ARTICLE IN PRESS
JID: NME [m5G;June 24, 2016;14:50]

Nuclear Materials and Energy 000 (2016) 1–8

Contents lists available at ScienceDirect

Nuclear Materials and Energy

journal homepage: www.elsevier.com/locate/nme

Evaluation of helium effect on ion-irradiation hardening in pure tungsten by nano-indentation method

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ARTICLE INFO

Article history:
Received 25 November 2015
Revised 7 June 2016
Accepted 14 June 2016
Available online xxx

Keywords:
Tungsten (W)
Nano-indentation
Size effect
Helium effect
Irradiation hardening

ABSTRACT

As-received and recrystallized pure tungsten (W) were irradiated with 6.4 MeV Fe^{3+} up to 2 dpa with or without He^{+} at 300 °C, 500 °C, 700 °C and 1000 °C respectively. Irradiation hardening was measured by the nano-indentation method. An equation to evaluate the bulk equivalent hardness was derived on the assumption that the geometrically necessary dislocation (GND) densities at an indentation depth were the same before and after irradiation. Ion-irradiation always induces hardening in both as-received and recrystallized W at all the experiment temperatures. In the case of single-beam irradiation, the recrystallized W exhibited higher hardening than as-received one. The effect of helium on the irradiation hardening is dependent on the material condition: as-received W showed an additional hardening by helium at all the irradiation temperatures, while in recrystallized W the hardening was not affected by helium below 700 °C.

1. Introduction

Tungsten was selected as the armor material for ITER diverter and has been considered as a candidate first-wall material for DEMO-like reactors. Since the armor and first wall will receive both high neutron irradiation and high heat flux, researches on irradiation effects on W at a wide variety of temperatures have been conducted so far: microstructure evolution under self-ion irradiation [1–3], alien heavy ion irradiation [2,4,5] and helium [6–8] or deuterium-ion implantations [9]. Special attention was paid to the recrystallization effects on the formation of irradiation-induced microstructures as recrystallization may occur in the materials after suffering high heat load during operation. As for neutron irradiation effect, Fukuda et al. reported irradiation hardening up to 0.47 dpa was highest at 583 °C in the irradiation temperature range from 531 °C to 756 °C [10]. They also reported that recrystallized W showed a larger hardening than the stress relieved (as-received) W after neutron irradiation at 600 °C and 800 °C [11]. However, irradiation data of W is still insufficient especially combined with detailed characterization of the irradiation-induced microstructures which are strongly correlated to the mechanical properties.

In the previous studies on ion-irradiation effects in W [4,12–14], nano-indentation method was adopted to measure the ion-irradiation hardening because of the limitation of the damaged depth to less than several microns. Himei et al. reported that helium in the dual-beam irradiation will enhance the growth of dislocation loops and cavities and therefore increase hardening [4]. Because the ion-irradiated material has a depth dependent profile of displacement damages, the radiation damage microstructures are heterogeneously distributed, which should be taken into account when evaluating ion-irradiation hardening by means of nano-indentation method.

Although the nano-indentation method is effective to measure the ion-irradiation hardening, there is a technical issue of size effect [15] introduced by Nix-Gao model. The size effect is sometimes described by “the smaller, the harder”, which means when indented in a shallow depth, the hardness will be larger than in deeper depth.

The equation derived from the Nix-Gao model is written as:

$$\left(\frac{H}{H_0}\right)^2 = 1 + \frac{h^*}{h} \tag{1}$$

Where H_0 stands for the original hardness of the material or “bulk equivalent hardness”. H is the hardness measured at a depth

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http://dx.doi.org/10.1016/j.nme.2016.06.010

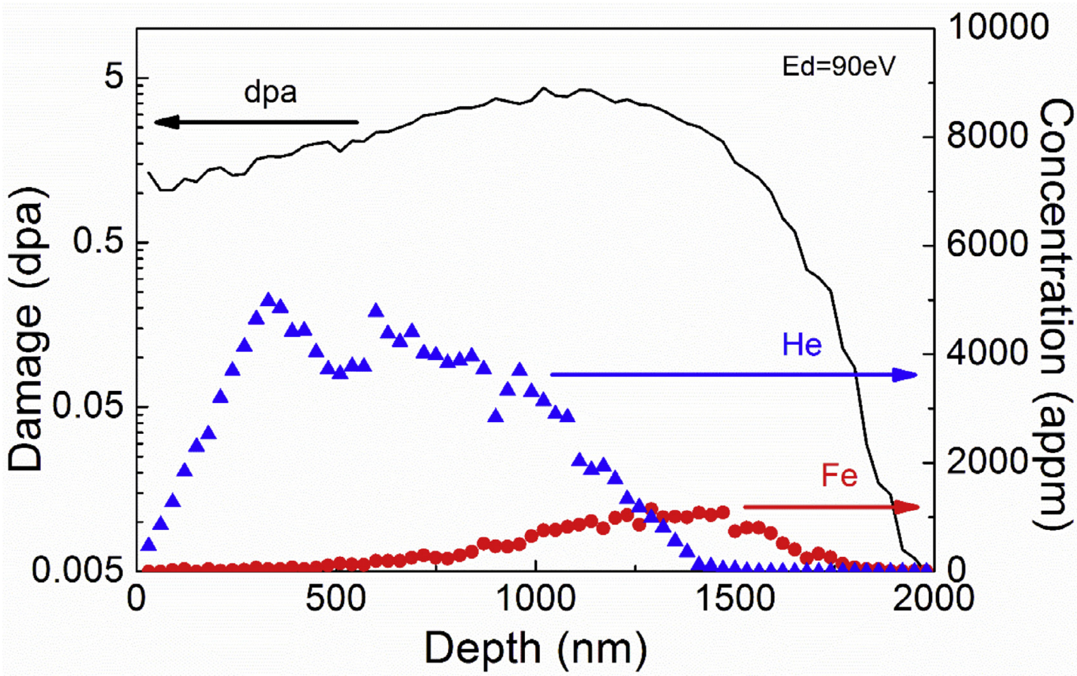

Fig. 1. Depth profiles of damage, He concentration and Fe concentration calculated by SRIM.

Table 1
Chemical composition of as-received tungsten sheet (μg/g).

W	Mo	Fe	Cr	C	Ta	P	O	Si	N	S	Nb	Al
matrix	<100	<30	<20	<30	<20	<20	<20	<20	<5	<5	<10	<15

h, and h^* is a characteristic length. Kasada et al. applied this model to evaluate ion-irradiation hardening, where H_0 was the intercept given by the linear relation between inverse depth (h^{-1}) and hardness (H) in the irradiated area [16,17]. However, in tungsten, the Nix-Gao plots never draw a straight line, even before ion-irradiation. The shortcomings from the application of Nix-Gao model were discussed by Pharr et al.[18], one speculation of which is in the shallow area the model would overestimate the density of GNDs. Huang et al. [19] modified the model with the assumption that there is a saturation of GND density in the shallow area. An EBSD observation suggested the GNDs might transform into subgrain boundaries.As a result, the size effect will be induced by not only GNDs but also multiplication of grain boundaries [20]. The TEM observation of the cross-section of indented region by Katoh et al. [21] revealed that in ferrous alloys the GND affected zone strongly depended on the indentation depth: the GND zone was anisotropic when the indentation depth is less than 100 nm, but it was quite semispherical when exceeding 300 nm. These phenomena indicate that Nix-Gao model does not fit at a shallow area but fits well in deep area. As a result, the bulk-equivalent hardness of the unirradiated specimen could still be estimated by Nix-Gao plots of the deeper area. The hardness of the irradiated layer in the shallow area needs a more adequate evaluation method.

In this research, we investigated the effect of helium on the ion-irradiation hardening of W with and without recrystallization at a wide range of irradiation temperatures by means of nano-indentation method.

2. Experimental procedure

The material used in this research was commercial 99.95 wt% pure rolled W from Nilaco Co. Ltd, denoted as “as-received”, with an average grain size of 1.7 μm. The impurity concentrations are listed in Table 1. Recrystallized W was obtained from as-received one annealed at 1400 °C for 1 h, with an average grain size of 27 μm. The specimens for ion-irradiation experiments were mechanically polished with SiC emery papers of #500~4000 and buff-polished with diamond pastes until a powder diameter of 0.25 μm. Finally the surface was electrolytically polished with a 1% NaOH aqueous solution at a constant voltage of 20 V at ambient temperature.

Ion-irradiations were performed using a dual ion-beam accelerator in Kyoto University, DuET, which could yield a beam of 6.4 MeV Fe^{3+} ions and an energy-degraded beam from 1 MeV He^+ ions simultaneously. For dual ion-beam irradiation, an energy degrader was set up to get a rather homogeneous injection of He^+ which resulted in a total He concentration of 3000 appm in the range of 1.5 μm depth. The irradiation temperatures were 300 °C, 500 °C, 700 °C and 1000 °C. Fig. 1 shows the depth profile of damage and the concentration of implanted elements, which were simulated by SRIM software [22] and calculated following the “Energy damage” method introduced by Stoller et al. [23]. The threshold energy was selected as 90 eV [24]. The averaged dpa of the 2 μm irradiation area is about 2 dpa.

Nano-indentation hardness was measured by Agilent Technologies Inc. Model Nano Indenter G200 with a Berkovich tip. CSM method was applied with the area function calibrated on standard fused silica by Oliver and Pharr method [25]. The nominal strain rate was 0.05 /s and the oscillations set as 2 nm. The maximum penetrating depth was 2000 nm. Testing temperature was controlled to 25 ± 4°C. For an ideal Berkovich tip, the area-function, A, is defined as $A = 24.56\,h^2$, where h is indentation depth. Since nano-indentation hardness is remarkably affected by A, the tip blunting that alters h, should be considered. The area-function