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Effect of tungsten crystallographic orientation on He-ion-induced surface morphology changes ☆

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Abstract

In order to study the early stages of nanofuzz growth in fusion-plasma-facing tungsten, mirror-polished high-purity tungsten was exposed to 80 eV helium at 1130 °C to a fluence of 4×10^{24} He m^{-2}. The previously smooth surface shows morphology changes, and grains form one of four qualitatively different morphologies: smooth, wavy, pyramidal or terraced/wide waves. Combining high-resolution scanning electron microscopy (SEM) observations to determine the morphology of each grain with quantitative measurement of the grain's orientation via electron backscatter diffraction in SEM shows that the normal-direction crystallographic orientation of the underlying grain controls the growth morphology. Specifically, near-⟨001⟩ ‖ normal direction (ND) grains formed pyramids, near-⟨114⟩ to ⟨112⟩ ‖ ND grains formed wavy and stepped structures and near-⟨103⟩ ‖ ND grains remained smooth. Comparisons to control specimens indicate no changes to underlying bulk crystallographic texture, and possible explanations of the structure growth, particularly loop-punching, are discussed. Future developments to control tungsten texture via thermomechanical processing, ideally obtaining a sharp near-⟨103⟩ ‖ ND processing texture, may delay the formation of nanofuzz.

Keywords: Tungsten; Electron backscatter diffraction; Ion irradiation; Helium; Dislocation loop punching

1. Introduction

Because of its high melting point, excellent erosion resistance and material strength, tungsten is favored for wall materials for next-generation magnetic fusion devices. In deuterium-tritium (D–T) fusion operation, significant He ash will be formed at 3.5 MeV, and its interaction with tungsten wall materials is currently undergoing extensive evaluation.

Under certain conditions of wall temperature and He fluence, He ions were found to cause significant morphology changes in the W surface [1–5], even for impact energies below the physical sputtering threshold of ~200–300 eV. While still not completely understood, such surface changes are thought to result from near-surface He trapping at intrinsic or extrinsic defect sites. Due to favorable energetics, the trapped He atoms form clusters, which result in progressively higher lattice distortions, which relax by dislocation loop punching, producing He-filled cavities (bubbles) of increasing size. These He bubbles can eventually burst at high sample temperatures where the tungsten has lower effective viscosity or yield strength, causing surface pinholes, and eventually evolving into an increasingly random nanostructuring of the surface that can ultimately lead to the production of nanofuzz [6–9] Orientation dependence on nanostructuring has been reported [10]. There is concern that such surface nanostructuring may adversely affect the

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robustness of the W surface in the presence of the high-power fusion plasma due to increased W dust formation, as well as affect H and He retention. In addition, the effect of the bulk material damage due to 14.1 MeV neutrons also produced in the D–T fusion reactions on these near surface He–W interactions has yet to be definitely assessed.

In this experiment, we exposed highly polished tungsten coupons to 80 eV helium ions of flux 10^{20} m^{-2} s^{-1} to a fluence of 4×10^{24} He m^{-2} at 1130 °C (1400 K) to study the early stages of nanofuzz formation. Scanning electron microscopy–electron backscatter diffraction (SEM–EBSD) was used to correlate the changes in the surface morphology of the grains caused by the helium exposure to the underlying crystallographic orientation, revealing a strong and systematic effect.

2. Experimental details

2.1. Ion irradiation

High-purity tungsten sheet 0.7 mm in thickness was fabricated by electrical discharge machining (EDM) into 13×13 mm coupons and polished to colloidal silica (0.05 μm) for a mirror polish. Tungsten substrates were examined via X-ray photoelectron spectroscopy (XPS) as-cut and after cleaning with detergent and water rinse. Aside from W, carbon and oxygen were the primary surface species observed. Minimal argon-ion sputtering reduced the C from 30 at.% to ~5 at.% and O from 20 at.% to ~4 at.%, showing that these elements were adsorbed atmospheric contaminants. Several of the He-exposed substrates were examined and showed no additional surface contamination from the He processing.

The irradiations were performed at the ORNL Multicharged Ion Research Facility (MIRF) [11] using a new high-current beam deceleration module installed in the beamline of our CAPRICE electron cyclotron resonance (ECR) ion source, which has been described elsewhere [12]. This deceleration module provides high-flux He ion beams at energies down to 50 eV to W targets floating at the deceleration potential. The W samples can be raised to temperatures exceeding 1300 °C by electron beam heating. A recently added beam profile measurement device consisting of a positively biased beam catcher inside an enclosure with a 1 mm^2 aperture can be scanned across the incident He ion beam via a PC controlled, stepper motor driven, x–y–z manipulator to determine the two-dimensional flux distribution of the incident beam [13].

2.2. Electron microscopy

Specimens were examined in a JEOL JSM-6500F field-emission analytical SEM. High resolution secondary electron (SE) images were acquired at 5 keV with small beam current (<100 pA) and short working distance (~4–7 mm).

EBSD was performed using an EDAX-TSL Hikari high-speed camera. Beam conditions were 30 keV, 4.0–4.5 nA, with a 19–20 mm working distance. The tungsten coupons were attached to the EBSD specimen holder with silver paint, rather than conductive tape, to minimize sample drift and to allow close correlation between SE images (acquired immediately before EBSD) and the EBSD data. Grain calculations are based upon the definition that a >15° pixel-to-pixel misorientation defines a grain boundary.

Texture pole figures were calculated with 5.0° half-width, and order $L = 34$ harmonic series expansion, triclinic sample symmetry and without using any data cleaning methods [14] of the raw data unless explicitly noted below.

3. Results

3.1. Irradiated areas

The irradiated specimen (Fig. 1a) showed significant surface morphology changes from the pre-irradiated, mirror-polished condition. Four broad morphology categories are identified, and in Fig. 1b these are marked (i)–(iv), and shown in more detail in Fig. 1c and d. In Fig. 1b, (i) denotes a smooth or nearly-smooth region, (ii) denotes a wavy morphology, (iii) denotes pyramidal morphology and (iv) denotes terraced morphology. Obviously, these are qualitative assessments and particular grains may show an intermediate or ambiguous morphology. Judgment was used to differentiate the types of morphology, and as will be seen below, this provided results that correlate well to underlying crystallography.

A large-area texture scan of the irradiated area is shown in Fig. 2. Fig. 2a shows a normal direction (ND)-projected inverse pole figure (IPF) map, and Fig. 2b the calculated pole figures (PFs) and an ND IPF. PFs indicate the relative number of grains with a given orientation ⟨h k l⟩ oriented in a particular sample direction, measured relative to a random polycrystal. A strong (≈8 × random) ⟨0 0 1⟩ ∥ ND grain texture is seen, also with ⟨0 0 1⟩ in-plane. Relatively few ⟨1 0 1⟩ ∥ ND or ⟨1 1 1⟩ ∥ ND-type grains are seen. The average effective grain diameter is $\sim 11 \pm 7$ μm, indicating a wide distribution. The sharp ⟨0 0 1⟩ texture is consistent with reports of rolling and recrystallization in tungsten [15–17].

It is important to note that EBSD data require significant tilting of the specimen away from the surface normal, 70° away from the normal electron-beam incidence angle in this case. Fig. 3 shows a small area imaged using the EBSD software (which compensates for dynamic focus and the ~3:1 difference in pixel size in the Y and X scan directions) imaged at 70° tilt, and the same area imaged with the SEM software at 0° tilt. First, note that the images match well; second, note that the features' third-dimension information, such as the relative heights and angles of the pyramids or waves, are greatly exaggerated in the EBSD-tilt-acquired image. Grains (i) and (ii) are of a wave morphology when imaged at 0°, but appear to be sharp pyramids at 70° tilt.