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Crystal orientation dependence of ion-irradiation hardening in pure tungsten

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ABSTRACT

Pure tungsten (W) single crystals of {001} and {011} surface orientations were irradiated with 6.4 MeV Fe^{3+} ions up to 1 dpa at 573 K. The TEM examination revealed that there was a very small orientation dependence in the radiation damaged microstructure, showing that both W{001} and W{011} exhibited a double black band structure with high number density of dislocation loop rafts in the black bands. However, the depth profile of ion-irradiation hardening evaluated by nanoindentation (NI) technique turned out to show a clear orientation dependence, namely, W{001} showed a deeper NI hardness profile than W{011}.

1. Introduction

Tungsten (W) is regarded as the plasma facing material in fusion power plants. In order to investigate the expected irradiation effects, ion-irradiation is intensively used to study irradiation effects on materials. The existing studies on ion-irradiation effects on W mainly focus on polycrystalline W neglecting grain orientation dependence on the results. Only a few studies consider the crystal orientation in pure W when investigating the ion-irradiation effects, such as [1–5]. For example, Liu et al. [4] investigated the effect of low flux helium and hydrogen ion irradiation on W polycrystal with large size of grains whose grain orientations were near {011}, {101} and {111} planes on the surface, and found that the helium-induced blistering depends strongly on the grain orientation. The most resistant orientation to surface morphology changes by the irradiations was near {001} plane [4].

Generally, the irradiation with several MeV of heavy ions induces the formation of radiation damage structures in the limited zone that is within a few microns from the surface. Grzonka et al. [5] carried out self-ion irradiation at room temperature on W polycrystal with an average dose level of 2.3 and 6.36 dpa. They found by TEM observation that the damaged zone inside a [110] grain extended deeper compared to the adjacent [012] grain at 2.3 dpa, while they found no orientation dependence in adjacent grains after irradiation up to 6.36 dpa. Although they considered that channeling and/or grain boundary effect that is the interaction of dislocation with grain boundaries within the area of damage zone could be the reason for the difference in the detected damage depth, no clear experimental evidence has been obtained.

Three of co-authors reported recently that the distribution of defect structures at 1273 K extended to deeper area than SRIM code calculation result of which the mechanism was attributed for vacancy migration at such a high irradiation temperature [6].

Our objective is to investigate the potential effect of crystal orientation on the microstructural change and hardening evolution in ion-irradiated W without considering grain boundary effects.

2. Material and methods

As a starting material, a W single crystal of 99.7% purity was used in this study. From the W single crystal, small specimens with the surface orientations of {001} and {011} were sampled and denoted as W{001} and W{011}, respectively. The exact surface orientation of the specimen was measured by electron backscatter diffraction patterns method. More details on the sample preparation can be found in the previous work [7].

The specimens were irradiated using an accelerator, DuET, at Kyoto University with 6.4 MeV Fe^{3+} ions up to a nominal damage level of 1 dpa at 573 K. The SRIM code [8] results are shown in Fig. 1. The nominal dpa is defined as the displacement damage occurring at the depth of 600 nm and the threshold energy of W is selected to be 90 eV. As shown in Fig. 1, the ion-irradiation induced damaged zone reaches up to 1800 nm. After preparation

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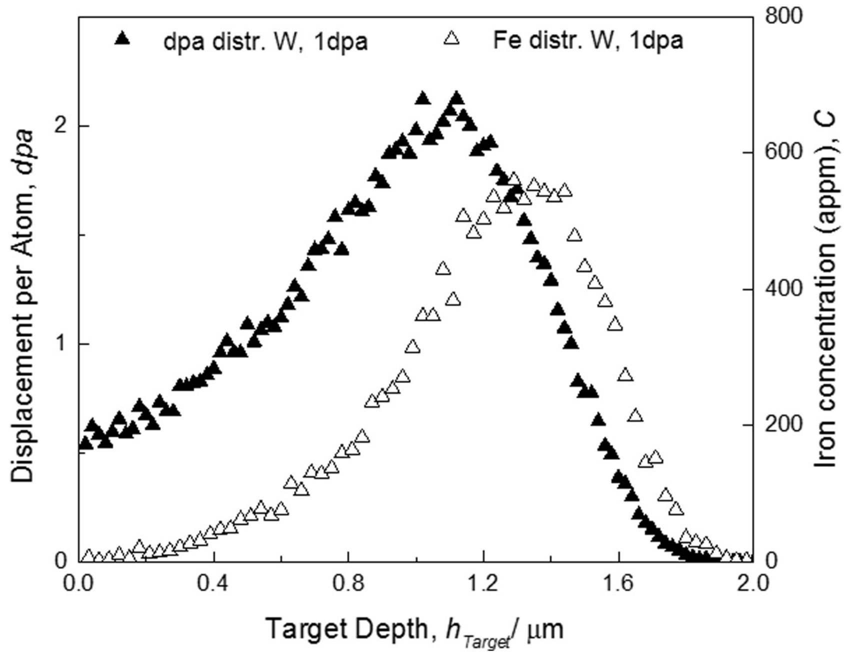

Fig. 1. Damage and iron distribution in W.

of thin foils by focused ion beam apparatus (HITACHI FB2200) and flashing electrolytic polishing, TEM microstructures were examined by JEOL JEM-2010 at 200 kV.

The hardness was measured by a nanoindenter (Agilent Technologies Inc. Model NanoIndenter G200) using a Berkovich tip before and after ion-irradiation. The indenter has been positioned in a way that one edge of the Berkovich triangle was perpendicular to a $\langle 1\,1\,1 \rangle$ orientation. Constant strain rate (CSR) test method including continuous stiffness method (CSM) was adopted, which allows a continuous detection of hardness H and indentation modulus E_{IT} over indentation depth h and is suitable to obtain a whole H-h profile. The testing conditions were as follows: the maximum contact depth was 2000 nm, the nominal strain rate was 0.05 s^{-1}, the oscillation amplitude was 1 nm and the testing temperature was (299 ± 2) K. For each testing condition, 10 tests were carried out within an uncertainty of ±0.2 GPa.

3. Results and discussion

In Fig. 2, the TEM microstructures of irradiated a) W{001} and b) W{011} single crystals are shown. The radiation damaged zone appears to be a bit deeper than the prediction by SRIM code in both orientations. It should be noticed that a slight crystal orientation dependence of the final damaged depth is visible: W{001} shows 2400 nm compared to 2200 nm in W{011}. In both specimens, a double black band structure with a high number density of loop rafts was observed. The upper boundary of the shallower black band is located at around the same depth, 700 nm, in both specimens. The upper and lower boundaries of the two black bands are marked by white lines in Fig. 2. As for the type of defects, both orientations show dislocation loop clusters, mostly formed to rafts, as the major defect type.

Fig. 3 shows the H-h profiles for CSR tests of unirradiated and irradiated specimens, indicating an unexpected significant difference between the profiles of irradiated W{001} and W{011}, in contrast to the results in TEM microstructural observation which showed rather small difference in damage depth. The characteristic hardness shoulders at the depth h_{crit} [9,10] are observed in both of the profiles. The so called critical indentation depth h_{crit} is larger in the case of W{001} than in the case of W{011}: around 500 nm and 400 nm, respectively. But generally, both orientations show a high irradiation hardening behaviour by 1dpa irradiation at 573 K.

According to Kasada et al. [9] for ion-irradiated materials and originally denoted by Manika et al. [10] for film on substrate

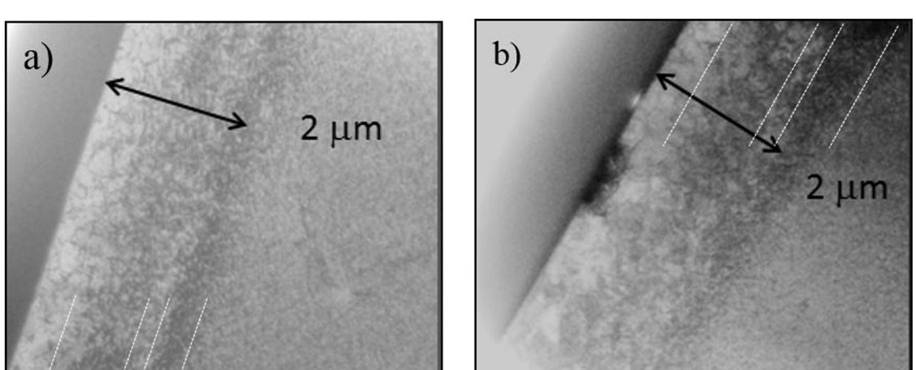

Fig. 2. TEM microstructural overview of a) W{011} and b) W{001}. (The two black band positions are marked with white lines).

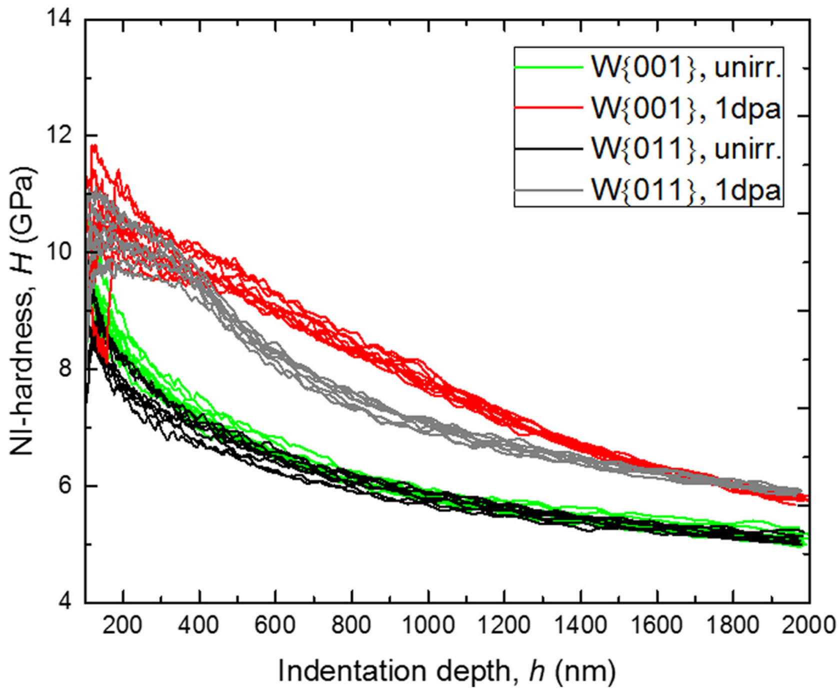

Fig. 3. NI-hardness – indentation depth (H – h) profiles of unirradiated and irradiated W{001} and W{011}.

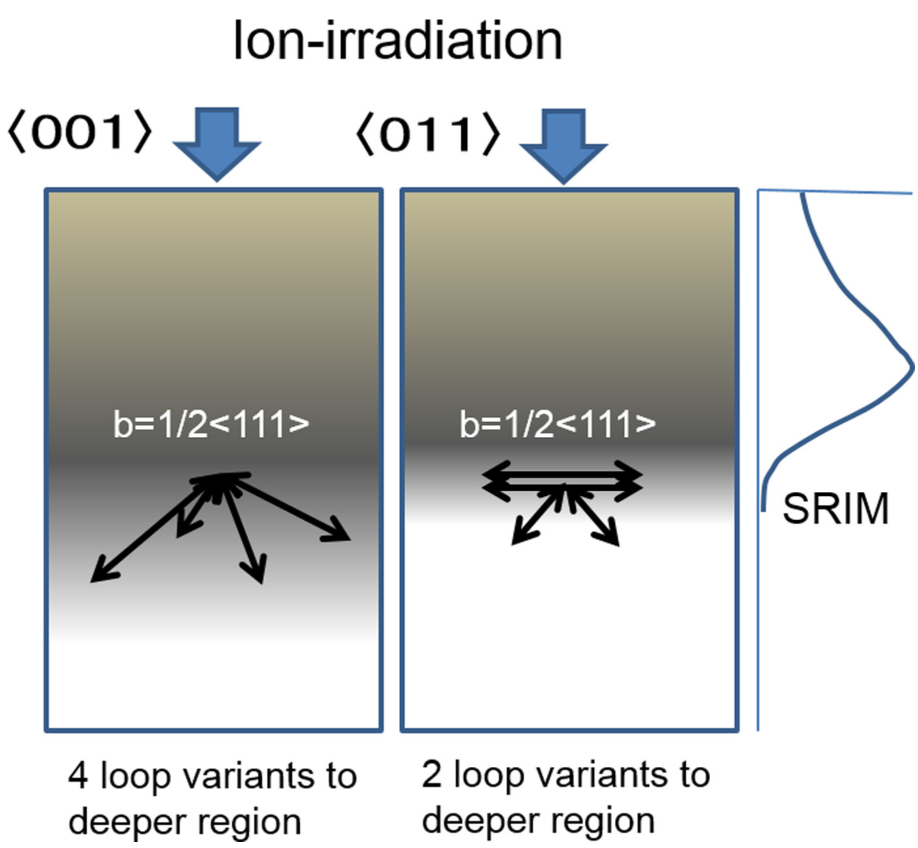

Fig. 4. A possible interpretation of hardness profiles of W{001} and W{011}. 1D motion (arrows in the figure indicate the possible direction of 1D motion) of very fine dislocation loops with b = 1/2⟨1 1 1⟩ which are invisible by TEM but effective for hardening.

systems, the hardness shoulder depth, h_{crit}, is defined as the indentation depth where the NI hardness start to reflect the hardness of the unirradiated region below the irradiated one. For example in Fe-1wt.%Cu and Fe-1.4wt.%Mn, Fe^{3+} irradiated up to 1 dpa at 563 K, the hardness shoulder was at around 500 nm and 300 nm of indentation depth, respectively [9]. In polycrystalline pure W Fe^{3+} irradiated to 2 dpa at 573 K to 1273 K the hardness shoulder was at around 300–350 nm [11]. In our previous material pile-up formation study [12] including basic hardness testing method according to ISO standards [13] on unirradiated and irradiated W{001}, we reported a hardness shoulder for 1 dpa W{001} at around 400–500 nm and mentioned a possible existence of a deeper one somewhere between 650–950 nm. The statement was vague because of the used testing procedure of basic hardness method, which serves a rather scattered *H-h* profile. In this study here, by using CSR testing method, which allows a continuous measurement of hardness over indentation depth by a single indentation, and subsequently by analyzing the *H-h* profile of irradiated W{001} and irradiated W{011} by Nix and Gao's [14] interpretation of the depth dependence of hardness, i.e. by a $H^2 - 1/h$ profile, we can confirm that irradiated W{001} shows a second hardness shoulder at an indentation depth of around 1000 nm. The exact reason is unclear at this moment, but may be attributed to the deeper black band.

A double black band structure can be interpreted on the bases of SRIM code calculation results indicating two different depth profiles of lattice damage structures and implanted Fe^{3+} ions. The shallower black bands may consist of lattice damage structures such as self-interstitial loops and/or vacancy clusters, while the deeper one may consists of Fe interstitial loops.

As for the orientation dependence of the NI hardness profile, the mechanism is not clear. Since the TEM microstructure appears to be independent of crystal orientation, it is difficult to explain the orientation dependence in terms of the difference in the damaged depth in each single crystal visible by TEM observation. Although more studies are necessary, a possible interpretation of the crystal orientation dependence can be addressed to the 1D motion of very fine interstitial loops which are invisible by TEM but effective for the hardening. The longer distance of the 1D motion of interstitial loops in W{001} than in W{011} is considered to be due to the difference in the geometrical orientation of the direction of the 1D motion along the direction parallel to the Burger's vector, ***b***, of the loops. According to Arakawa et al. [15], the mobile loops by thermal diffusion are those almost parallel to the surfaces of which the ***b*** is 1/2[1–11] and 1/2[11–1] when the surfaces of the TEM thin foils were set at almost (011). As shown in Fig. 4, in the case of ion-irradiation on W(011), the penetration depth of loops beyond SRIM code calculation can't be possible for 2 variants of $1/2\langle 1\,1\,1\rangle$. However, in the case of W(001), the mobile loops can be 4 variants which have a ***b*** of 1/2[1 1 1], 1/2[-1 1 1], 1/2[1–11] or 1/2[11–1], where the force applied onto these loops along their direction of motion from the surfaces is rather small. Here, the penetration depth of the 1D motion of dislocation loops becomes deeper than the SRIM code calculation results showing 2 times more dislocation loops in deeper region in W(001) than in W(011). More systematic irradiation experiments and detailed microstructural examinations are necessary in the future.

4. Conclusions

In order to investigate the effect of crystal surface orientation on the irradiation hardening of pure W, single crystals of W{001} and W{011} were irradiated with 6.4 MeV Fe^{3+} ions up to 1 dpa at 573 K. The TEM examinations revealed that there is a small orientation dependence in the radiation damaged structure, showing that 1 dpa W{001} exhibits deeper damaged structure and that both 1dpa W{001} and 1 dpa W{011} exhibited a double black band structure with high number density of dislocation loop rafts in the black bands. Even though the change in the damaged depth between the different orientations of single crystals was rather small, the ion-irradiation hardening behaviour evaluated by NI technique turned out to be clearly orientation dependent, namely, W{001} showed a deeper NI hardness profile than W{011}.

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