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Effect of starting microstructure on helium plasma-materials interaction in tungsten☆

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ABSTRACT

In a magnetic fusion energy (MFE) device, the plasma-facing materials (PFMs) will be subjected to tremendous fluxes of ions, heat, and neutrons. The response of PFMs to the fusion environment is still not well defined. Tungsten metal is the present candidate of choice for PFM applications such as the divertor in ITER. However, tungsten's microstructure will evolve in service, possibly to include recrystallization. How tungsten's response to plasma exposure evolves with changes in microstructure is presently unknown. In this work, we have exposed hot-worked and recrystallized tungsten to an 80 eV helium ion beam at a temperature of 900 °C to fluences of 2×10^{23} or 20×10^{23} He/m^2. This resulted in a faceted surface structure at the lower fluence or short but well-developed nanofuzz structure at the higher fluence. There was little difference in the hot-rolled or recrystallized material's near-surface ($\leq$50 nm) bubbles at either fluence. At higher fluence and deeper depth, the bubble populations of the hot-rolled and recrystallized were different, the recrystallized being larger and deeper. This may explain previous high-fluence results showing pronounced differences in recrystallized material. The deeper penetration in recrystallized material also implies that grain boundaries are traps, rather than high-diffusivity paths.

1. Introduction

Plasma-facing materials (PFMs) and components (PFCs) in a magnetic fusion energy (MFE) device, such as a tokamak, will be subjected to high fluxes of heat, plasma ions (He and D/T), and neutrons. Because of its high melting point and low sputter yield, tungsten is the primary candidate for MFE applications such as divertors in ITER. Under transient thermal loads, such as might be experienced when a plasma disruption strikes the PFC, the temperature at the surface of the tungsten might undergo a large excursion and result in recrystallization of the material. Macroscopically, recrystallized tungsten has poor mechanical properties in comparison to tungsten with a hot worked or fine-grained structure.

A less explored topic is how changes in the underlying microstructure (such as deformed vs. recrystallized) will affect the interaction of the plasma ions (particularly He) with the underlying material). Studies on deuterium found highly recrystallized material, with very low intrinsic defect density, retained much less deuterium than hot-worked, highly defective material [1]. Phenomenologically, linear plasma devices found comparatively worse helium plasma response in recrystallized material at high plasma fluences [2]. Microscopically, with greatly reduced grain boundary area and dislocation density, recrystallized tungsten presumably undergoes different helium disposition and bubble growth behavior, but this is not well characterized.

Previous work has explored the high-fluence microstructural changes [2–4], and found significant changes in the overall performance of different grades of tungsten after long exposures to He plasmas. Although the differences between most of the tungsten grades were minor, the recrystallized material showed significant growth of nanofuzz deep within the specimen, which is surprising and difficult to explain [2].

Grain boundaries and dislocations have been shown, both by experiment and by calculations, to increase helium trapping and form excess densities of bubbles at the grain boundaries or

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dislocation lines. However, most of the dislocation work on low-energy (sub-displacement-threshold) helium has used theoretical simulations, rather than experiment, and have examined punched dislocation loops from growing bubbles, rather than line dislocations [5,6]. However, experiments have indicated that line dislocations will collect helium preferentially [7,8], but comparisons of dislocation effects under low-energy helium across different tungsten microstructures are lacking. Grain boundaries also sequester plasma-introduced helium differently than the matrix. Results from theory approaches (molecular statics and molecular dynamics [MD]) indicated that the {112} 70.5° Σ3 coincident site lattice (CSL) special grain boundary in tungsten would attract significantly more helium, and therefore form larger, denser, and deeper bubbles than the surrounding matrix [9].

A number of computational (primarily MD or related methods) studies have examined the influence of bubble growth on nanotendril fuzz formation. Bubble formation and bursting, especially leading to changes in the local concavity and convexity, appear to drive growth of nanotendrils or surface flaking [10,11]. Collection of helium platelets onto {101} planes was used to explain scanned-probe microscopy results on faceting tungsten surfaces [12]. Bubbles can also make local nucleation of new satellite bubbles easier [13], and the nucleation of new bubbles within tendrils appears to drive further growth [14]. New grains also appear to nucleate randomly along the length of the growing tendrils [15]. He fluence will also change the disposition of the helium, absent any differences in microstructure. Theory results have indicated that changes in fluence affect the growth of the helium bubbles, where higher flux caused the bubbles to grow with a bias toward the surface, and concomitantly more surface damage [16]. Changes in the propensity for trap mutation as a function of depth (distance from the free surface, and its image forces) were also seen to make trap mutation and loop punching easier in a shallow bubble [17,18].

Although the above summary shows that many pieces of the puzzle of tungsten surface changes under He exposure are known, there are also many unknowns remaining. Particularly, much of the previous work has used computational rather than experimental methods, and MD or other theory approaches have trouble reaching the long exposure times at low fluxes needed to match in-tokamak service. Experimental studies (e.g., Refs. [3,7,8,19,20], amongst many others) have well-explored individual aspects of surface morphology changes.

Here, we explore the influence of the starting structure of high-purity tungsten – specifically, in a hot-rolled state containing many grain boundaries and dislocations vs. a recrystallized state with very few of these intrinsic defects – on the evolution of the surface morphology and the near-surface microstructure.

We hypothesize that He interaction with pristine or near-pristine matrix, as would be produced in a recrystallized region away from grain boundaries, will show a different bubble size population and bubble distribution than in a highly deformed matrix. Specifically, we hypothesize that in hot-worked material, with more grain boundaries and dislocations, helium bubbles in the matrix are likely to be smaller, owing to easier and denser nucleation at heterogeneous sites. We also hypothesize that in recrystallized material, helium bubbles introduced by helium impingement will be larger and less dense, due to a greater necessity for homogeneous nucleation (such as the trap mutation [21]). Therefore, we exposed hot-worked and recrystallized tungsten to 80 eV He ions at 900 °C in order to measure the helium bubble and nanofuzz features to test this hypothesis. We restricted the experiment to moderate fluences (~10^{23}–10^{24} He/m^2) in order to avoid the formation of later, high-density defects that would preclude electron microscopic characterization of individual defects.

2. Experimental procedure

Samples were high-purity (nominally 99.99%) tungsten from ESPI metals, USA. The sample was received in the form of hot-rolled plate, ~3 mm in thickness. Sample coupons were cut to a size of 10 × 15 × 1 mm and mechanically polished with SiC polishing pads, diamond suspension, and a colloidal silica mixture. Some coupons were heat treated under vacuum to 1600 °C for 2 h to recrystallize the structure. We will call the hot-rolled samples HR, and the recrystallized, RX.

Helium exposures were performed on the Multicharged Ion Research Facility (MIRF) at ORNL, which uses a deceleration module [22,23] to produce a high-flux 80 eV helium ion beam. In these experiments, peak fluxes were ~1–2 × 10^{20} He/m^2sec. Exposures were at 900 °C to nominal fluences of 2 × 10^{23} He/m^2 or 20 × 10^{23} He/m^2, yielding an overall matrix of four conditions: HR 2 × 10^{23}, HR 20 × 10^{23}, RX 2 × 10^{23}, and RX 20 × 10^{23}. The fluence was delivered over a spot ~2 × 3 mm in size and lenticular in shape; SEM, FIB, and TEM were all performed near the geometric center of the beam spot (~1 mm diameter region), where the peak flux occurred. The RX 20 × 10^{23} sample actually received an estimated peak fluence of approximately 18 × 10^{23} He/m^2 (~10% less), but will be called “RX 20 × 10^{23}” for consistency. Similarly, the RX 2 × 10^{23}received an estimated fluence of 2.3 × 10^{23} He/m^2 (~15% more), but will be called “RX 2 × 10^{23}” for consistency. Best estimates of fluence for the HR samples are, indeed, 2 and 20 × 10^{23} He/m^2.

Microstructural investigations were performed using a JEOL JSM 6500F field-emission scanning electron microscope (SEM) equipped with electron backscatter diffraction (EBSD). Focused ion beam (FIB) was used to perform sample preparation for scanning/transmission electron microscopy (S/TEM). The FIB used was an FEI Quanta 3D DualBeam™ FIB-SEM. Additionally, high-resolution SEM and electron-channeling contrast imaging (ECCI) [24] were performed in an FEI Versa 3D DualBeam™ FIB-SEM equipped with a high-resolution field-emission SEM column, using an annular solid-state backscatter electron detector. ECCI was performed at 30 keV at very short (~4–5 mm) working distance, imaging using the outermost sector of the four-sector annular detector. S/TEM was performed using a JEOL JEM2100F S/TEM or an FEI Talos F200X S/TEM. TEM samples from helium-exposed regions were prepared using FIB methods. TEM samples from the unirradiated specimens (Fig. 2) were prepared by electropolishing 3 mm disks in an NaOH solution. Both S/TEMs operated at 200 keV. The JEOL JSM 6500F is located in the Materials Characterization Facility (MCF) at ORNL, and the other instruments at the Low Activation Materials Development and Analysis (LAMDA) facility [25,26] at ORNL.

3. Results

3.1. Surface observations

The pre-exposure microstructures were those expected for HR and RX microstructures. EBSD maps of the gross microstructures, Fig. 1, show the HR and RX grain structures. The HR shows a fine subgrain structure and the RX shows large, nearly strain-free, grains; Fig. 1a–b. Grain sizes are HR: 2.2 ± 2.0 μm and RX: 16.1 ± 10.3 μm. These values are mean ± standard deviation of the equivalent diameters, and with a 5° misorientation needed to define a grain boundary (grain tolerance angle). These calculations were performed using EDAX OIM-Analysis software, v7. The distributions are positively skewed, with most grains below the mean and a small number of outliers above the mean.

Inverse pole figure calculations of the texture in the Z-direction indicate strong <001> preferred orientation in both samples; the