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Characterizing the recovery of a solid surface after tungsten nano-tendril formation

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ABSTRACT

Recovery of a flat tungsten surface from a nano-tendril surface is attempted through three techniques; a mechanical wipe, a 1673 K annealing, and laser-induced thermal transients. Results were determined through SEM imaging and elastic recoil detection to assess the helium content in the surface. The mechanical wipe leaves a ~0.5 μm deep layer of nano-tendrils on the surface post-wipe regardless of the initial nano-tendril layer depth. Laser-induced thermal transients only significantly impact the surface morphology at heat loads of 35.2 $MJ/m^2 s^{1/2}$ or above, however a fully flat or recovered surface was not achieved for 100 transients at this heat load despite reducing the helium content by a factor of ~7. A 1673 K annealing removes all detectable levels of helium but sub-surface voids/bubbles remain intact. The surface is recovered to a nearly flat state with only some remnants of nano-tendrils re-integrating into the surface remaining.

1. Introduction

The growth of tungsten (W) nano-tendrils, or "fuzz", on surfaces at high temperatures (>1000 K) exposed to helium (He) plasma ions is well established in linear devices [1–3] as well as tokamaks [4]. The exact growth mechanism and impact of these tendrils is still under debate. In an experimental device, such as ITER, if plasma conditions where nano-tendril growth can occur are accessed in the W divertor, removal techniques, preferably in-situ, will be desired to recover a pristine W surface.

Studies have shown several methods to "recover" from a nano-tendril surface (i.e. removal of nano-tendrils from a tungsten surface). Due to the mechanical fragility of W nano-tendrils, mechanical removal of the tendrils is an option [5] and will leave the bulk material unaffected. Nano-tendrils have also been found to re-integrate into the surface from high temperature annealing [5–8], which releases the He trapped in the W. Finally, thermal transients on the surface have also been shown to modify nano-tendril structures [3,9].

These different surface "recovery" techniques have subtle differences. The mechanical removal simply removes the tendrils but has no effect on the bulk. The high temperature anneal heats the tendrils and the bulk equally. The thermal transients deposit most of their energy, and thus induce temperature increases mostly in the tendrils themselves while the temperature of the majority of the W bulk is unaffected. The purpose of this study is to compare the effects of these three surface recovery techniques in terms of the changes to the surface and sub-surface morphologies as well as the He content of the targets. Differences observed between these techniques will give insight into the mechanisms leading to tendril removal or re-integration into the surface. This, in turn, may provide further clues as to the mechanisms that induce the nano-tendril growth initially.

2. Experiment

2.1. Mechanical removal of W nano-tendrils

The nano-tendril surfaces to be recovered via mechanical wipe were grown in the helicon plasma source of DIONISOS [10]. The nano-tendril surfaces were grown on a polished 99.95 at.% pure W target. The targets were exposed to pure He plasma for 3600 s and plasma flux of $(3 \pm 1) \times 10^{22}$ m^{-2} s^{-1}. One target was exposed at a temperature of 1250(±100) K and a bias of −60 V and a second target was exposed at a target temperature of 1400(±50) K and a bias of −90 V.

The mechanical wipe was performed on a Buehler Ecomet 3000 polisher with an Automet 2 head. A Chemia MB fine polishing cloth was mounted on the Ecomet 3000 rotating at 10 rpm. The W

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nano-tendril sample was mounted on the Automet 2 head at 60 rpm in co-rotation with the base. The W nano-tendril surface was pressed into the polishing cloth with 8.9 N of perpendicular force for 3 s.

The surface and sub-surface morphology were examined with SEM and focused ion beam cross-sectioning. Images were taken before and after the mechanical removal of the nano-tendrils. The He content of the surface was measured via heavy ion elastic recoil detection (ERD) [11]. For this work, a 6 MeV O^{3+} ion beam was used with a scattering angle of 35°. A 6 μm mylar foil was placed in front of the solid-state detector to block the reflected O^{3+} but allow the forward-scattered He and hydrogen from the surface to pass through. With this set-up and these O^{3+} energies, the He concentration can be measured across an areal density of $\sim 3 \times 10^{17}$ W/cm^2, which is equivalent to a depth of 50 nm in bulk W or 500 nm of W fuzz if it is assumed ~90% porosity for W nano-tendril layers. The He content in this layer is quantified by using simNRA [12] to fit experimental spectra. The He content detection limit for the ERD conditions used in this work is ~0.1 at.%. Previous work [11] has found that the He concentration is roughly constant within the nano-tendril layer and that holds true for this work as well (i.e. He concentration for the entire detection depth falls within the error bars of Fig. 4).

2.2. High temperature annealing of W nano-tendril surfaces

The W nano-tendril surfaces for the high temperature annealing were also grown in DIONISOS under similar plasma fluxes and conditions for 3600 s. One target was exposed at a temperature of 1200(±50) K and a bias of −70 V and a second target was exposed at a target temperature of 1450(±50) K and a bias of −80 V.

Both targets were annealed at the same time. The annealing was performed in a R.D. Webb vacuum furnace. The furnace temperature was ramped at a rate of 1 K/s to 1673 K and held at that temperature for 3600 s.

The same procedures to investigate surface and sub-surface morphologies and surface He content from Section 2.1 were used on the annealed targets.

2.3. Transient heating of W nano-tendril surfaces via laser pulse

The W nano-tendril surface for transient heating was grown in PILOT-PSI [3]. The W target was exposed to pure He plasma with a central flux density of 9×10^{23} m^{-2} s^{-1} for a total of 630 s distributed over 9 equal plasma discharges. The surface temperature during plasma exposure was 1500 ± 50 K and the target was biased at −38 V.

Different spots of the nano-tendril surface were exposed to different thermal transients. In this study we investigate three spots denoted by A, B, and C. The conditions for each laser spot are defined by power, heat flux factor (F_{HF}) and the number of pulses (see Table 1). All laser pulses are 1 ms in duration, which results in a heat penetration skin depth of ~50 nm in bulk W and ~500 nm in a 90% porous nano-tendril layer. The temperature rise (ΔT) of the surface was measured by high-speed IR camera (SC7500M, FLIR Systems Inc, 1.5–5.1 μm) and the bulk tungsten was kept at room temperature. On the initial laser pulse, where an unmodified, fully formed nano-tendril layer with emissivity of 0.8 [13] is assumed, ΔT = 3100 ± 200 K for all three spots. In the absence of a method to measure the surface emissivity directly we have used estimates from previous work, however any over- or under-estimation of the emissivity will have an impact on the measured ΔT. The temperature rise for subsequent pulses and the average for the total number of pulses are lower than for the initial pulse but difficult to quantify accurately as the absorbance and emissivity of the surfaces can change as the surface morphology evolves. More details on the temperature measurement techniques can be found in [14] and [15].

Analysis techniques of the surface are similar to Sections 2.1 and 2.2, but with some key differences. Focused ion beam cross-sectioning and SEM imaging was used on the laser spots to investigate the final state of the surface and the nano-tendrils just outside the laser spot (but at a similar radial location on the target) are assumed to be representative of the surface before the laser heating. This was also assumed for the He content ERD measurements. A spot just outside the laser spot is assumed to be the unperturbed He content of the nano-tendrils on the surface. To measure the He content of the surface after the laser pulses, a 0.3 mm beam aperture was used such that the entire beam spot could fit within the ~2 mm diameter laser spot.

Table 1
Details of thermal transient conditions for laser spots.

Spot	Power (GW/m^2)	F_{HF} (MJ/m^2 s$^{1/2}$)	# of pulses	Pulse rate (Hz)
A	1.11	35.2	100	10
B	0.61	19.4	1000	40
C	0.62	19.5	10000	40

3. Results and discussion

3.1. Mechanical removal of W nano-tendrils

The SEM images show that the mechanical wipe was unsuccessful in fully removing the nano-tendril structures from the surface for both 1250 K and 1400 K exposure temperatures (see Fig. 1). From the cross-section images it is seen that the initial fuzz layer was significantly thicker for the 1400 K exposure (~2 μm layer depth) than for the 1250 K exposure (~0.8 μm layer depth), but

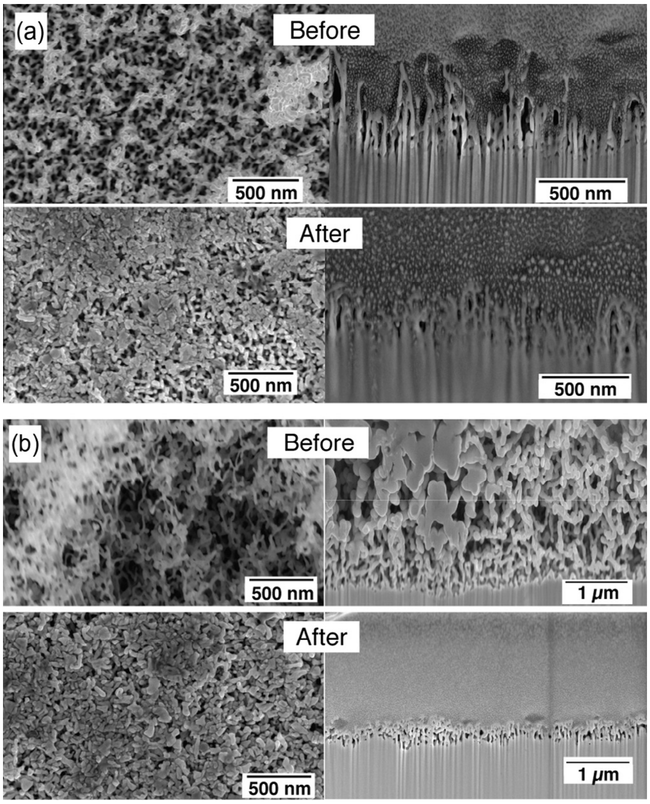

Fig. 1. Normal and cross-section SEM images before and after a mechanical wipe of W nano-tendril surfaces grown at (a) T_{surf} = 1250 K, and (b) 1400 K. Viewing angle is 52° for cross section images.