that of the other samples. These results are confirmed by the enhanced nano-scale smoothening observed by high-resolution AFM studies (Figure 6).

IV. Conclusions

The present study suggests a novel method of processing Nb₂O₅ using a high-flux, low-energy He⁺ irradiation to substantially increase surface porosity and surface area. The resulting micro- and nano-scale surface pores and tendril-like structures have tunable size, depending on the Nb target temperature during irradiation. Mirror-polished Nb surfaces were irradiated with 100 eV He⁺ having a flux of 1.2×10^{21} ions m⁻² s⁻¹ to a total fluence of 4.3×10^{24} ions m⁻² with simultaneous target annealing in the temperature range 773-1223 K. Tendril-like structures (fuzz) were observed for the target temperatures of 773-823 K; on the other hand, surface pores were observed for samples irradiated at 1023-1223 K. Microscopy studies indicate that the sample irradiated at 923 K demonstrates the transition between these tendril-like and pore structures. Average

surface pore diameter increases with increasing target temperature. At the highest target temperature studied (1223 K), surface pore diameter reaches up to 230 nm. However, the presence of two pore diameter distributions (determined by deconvoluting histogram data) may suggest that these larger pores are formed due to a percolation of the smaller pores (~60 nm in diameter). It was also observed that the surface porosity increases with decreasing target temperature (in the temperature range of 823-1223K). Compared to our previous report on Mo under similar conditions [23], Nb had a somewhat lower temperature window for “fuzz” formation (773-923 K, compared to the Mo temperature window of 873-1073 K). Above the upper temperature limit for fuzz formation, we observe quite different trends between the two materials. Nb shows a vast network of surface pores (diameter increasing with temperature) whereas Mo forms a nanostructured surface consisting of nano-pillars. While the discrepancy in behavior between these two materials is not yet clear, future work will look at combined experimental and modeling approaches to determine what material characteristics are responsible for these various surface structures. High-resolution XPS spectra for all Nb 3d doublet peaks show that the vast majority of the Nb surface, both before and after ion irradiation, is oxidized in Nb₂O₅ state. Optical reflectivity measurements show the degradation in the reflectivity of the Nb surface after irradiation. The reflectivity value corresponding to 1100 nm decreases with increasing target temperature. For samples irradiated at highest temperature (1223 K), the reflectivity value reduces up to ~3%. On the other hand, the reflectivity corresponding to the 200 nm wavelength also decreases with increasing target temperature, indicating a nano-scale surface roughening due to He⁺ irradiation. However, the reflectivity in this region recovers slightly for the samples irradiated at the

highest temperature case, most likely due to enhanced thermal annealing. Future studies will focus on the effect of He^+ ion energy and fluence on Nb and Nb_2O_5 microstructure evolution to further predict the optimum irradiation conditions relevant to various applications.

Acknowledgements

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Table 1: Summary of the binding energy (BE) and full-width half-maximum (FWHM) values for each fitted peak in the high-resolution XPS spectra.

Sample	Nb 3d _{5/2}		Nb 3d _{3/2}		Nb ₂ O ₅ 3d _{5/2}		Nb ₂ O ₅ 3d _{3/2}	
	BE (eV)	FWHM (eV)	BE (eV)	FWHM (eV)	BE (eV)	FWHM (eV)	BE (eV)	FWHM (eV)
Virgin, Sputter-cleaned	202.89	1.69	205.58	2.50	-	-	-	-
Virgin	203.44	1.90	206.06	2.41	208.33	1.92	210.78	2.13
823 K	202.79	1.65	205.30	1.82	207.71	1.89	210.44	2.14
1023 K	202.78	1.74	205.34	1.91	207.76	1.90	210.49	2.11
1223 K	202.99	1.72	205.61	2.12	208.06	1.91	210.78	2.13

Table 2: Summary of calculated atomic concentrations each peak in the high-resolution XPS spectra.

	Number of Counts (%)				Total Atomic Concentration (%)	
Sample	Nb 3d _{5/2}	Nb 3d _{3/2}	Nb ₂ O ₅ 3d _{5/2}	Nb ₂ O ₅ 3d _{3/2}	Nb	Nb ₂ O ₅
Virgin, Sputter- cleaned	48.63	51.37	0	0	100	0
Virgin	4.16	8.27	51.02	36.55	12.43	87.57
823 K	4.41	3.94	52.92	38.73	8.35	91.65
1023 K	5.99	5.19	51.57	37.25	11.18	88.82
1223 K	7.28	7.34	49.52	35.86	14.62	85.38

Figure 1: FE-SEM micrographs of (a) a virgin, polished Nb surface, (b)-(g) Nb surfaces irradiated with He⁺ to a fluence of 4.3×10^{24} ions m⁻² at a constant temperature of 773, 823, 923, 1023, 1123, and 1223, respectively, and (h) a plot of the pore number density vs. sample temperature for the samples irradiated at 1023, 1123, and 1223 K.

Figure 2: Histograms of pore diameters that were observed in Nb samples irradiated with He⁺ to a fluence of 4.3×10^{24} ions m⁻² at a constant temperature of 1023, 1123, and 1223 K, respectively.

Figure 3: The surface porosity (defined as the percent of Nb surface area populated by pore formation) vs. sample temperature for Nb samples irradiated with He^+ to a fluence of $4.3 \times 10^{24} \text{ ions m}^{-2}$ at constant temperature of 1023, 1123, and 1223 K, respectively.

Figure 4: (a) AFM micrograph of an Nb surface irradiated with 100 eV He⁺ to a fluence of 4.3×10^{24} ions m⁻² at 1023 K. The inset plot in (a) is a line profile of the surface pore marked by the black arrow. Fig. (c) and (e) represent 3D AFM images of the same sample showing separation between two neighbor submicron size grains and submicron size grains along with surface pores, respectively. Fig. (b) and (e) show the line profiles of the marked regions in the below 3D AFM images.

Figure 5: (a) AFM micrograph of an Nb surface irradiated with 100 eV He⁺ to a fluence of 4.3×10^{24} ions m⁻² at 1223 K. Fig. (c) and (e) represent 3D AFM images of the same sample showing two typical surface-pores on the Nb surface. Fig. (b) and (d) show the line profiles of the marked regions in the below 3D AFM images.

Figure 6: *Ex-situ* XPS survey spectra of selected Nb samples. The top three spectra correspond to Nb samples irradiated with He^+ to a fluence of 4.3×10^{24} ions m^{-2} at a constant temperature of 1223, 1023, and 823 K, respectively. The bottom two spectra correspond to a virgin, mirror-polished Nb sample both before and after 1 keV Ar^+ ion sputter-cleaning process to remove native contaminants and resolve the pure Nb XPS peaks.

Figure 7: High-resolution XPS region spectra of (a)-(c) Nb samples irradiated with He^+ to a fluence of $4.3 \times 10^{24} \text{ ions m}^{-2}$ at a constant temperature of 1223, 1023, and 823 K, respectively and similar XPS region spectra of a virgin, mirror-polished Nb sample both before (d) and after (e) a sputter-cleaning process to resolve the pure Nb XPS peaks.

Figure 8: Optical reflectivity measurements for selected Nb samples that were irradiated with He^+ ions to a fluence of 4.3×10^{24} at a constant temperature of 823, 923, 1123, and 1223 K, respectively. These spectra are referenced against a virgin, mirror-polished Nb sample to demonstrate the relative decrease in reflectivity of irradiated samples.