Ion Beam Irradiation-Induced Effects on the Microstructure and Mechanical Properties of Molybdenum

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Date: 2025-08-25T00:00:00+00:00

Abstract

Molybdenum is used as a plasma-facing component (PFC) in tokamaks where it is exposed to high-energy neutrons and plasma particles (He and H ions). This study examines the effects of high-energy copper ion beam with varying doses (0.108, 1.08, and 10.8 dpa) on the microstructure and mechanical properties of molybdenum samples, to emulate the impact of high-energy particle cascade damage. Following exposure to high-energy copper ion beams, changes in microstructure, crystal size, lattice constant, micro-strain (%), and surface morphology were observed. Lower ion dose (0.108 dpa) resulted in severe cracking as shown in SEM images, attributed to a high dislocation density as a result of increased micro-strain (%) and decreased crystal size. Features like dents, melt craters, and a rough surface were observed at high ion doses of 1.08 and 10.8 dpa.Initially, crystal size decreased (fragmentation) at low ion dose of 0.108 dpa. However, at higher ion doses of 1.08 and 10.8 dpa an increase in crystal size (growth) was observed and the microstrain was dropped to lower values. Decrease in lattice constant seems linear with the ion dose as clear from XRD results, indicating the progressive radiation damage. The change in hardness with increasing dose was nonlinear. Hardness was observed to increase first at lower ion dose (0.108 and 1.08 dpa), however at higher ion doses of 10.8 dpa, a decrease in hardness is observed due to crystal size coarsening and structure recovery/recrystallization effects.

Full Text

Preamble

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Abstract

Molybdenum is used as a plasma-facing component (PFC) in tokamaks where it is exposed to high-energy neutrons and plasma particles (He and H ions). This study examines the effects of high-energy copper ion beams with varying doses (0.108, 1.08, and 10.8 dpa) on the microstructure and mechanical properties of molybdenum samples to emulate the impact of high-energy particle cascade damage. Following exposure to high-energy copper ion beams, changes in microstructure, crystal size, lattice constant, micro-strain (%), and surface morphology were observed. Lower ion dose (0.108 dpa) resulted in severe cracking as shown in SEM images, attributed to high dislocation density resulting from increased micro-strain (%) and decreased crystal size. Features like dents, melt craters, and a rough surface were observed at high ion doses of 1.08 and 10.8 dpa. Initially, crystal size decreased (fragmentation) at the low ion dose of 0.108 dpa. However, at higher ion doses of 1.08 and 10.8 dpa, an increase in crystal size (growth) was observed and the micro-strain dropped to lower values. The decrease in lattice constant appears linear with ion dose as evident from XRD results, indicating progressive radiation damage. The change in hardness with increasing dose was nonlinear. Hardness increased first at lower ion doses (0.108 and 1.08 dpa), but at the higher ion dose of 10.8 dpa, a decrease in hardness was observed due to crystal size coarsening and structure recovery/recrystallization effects.

Keywords: radiation defects, radiation hardening, dislocation density, recrystallization, softening

Introduction

Nuclear fusion is envisaged as an attractive source of sustainable, green energy due to its zero-carbon emission, high energy density, and abundant fuel supply. During both transient events and steady-state operation, high-energy particles and heat loads in the range of MW/m² to GW/m² are expected to reach the first wall material of the tokamak. Plasma-facing components (PFCs) will be exposed to 14 MeV neutrons, which can cause more than 15 dpa damage over a year to tungsten and molybdenum PFCs. Due to these intense loads, cracking, melting/re-deposition, and swelling can occur, leading to severe damage. Although mitigation techniques are implemented to reduce heat loads to tolerable limits for plasma-facing components, heat fluxes reaching them can still be as high as ~1 MJ/m², causing severe damage.

Tungsten is considered the most reliable material for harsh environments due to its high melting point, low sputtering yield, high thermal conductivity, and low tritium retention. However, low-energy helium irradiation can cause fuzz formation on tungsten surfaces, leading to reduced thermal conductivity and degraded mechanical and structural properties. Additionally, bending, milling, and machining of tungsten are challenging because of its high hardness. Hydrogen isotopes escaping from the plasma core can penetrate irradiated tungsten structures and combine with dislocations, resulting in swelling and cracking of PFCs. On the other hand, molybdenum, being a refractory metal, has emerged as another attractive candidate for PFCs in tokamak devices and is also used in plasma optical diagnostic mirrors.

Molybdenum possesses the required properties to be used as an alternative to tungsten. Its high evaporation temperature helps prevent evaporated particles from entering the plasma and mitigates plasma cooling/quenching problems. It also prevents the retention of hydrogen atoms in the temperature range of 823-1073 K. Hydrogen retention in molybdenum is lower in a narrow operating temperature window compared with tungsten, which has a wider hydrogen retention temperature range (1000-2000 K). Molybdenum can also accommodate transient heat loads during disruption events like edge localized modes (ELMs) and vertical displacement events (VDEs).

Energetic particles produced by particle accelerators (like the Large Hadron Collider at CERN), ion implanters, and nuclear reactors interact with matter and induce four main effects: ionisation, nuclear transmutation, displacement damage, and phase transformation. These effects can alter surface morphology, microstructure, and degrade material properties. Among them, displacement damage, transmutation, and phase transformation effects are critical to material performance and must be thoroughly addressed before materials are deployed in nuclear reactors. Displacement damage causes lattice disorder in the crystal structure through ion-matter interaction. When high-energy particles interact with matter, they transfer energy that results in the formation of lattice defects. Part of the incoming ion's energy is also dissipated as phonons, electronic excitations, secondary electrons, photons, and heat, leading to localized remelting and recrystallization. Lattice defects are further classified as point defects (vacancies and interstitials), line defects (screw or edge dislocations), and plane defects (voids, pores, or cracks). On the surface, radiation damage can manifest as craters, adatoms, and ripples. Transmutation effects cause production of helium and hydrogen inside the structure, and coalescence of these gas atoms with vacancies results in bubble formation and swelling. Phase transformation can produce undesirable precipitates or dissolve desirable ones, causing deterioration of mechanical properties.

Irradiation damage is a critical issue in nuclear reactors. In early-stage irradiation damage, dislocations are produced through population multiplication and clustering. These defect clusters lead to void formation and degrade the mechanical properties of materials. When molybdenum is used as a plasma-facing component in tokamaks, it encounters high-energy neutrons, high heat flux, and hydrogen and helium ions. Neutron irradiation can degrade the molybdenum surface and affect the component's lifespan. Surface damage can limit the use of molybdenum as optical diagnostic mirrors and as PFCs in tokamaks. Ion damage level and operating conditions can affect the retention and desorption of hydrogen and helium ions. For molybdenum, numerous ion irradiation studies have focused on transition metal or noble gas ions (e.g., Fe, Xe, Kr, Si, He) over dose ranges of 1-100 dpa and temperatures spanning from room temperature to 900°C. These investigations have revealed important phenomena such as dislocation loop formation, void nucleation, radiation-induced hardening, and phase stability changes, typically using characterization techniques like transmission electron microscopy (TEM) and nanoindentation. Despite these advances, several important gaps remain in the literature. Firstly, copper ion irradiation of Mo has been rarely explored, even though Cu ions exhibit distinct nuclear stopping powers and may produce different cascade morphologies compared to commonly studied ions. Secondly, the early stages of radiation damage (at sub-dpa levels) are poorly characterized for Mo, limiting our understanding of defect nucleation and incubation regimes. Furthermore, much prior work emphasizes TEM-based defect imaging and nanoindentation hardness testing, with comparatively fewer studies correlating bulk crystallographic changes (e.g., lattice strain, defect-induced broadening) from X-ray diffraction (XRD) with mechanical property evolution. The response of materials under severe harsh environments can be understood by gaining knowledge of irradiation effects on microstructure, transmutation, phase transformations, precipitation, and dislocation/defect behavior.

In this research, our objective was to understand the influence of high-energy copper ion beams with varying doses (sub-zero to tens of dpa) on the surface morphology, microstructure, residual strain, and hardness of molybdenum samples. A Pellatron accelerator (5 MV) was utilized to generate ion beams with the desired parameters. Following irradiation, characterization techniques including X-ray diffraction (XRD), scanning electron microscopy (SEM), Micro-Vickers hardness testing, and electron probe microanalysis (EPMA) were employed to evaluate modifications in microstructure, residual strain, surface morphology, roughness, and hardness within the crystal lattice.

Materials and Methods

Molybdenum, being an attractive candidate as PFCs in tokamaks, was selected as the sample material to decipher its response under extreme radiation environments. The composition of the samples (99.9% pure Mo) was confirmed using energy dispersive spectroscopy (EDS). Samples of 5 mm thickness were cut from a molybdenum rod with a diameter of 6 mm. To relieve stresses induced during disc cutting and machining, samples were annealed at 600°C for 6 hours in an inert gas environment using a tube furnace.

Rough polishing was performed using silicon carbide emery papers of 400, 600, 1200, 1500, 2000, 3000, and 4000 micron grit sizes to achieve a smooth surface. Fine polishing was then carried out using diamond paste of 5-micron and alumina powder of 1 to 0.012 micron grit sizes in aqueous medium. To remove impurities, dirt, and oil contaminants, samples were cleaned ultrasonically using an acetone bath.

A Pellatron accelerator (5 MV) was utilized to generate 5 MeV copper ion beams. Irradiation was conducted at room temperature under a vacuum of 4×10^{-6} mbar pressure at different fluences. Copper ion beams are used to create high cascade damage in materials, as heavy ions are preferred for achieving high displacement damage. The flux used for sample irradiation was 1×10^{12} ions/cm^2-s.

The crystal structure, crystal size, and micro-strain (%) of pristine and irradiated samples were characterized using a Rigaku Smartlab XRD machine. The θ-2θ scan was used to obtain diffraction patterns. A copper radiation source was used for the scans with Cu Kα average wavelength of $\lambda = 0.15406$ nm. The scan rate was set at 0.083 degrees per second with an increment of 0.01 degrees in the range of 30 to 90 degrees. It is important to note that the broadening of the Bragg peak encompasses both instrument-related broadening and sample-induced broadening. To determine the instrumental broadening $\beta_{\text{instrument}}$, the line broadening measured from a standard silicon sample was subtracted from the measured total broadening (β_{total}) to obtain the actual sample broadening. The instrumental corrected broadening β_{sample}, corresponding to each diffraction peak from molybdenum before and after irradiation, was calculated using the following equation [31]:

$$\beta_{\text{sample}} = \sqrt{\beta_{\text{total}}^2 - \beta_{\text{instrument}}^2}$$

Molybdenum has a body-centered cubic (BCC) structure; therefore, the diffraction peaks analyzed in the study are {110}, {200}, {211}, and {220}.

Surface morphology and elemental composition of pristine and irradiated samples were examined using field emission scanning electron microscopy (FE-SEM) and energy dispersive spectroscopy (EDS). Electron probe microanalysis (EPMA, Rigaku) was also used to confirm elemental composition and surface morphology. Changes in hardness of irradiated samples were measured through micro-Vickers hardness testing using varying indentation loads (10-100 gf).

Results and Discussion

Simulation

SRIM simulation code was used to simulate the irradiation conditions. A displacement threshold energy of 60 eV for molybdenum [29] was used in the simulation. The Quick Calculations of Damage (Q-C) model was used to calculate irradiation parameters. The results indicate that under the irradiation conditions, copper ions penetrate molybdenum to a peak depth of 1.8-2.0 µm, causing peak damage at a depth of 1.3-1.4 µm. The following values and calculations (Equation 1) are used to calculate the displacement damage per atom (dpa) for the 5 MeV copper ion beam fluences of 1×10^{14}, 1×10^{15}, and 1×10^{16} Cu/cm^2.

Total vacancies = 0.69 V/(A-ion)

dpa = $\frac{\Phi \times N_{\text{vac}}}{N_{\text{at}}}$

Copper ion beam irradiation parameters for the molybdenum samples are listed in Table 1. The copper ion concentration in molybdenum and the total number of vacancies produced due to irradiation are shown in Figures 1(a) and 1(b). The calculated peak value of dpa after calculations is 10.8 at a peak depth of 1.3 µm for the copper ion fluence of 1×10^{16} Cu/cm^2, as shown in Figure 2(a). Electron (S_e) and nuclear (S_n) stopping powers of copper ions in molybdenum are shown in Figure 2(b). For 5 MeV copper ions, energy loss by electronic collision is 4.7 keV/nm and energy loss by nuclear collision is 0.4 keV/nm. Electronic stopping power increases with copper ion energy, showing that cascade damage tendency decreases with ion energy.

X-Ray Diffraction

X-ray diffraction patterns of pristine and copper ion beam irradiated molybdenum samples are presented in Figure 3, where the black-line pattern represents the pristine sample. Distinct molybdenum peaks can be observed at 2Θ angles of 40.4°, 58.57°, 73.58°, and 87.56°. After copper ion beam irradiation, peaks show variations in position, intensity, and full width at half maxima (FWHM). Molybdenum peaks shift slightly to the right with increased intensity for the ion dose of 0.108 dpa, while peaks shift to higher 2Θ values with decreased intensity at ion doses of 1.08 and 10.8 dpa, as can be seen in the zoomed (110) peak in Figure 3(a). This peak shift is due to multiple factors including strain, interplanar spacing, and instrumental error. The value of peak shift versus ion beam fluence is shown in Figure 3(b). Figure 3(c) illustrates the variation in lattice constant with ion dose, showing a linear decrease after irradiation.

The lattice constant declines with increasing ion dose, which is indicative of radiation damage in the microstructure. Ion irradiation leads to three primary processes that modify the crystal lattice: (1) ballistic collisions, where energetic ions displace atoms from their lattice sites, creating point defects (vacancies and interstitials); (2) thermal spikes, in which excess primary knock-on atom (PKA) energy is rapidly dissipated as localized vibrations and heat, potentially causing localized melting, phase changes, or amorphization and recrystallization; and (3) atomic displacement cascades, resulting in the formation of defect clusters and dislocations. These processes significantly influence the lattice constant by introducing defects that distort the crystal lattice and alter interatomic spacing. Lattice constant variations accompanied by point defects, phase transformation/amorphization, and dislocations can degrade physical and mechanical properties. Specifically, such damage can reduce thermal/electrical conductivities, lower ductility, promote hardening, and increase the tendency for microcracking. Variation in lattice constant as observed by XRD serves as an atomic-scale indicator of irradiation damage. Radiation can also modify lattice spacing through segregation effects, such as local depletion and enrichment of alloying or implanted species (such as copper in this study). Therefore, the observed decrease in lattice constant with increasing ion dose is consistent with the accumulation of defects and elemental redistribution and is a well-established signature of radiation damage in metals.

To estimate the effect of irradiation on the microstructure of molybdenum crystals, the Williamson-Hall method was used for better accuracy and reliability. An increase in ion dose (1.08 dpa) leads to a decrease in the intensity of major diffraction peaks, indicating that radiation causes localized amorphization or structural disorder. Variation in crystal size and micro-strain as estimated by the Williamson-Hall (W-H) method is shown in Figure 4. According to the W-H method, micro-strain is obtained from the slope of the line and crystal size from the intercept using the following linear equation:

$$\beta \cos\theta = \frac{k\lambda}{D} + 4\epsilon \sin\theta$$

Where the intercept is $\frac{k\lambda}{D}$ and the slope is 4ϵ. From XRD analysis it is clear that crystal size refinement occurs with an increase in ion dose (0.108 dpa) due to fragmentation and recrystallization. The crystal size of pristine samples is 113 nm. After irradiation, crystal size varies from 63.3 nm at 0.108 dpa to 135 nm at 1.08 dpa and 121 nm at the higher dose of 10.8 dpa. Crystal size refinement occurs at lower ion dose, while crystal growth is observed at higher ion dose with crystal size increasing to 135 nm at 1.08 dpa and 121 nm at 10.8 dpa. Variation in micro-strain (%) is also observed. For pristine samples, micro-strain of 0.0426% is observed. Residual strain increases at the lower ion dose of 0.108 dpa to a value of 0.0497%. At higher ion doses of 1.08 dpa (0.0462%) and 10.8 dpa (0.0431%), micro-strain decreases. At lower ion dose (0.108 dpa), micro-strain increases while crystal size decreases. At higher ion doses, micro-strain decreases to lower values (0.0462 at 1.08 dpa) while crystal size increases; however, at an ion dose of 10.8 dpa, micro-strain decreases to 0.0431 while crystal size decreases to 121 nm. A reverse trend between micro-strain and crystal size is observed at the higher ion dose (10.8 dpa). This behavior at higher ion dose might be related to the growth of defects or formation of nanocrystals due to recrystallization. These nanocrystallites are stress-free, resulting in decreased micro-strain and decreased dislocation density as observed in this study. This decrease in dislocation density results in decreased hardness of the material. Material recovery or recrystallization has been observed at higher ion doses by other researchers.

The increase in ion dose leads to a decrease in peak intensity of major peaks (radiation causing amorphization or disorder), accompanied by an increase in micro-strain, while crystal size refinement occurs within the range of <1.5 dpa. XRD results can be used to estimate dislocation density in pristine and irradiated samples. The dislocation density is composed of two contributions: the domain contribution and the deformation contribution. The domain contribution is given by equation 2 and the deformation contribution is given by equation 3, where k is the material constant, D is the crystallite size, ϵ is RMS micro-strain, and b is the Burgers vector. The Burgers vector for BCC is calculated using $(a/2) \times \sqrt{h^2 + l^2 + k^2}$, and the slip plane in BCC structure is (110). Using values of domain and deformation contributions, we obtain equation 7, which is used to estimate dislocation defect density before and after irradiation:

$\rho = \frac{2\sqrt{3}\epsilon}{Db}$

Where ϵ is micro-strain, D is crystal size (nm), and b is the Burgers vector. The value of Burgers vector used for calculations is 0.272 nm. By extracting dislocation density information, one can correlate the effect of displacement damage with dislocation density. It is observed that with increasing ion dose, dislocation density first increases at lower ion dose and then decreases at higher ion dose, as shown in Table 3. At higher dose, chances of dislocation coalescence to voids increase and dislocation density decreases. Dislocation density is inversely related to crystal size and directly related to micro-strain. Increase in micro-strain results in increased dislocation density.

Scanning Electron Microscopy

Changes in surface morphology were observed using field emission scanning electron microscopy (FE-SEM). The FE-SEM micrographs of the pristine sample are shown in Figure 5(a), showing a smooth surface finish without polishing marks. Micrographs of molybdenum samples irradiated with ion doses of 0.108, 1.08, and 10.8 dpa are shown in Figure 5(b-n). Additionally, Figure 5(j) presents the EDS spectrum after ion beam irradiation.

Crack formation is observed on the surface of samples irradiated with an ion dose of 0.108 dpa (Figure 5b-e), while dents and melt crater formation are observed at 1.08 dpa (Figure 5f-i). Defect clusters and deep pit formation are observed on samples irradiated with 10.8 dpa (Figures 5k-n). At low ion dose, high dislocation density (as calculated in the XRD section) increases material flow stress above the inherent fracture stress, resulting in brittle fractures and cracks. Crack formation, voids, blistering, and melt craters have been observed by many researchers following ion beam irradiation, highlighting the need to develop molybdenum alloys for the harsh tokamak environment. There are greater chances of forming higher dislocation density, defects, and vacancies at low ion dose, resulting in cracks. At low fluence, accumulation of high dislocation density elevates material flow stress above its inherent fracture stress, promoting brittle fracture and crack formation.

Higher ion dose (1.08 dpa) can result in dent and melt crater formation, as observed in our study and many others. Small deep pits are observed in Figure 5(f,i) and some pores can also be observed at higher ion dose (10.8 dpa). At higher ion dose (10.8 dpa), the growth rate of voids is limited due to void annihilation and low mobility at room temperature. Temperature plays an important role in void formation and defect coalescence. According to Jizhong Zhang et al., at 1×10^{15} ions/cm^2, surface damage with irregularly shaped voids and dents of varying sizes was evident. At a higher fluence of 1×10^{16} ions/cm^2, a peeling-off effect on the surface caused deep voids. With increasing ion fluence, they observed increased void length.

Elemental analysis confirms the samples are molybdenum with 99 weight% Mo, and after irradiation, no new peaks are observed in XRD and EDS patterns, indicating no phase change in the material, as shown in Figure 5(j).

Electron Probe Microanalysis

EPMA works by bombarding the specimen with a beam of monoenergetic electrons to generate characteristic X-rays. Because atoms have discrete electron energy levels, excitation by the electron beam causes them to emit photons at specific energies in the X-ray region. These characteristic X-rays uniquely identify each element and enable quantitative microanalysis. EPMA results are shown in Figure 6 to confirm radiation damage and observe changes in surface morphology of samples irradiated under varying ion doses. Figures 6(a-c) represent surface morphology at low ion dose (0.108 dpa), while Figures 6(d-f) and (g-i) represent morphology at 1.08 and 10.8 dpa. It can be seen from Figure 6 that pit size increases with ion beam fluence while pit density decreases with increasing fluence (Figure 6c, 6f, and 6i).

Vickers Hardness

Irradiation hardening is the primary cause of embrittlement in structural materials. To evaluate the effect of copper ion beams on the mechanical properties of molybdenum, sample hardness before and after irradiation was measured using a Vickers microhardness tester with indentation loads of 10, 25, 50, and 100 gf. Figure 7(a) shows Vickers hardness versus indentation loads for both pristine and copper ion beam treated molybdenum samples. It is clear from Figure 7(a) that hardness increases after ion beam irradiation compared to pristine samples at ion doses of 0.108 and 1.08 dpa. However, hardness decreases at the higher ion dose of 10.8 dpa, as observed by other researchers. Figure 7(b) shows hardness variation at different ion doses. Hardness increases up to 1.08 dpa, but at 10.8 dpa there is a decrease compared to lower dose samples.

All irradiated samples reveal hardening phenomena compared to unirradiated samples. For 0.108 dpa, hardness increases are observed at 10, 25, 50, and 100 g loads. At lower ion dose, there are more chances of interstitial and vacancy defects, unstable phase transformation, and grain size refinement (fragmentation). Higher dislocation density at low ion dose can impede dislocation movement/slip and result in hardening. At higher ion dose (10.8 dpa), a decrease in hardness is observed compared to pristine samples. At higher ion dose, coarsening of crystal size or increase in dislocation size occurs with decreased dislocation density, which is the main cause of the hardness decrease at higher fluence.

The change in hardness for different ion doses at an indentation load of 10 gf is shown in Figure 7(b). Hardness increases after molybdenum irradiation have been observed by several authors and might be due to defects like vacancies, SIA, and dislocation clusters/loops that affect dislocation mobility. Higher defect density is produced at lower ion beam fluence, resulting in hardness increases at lower ion doses (0.108 to 1.08 dpa). At higher ion dose (10.8 dpa), crystal growth and dislocation clustering cause the hardness decrease. Decreased hardness can also be attributed to defect recombination and some recrystallization/recovery. At higher dose, chances of dislocation coalescence increase and dislocation loop size increases. Formation of large dislocation loops contributes to material hardness reduction. Therefore, hardness decrease at higher doses could be due to two reasons: increased crystal size and formation of large dislocation loops or swelling.

The Hall-Petch relationship predicts hardness changes with grain size, reporting that hardness increases with decreasing grain size. The reason behind increased hardness is that with decreasing crystal size, there are reduced chances of dislocation formation and movement, retarding plastic deformation of the material (high resistance to deformation and high hardness). The values of average hardness for pristine and irradiated molybdenum samples and hardness changes are shown in Table 4. The observed variation in Vickers hardness with increasing irradiation dose from a pristine value of approximately 260 HVN, rising sharply to 371 HVN at 0.108 dpa and 401 HVN at 1.08 dpa, and subsequently declining to 336 HVN at 10.8 dpa is consistent with the known dose-dependent evolution of irradiation-induced defect structures in body-centered cubic (BCC) metals such as molybdenum. At low dose, hardness increases, but at higher dose, a decline is observed in this study.

Discussion

Copper ion beam irradiation induces significant structural damage in molybdenum, thereby altering its mechanical properties. Evidence of this structural damage is obtained from X-ray diffraction (XRD) analysis, which reveals changes in peak intensity, peak position (shift), and full width at half maximum (FWHM). These variations indicate irradiation-induced modifications in lattice spacing arising from either strain accumulation or relaxation mechanisms such as dynamic recovery. Grain growth or coarsening is also observed post-irradiation, which can influence mechanical properties according to the Hall-Petch relationship. At the lowest dose of 0.108 dpa, collision cascades generated by 5

MeV Cu ions produce a high density of point defects and small defect clusters. These defects act as strong barriers to dislocation motion, leading to pronounced radiation-induced hardening. XRD results suggest an increased density of such defect clusters, which are highly effective in obstructing plastic deformation. Consequently, a peak in microhardness is observed at 1.08 dpa, corresponding to a 54.2% increase compared to the unirradiated sample.

At higher doses (10.8 dpa), several competing mechanisms reduce the effectiveness of these irradiation-induced obstacles. Overlapping collision cascades promote dynamic recombination and annihilation of point defects, lowering net defect retention. Additionally, dislocation loops can grow and coarsen via defect absorption and cascade-induced mobility. As loops become larger and more widely spaced, their strengthening effect diminishes due to the inverse relationship between obstacle spacing and strengthening, as described by Orowan's law. Furthermore, in thin ion-irradiated layers (typically a few micrometers thick), free surfaces act as strong sinks for point defects, enhancing their annihilation and reducing retained damage. Even at room temperature, athermal mechanisms such as ballistic recombination within cascades and defect cluster migration induced by collision spikes can promote dynamic recovery. As a result, the defect population evolves from a high density of small, strong obstacles to a lower density of larger, less effective obstacles, leading to hardness decrease with continued irradiation. This phenomenon of initial ion irradiation-induced hardening followed by saturation or even softening at higher doses is widely reported in the literature for metals, including molybdenum, and reflects the complex competition between defect production, evolution, and annihilation processes under irradiation conditions. Further radiation-induced recrystallization nucleates strain-free nanocrystallites, resulting in lower dislocation density and decreased hardness. It is now evident that with increasing ion dose on molybdenum samples, hardness first increases (54.2%) up to 1.08 dpa, but then decreases (29.23% softening) at the high dose of 10.8 dpa due to increased crystal size, decreased dislocation density, and recovery/recrystallization of strain-free nanocrystallites. In summary, hardness evolution with increasing ion dose reflects typical behavior observed in irradiated metals: initial hardening due to defect accumulation, followed by saturation and softening at higher doses, governed by the complex interplay between defect generation, migration, recombination, and recovery processes.

Conclusions

Copper ion beam irradiation at doses of 0.108, 1.08, and 10.8 dpa resulted in significant changes in the microstructure, surface morphology, and mechanical properties of molybdenum samples. XRD results reveal that irradiation caused shifts in diffraction peaks, indicating variations in crystal size, lattice strain, and interplanar spacing. At a low ion dose of 0.108 dpa, crystal size decreased (fragmentation) to 63.3 nm, suggesting defect accumulation. At higher doses (1.08 and 10.8 dpa), crystal size increased (to 135 and 121 nm), consistent with radiation-induced grain growth or defect recovery (radiation annealing effects). Micro-strain increased at 0.108 dpa (from 0.0426 to 0.0497%), indicating initial lattice defect accumulation, but decreased at higher doses (0.0462% at 1.08 dpa, 0.0431% at 10.8 dpa), consistent with strain relaxation due to annealing. Crystal lattice contraction is observed with increasing ion dose.

At low ion dose of 0.108 dpa, surface cracking was observed, attributed to irradiation-induced hardening and increased flow stress exceeding fracture stress due to high dislocation density. Surface damage like dents, pits, and melt craters is observed at ion doses of 1.08 and 10.8 Cu/cm^2. The progression of surface damage with fluence indicates increasing radiation-induced defect clustering and surface morphology changes. Sample hardness increased at lower ion doses (0.108 and 1.08 dpa) due to higher dislocation density and crystal size refinement. However, at higher ion dose (10.8 dpa), material hardness decreased due to crystal growth (reducing grain boundary strengthening) and decreased dislocation density. The hardness trend indicates initial irradiation-induced hardening at low doses, followed by softening at higher doses due to defect recovery and coarsening effects. Pure molybdenum is unable to bear high damage levels; therefore, development of molybdenum alloys is recommended to withstand the harsh tokamak environment.

Acknowledgements

The authors would like to express gratitude to the faculty of the School of Nuclear Science and Technology, Lanzhou University, China. Financial support from the National Science Foundation of China through research project NSFC No. U2267253 is acknowledged. The authors are also grateful to the members of the National Center for Physics, Pakistan for their support in providing copper ion beams.

Credit Author Statement

Bilal Muhammad: Writing original draft, Data curation, Methodology, Investigation, Formal analysis.
Limin Zhang: Data curation, Validation, Supervision, Data analysis.
Kunshan Yang, Kashif Shahzad, Turab Ali, Zahoor Ahmad: Data curation, Writing review & editing.
Tieshan Wang: Project administration, Supervision, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Note: Figure translations are in progress. See original paper for figures.