



Ordered arrangement of irradiation-induced defects of polycrystalline tungsten irradiated with low-energy hydrogen ions



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ABSTRACT

Low-energy (20–520 eV) hydrogen ion irradiations were performed at W surface temperature of 373–1073 K and a fluence ranging from 5.0×10^{23} to $1.0 \times 10^{25}/\text{m}^2$. Conductive atomic force microscopy (CAFM) as a nondestructive analytical technique was successfully used to detect irradiation-induced defects in polycrystalline W. The size and density of these nanometer-sized defects were strongly dependent on the fluence of hydrogen ions. Both ion energy (E) and temperature (T) play a crucial role in determining the ordering of nanometer-sized defects. Ordered arrangements were formed at relatively high E and T . This can be attributed to the stress-driven ripple effect of defect growth at crystal grains, resulting in the movement of W lattice along one certain crystal planes.

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1. Introduction

In ITER, plasma-facing materials (PFMs), such as tungsten are subjected to high fluences of low-energy hydrogen and helium ions, leading to the hydrogen isotope retention on tungsten under D–T fusion circumstance [1–5]. The effect of W microstructures on the hydrogen isotope retention has been widely studied [6–8]. It has been accepted that impurities, dislocations, vacancies, and voids in W can trap H atoms if they are injected from the plasma of a fusion reactor. D atoms were mainly trapped at radiation damage sites. Both the number density of blisters and hydrogen accumulation at the grain boundaries were greatly decreased due to the significant trapping of hydrogen isotopes at radiation damage sites. Addition of 10% helium ions into the D plasma at exposure temperatures of 440–773 K significantly reduced the D concentration compared to that for the pure plasma exposure [9,10]. The interaction between hydrogen isotopes and irradiation-induced defects has to be taken into account when W is selected as a plasma facing material for ITER [11,12]. The relation between the irradiation defects and the implanted hydrogen remains unclear and needs to be further investigated.

In this study, conductive atomic force microscopy (CAFM) as a nondestructive analytical technique is used to investigate the interaction between H atoms and polycrystalline W. CAFM analysis shows the existence of nanometer-sized defects in polycrystalline W due to the irradiation of low-energy hydrogen ions. Depending

on the irradiation temperature and the energy of hydrogen ions, these defects may be arranged in order over each polycrystalline W grain. This study suggests that stress-driven ripple effect of defect growth should be formed due to the irradiation, leading to a change in the microstructure at polycrystalline W grains.

2. Experimental procedures

The materials irradiation experiment system (MIES) described previously [13] has been used to irradiate polycrystalline W specimens. The MIES mainly consists of a radio frequency (RF) plasma source, vacuum chamber, substrate holder, and laser heating system. In this MIES, 13.56 MHz RF plasma source (450 W) with a hole about 1.0 cm in diameter was used to generate a plasma beam with species of H^+ , H_2^+ , and H_3^+ . The H_2 pressure in the RF plasma source is fixed at 14 Pa. For these irradiation experiments, the ion beam bombards the W specimen at normal incidence. The negative bias applied onto the W specimen efficiently repels the electrons, and accelerates the H_x^+ ($x = 1–3$) ions. The ion current to the W specimen is about 10 mA, corresponding to a H_x^+ ($x = 1–3$) flux of $\sim 1.0 \times 10^{20}$ ions/ $\text{m}^2 \text{ s}$. The energy (E) of H_x^+ ($x = 1–3$) ions bombarding W specimen is adjustable when the W specimen placed on a sample holder is negatively biased in the range of 0 to –500 V. Thus, E is changed from 20 to 520 eV when taking into account the plasma potential of 20 V. The H_x^+ ($x = 1–3$) fluence is varied from 5.0×10^{23} to $1.0 \times 10^{25}/\text{m}^2$. The detailed irradiation conditions are listed in Table 1. A variable-power semiconductor laser is used to heat the backside of W specimen during the ion irradiation. The surface temperature (T) of W specimens measured

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Table 1

The detailed irradiation conditions of polycrystalline W by MIES.

Specimen	Bias (V)	H _x ⁺ energy (eV)	W surface temperature (T) (K)	Fluence (ions/m ²)
01	100	120	873	$5.0 \times 10^{23}/\text{m}^2$
02	100	120	873	$1.0 \times 10^{25}/\text{m}^2$
11	0	20	373	$1.0 \times 10^{24}/\text{m}^2$
12	100	120	373	$1.0 \times 10^{24}/\text{m}^2$
13	300	320	373	$1.0 \times 10^{24}/\text{m}^2$
14	500	520	373	$1.0 \times 10^{24}/\text{m}^2$
21	0	20	673	$1.0 \times 10^{24}/\text{m}^2$
22	100	120	673	$1.0 \times 10^{24}/\text{m}^2$
23	300	320	673	$1.0 \times 10^{24}/\text{m}^2$
24	500	520	673	$1.0 \times 10^{24}/\text{m}^2$
31	0	20	873	$1.0 \times 10^{24}/\text{m}^2$
32	100	120	873	$1.0 \times 10^{24}/\text{m}^2$
33	300	320	873	$1.0 \times 10^{24}/\text{m}^2$
34	500	520	873	$1.0 \times 10^{24}/\text{m}^2$
41	0	20	1073	$1.0 \times 10^{24}/\text{m}^2$
42	100	120	1073	$1.0 \times 10^{24}/\text{m}^2$
43	300	320	1073	$1.0 \times 10^{24}/\text{m}^2$
44	500	520	1073	$1.0 \times 10^{24}/\text{m}^2$

with an infrared STL-150B pyrometer is adjustable in the range of 373–1073 K (see Table 1).

Polycrystalline W (Honglu Corporation, China) with a purity of 99.99 at.% was used as irradiated specimens. The specimens were cut into pieces with a dimension of $10 \times 10 \times 2$ mm. The surfaces of W specimens were mechanically mirror-polished to a surface RMS roughness of $<0.1 \mu\text{m}$. W specimens were annealed at 1273 ± 20 K for 2 h in vacuum with a background pressure of 10^{-5} Pa to relieve internal stresses and reduce the large concentration of nanometer-sized defects.

CAFM has been widely utilized to detect the nanometer-sized defects in GaN films [14,15], study the dielectric degradation of ultrathin silicon oxide layers and other high-k films [16–18], and image the electric properties of quantum dots [19]. In our previous studies, CAFM (Veeco DI 3100) has been used to detect the nanometer-sized defects of He⁺-irradiated hydrocarbon films [20], single-crystalline 6H-SiC [21] and polycrystalline W materials [13]. The CAFM technique is nondestructive, and it does not make any damage to the irradiated materials. In the CAFM method, one laser system is used to keep the constant deflection of the

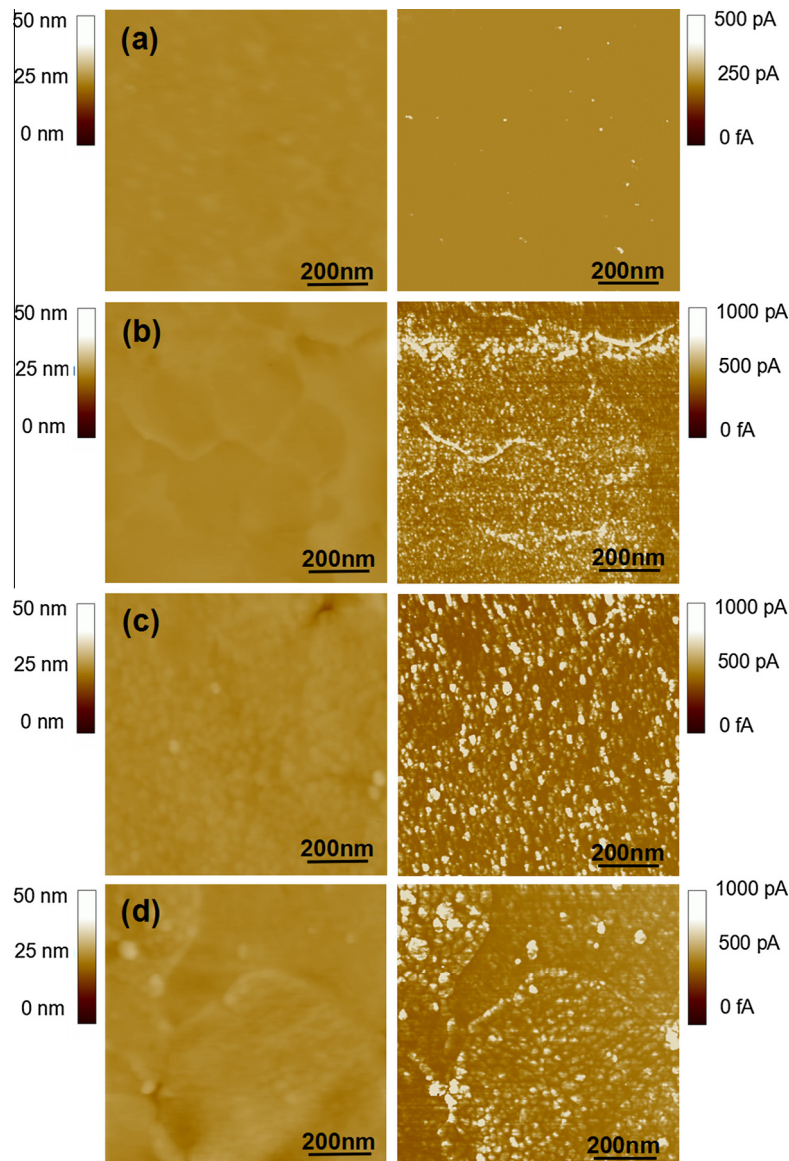


Fig. 1. Surface topography (left) and simultaneously measured current images (right) of W specimens irradiated at the H_x⁺ ($x = 1-3$) fluence of (a) 0, (b) $5.0 \times 10^{23}/\text{m}^2$, (c) $1.0 \times 10^{24}/\text{m}^2$, and (d) $1.0 \times 10^{25}/\text{m}^2$. These irradiations were performed at $T = 873$ K and $E = 120$ eV.

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