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Evaluation of multi-layered hardness in ion-irradiated stainless steel by nano-indentation technique

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ABSTRACT

Depth dependence of hardness in ion-irradiated 316 stainless steel was evaluated by the sectioning of damaged region and subsequent nano-indentation. The range of plastically deformed region by the nano-indentation was supplementarily investigated by transmission electron microscopy. When the indentation depth was the critical indentation depth, h_c, which was derived from the Nix–Gao plot, the dislocation structure was observed from the specimen surface to right below the bottom of the ion-irradiated region. To verify the depth dependence of hardness, the "multi-layer model" was introduced in this study. The multi-layer model is based on the following assumptions; (1) the ion-irradiated region can be divided into sub-layers having their own local hardness, H_L; (2) the hardness can be the product of f and H_L in each sub-layer where f is the volume fraction of a deformation zone; and (3) the deformation zone can be a hemisphere. Eventually, through the sectioning and following nano-indentation, H_L in each sub-layer was experimentally evaluated. Further the correlation between the displacement damage and the irradiation hardening ΔH in this study agreed with that of neutron irradiation experiments.

1. Introduction

Nano-indentation technique has been used to evaluate an irradiation hardening in ion-irradiated materials for simulating the change of mechanical properties under fission and fusion reactor conditions. The available irradiated volume created by ion irradiation, however, is small and limited from specimen surface. Additionally there is a damage gradient in the ion-irradiated volume. These features provide many scientific and technical challenges such as a hardness–depth profile, a softer substrate effect and an indentation size effect [1–4]. To utilize the hardness data of the ion-irradiated material obtained by nano-indentation in view of engineering aspects, the analyzing method needs to be developed further.

In this study, the depth dependence of the hardness in the ion-irradiated material was evaluated by means of sectioning and nano-indentation. Firstly, in order to obtain the relationship between the penetration depth and the range of deformed zone induced by an indent, the hardness–depth profile was evaluated and microstructure were examined by transmission electron microscopy (TEM). Then the ion-irradiated region of the specimen was sectioned and nano-indentation were carried out. The results were analyzed based on the "multi-layer model" as mentioned below.

2. Experimental

A 316 stainless steel was used in this study. Coupon type specimens with the size of 1 mm thickness, 6 mm length, and 3 mm width were solution-annealed at 1473 K for 15 min. Prior to irradiation, the irradiation surface was mechanically polished using alumina suspension with a particle size of 0.3 μm. To remove any deformation layer induced during mechanical polishing, the specimen surface was finished by electro-polishing.

Ion irradiation was carried out at the TIARA facility of JAEA with 10.5 MeV Fe^{3+} ions at the temperature of 523 K. The nominal displacement damage was up to 0.1–10 dpa at the depth of 1 μm from the specimen surface. The displacement damage reached to 2.5 μm depth and the peak of damage was at 2.1 μm depth based on SRIM calculation [5].

Nano-indentation test was carried out by using an Elionix ENT-1100a with a triangular pyramidal diamond indenter (Berkovich type tip). To gain the hardness–depth profile of the specimen, depth-control indentations were performed with a penetration depth of 50–800 nm with 10 nm interval. Average hardness from 3 indentations was obtained at each penetration depth. Therefore, each hardness–depth profile in this study contained information from more than 225 indents. To avoid overlapping of the plastically deformed zones, the indents were performed at intervals of 30 μm between the indents in the specimen surface.

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TEM investigation was performed to evaluate the range of the plastically deformed zone. For this purpose, cross-sectional TEM specimens through an indent were prepared by using focused ion beam (FIB) machine (JEOL JIB-4600F). To protect the imprint against Ga^+ ion damage, the specimen surface was covered by carbon deposition layer. Prior to the deposition of the carbon layer, the center of the imprint was marked to get the cross section right through the middle of the imprint.

The damage layer was sectioned to the depth of 0.6, 1.2, and 1.8 μm from the specimen surface by using FIB. Ga^+ beam of FIB was bombarded parallel to the specimen surface. The size of the sectioned area was about $40 \times 100\ \mu m^2$. Nano-indentation was carried out to "new surface" produced by the sectioning. Because of the limited size of the sectioned area, the distances between each of the imprint was 10 times penetration depth for each indentation.

3. Result and discussion

3.1. Hardness–depth profile and deformation zone in ion-irradiated region

Fig. 1 shows the plots of the square of hardness against the reciprocal indentation depth. The hardness was determined in the manner outlined by Oliver and Pharr [6]. The relationship between the square of the hardness and the reciprocal indentation depth showed an inflection for the irradiated materials. This inflection is due to the possible effect of softer substrate region beyond the irradiated region, which was discussed within the framework of the studies by Nix and Gao [7] and Kasada et al. [1]. Kasada et al. [1] showed that the Nix–Gao plot of the ion-irradiated material has an inflection at a critical indentation depth, h_c, and the contribution of bottom softer substrate beneath the indent is related to the hardness where the penetration depth is deeper than h_c. In Fig. 1, h_c appeared to depend on the displacement damage, and were obtained to be 330 μm for 0.1 dpa specimen, and 440 μm for 1.0 and 10 dpa specimen.

Cross-sectional TEM was performed to observe the plastically deformed zone which was induced by an indent. Fig. 2a–d shows the experimentally observed deformation zone of the unirradiated specimen and the irradiated specimen up to 10 dpa with indentation depth of h_c. For unirradiated specimen (Fig. 2a), the plastic deformation (mainly the dislocation structure), introduced by the

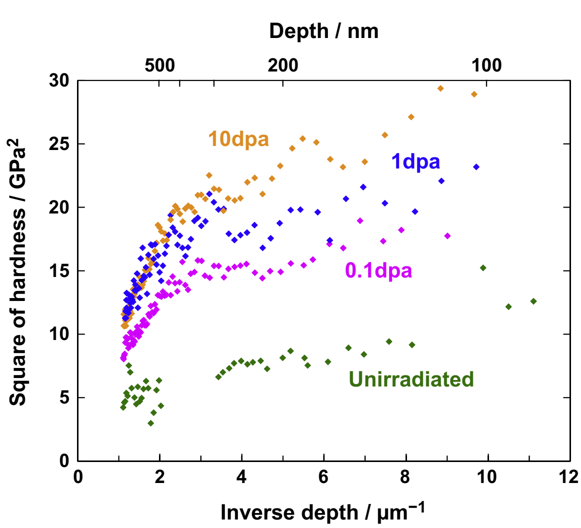

Fig. 1. Relationship between the square of the hardness and the reciprocal indentation depth for the specimens irradiated up to 0, 0.1, 1.0, and 10 dpa at 523 K.

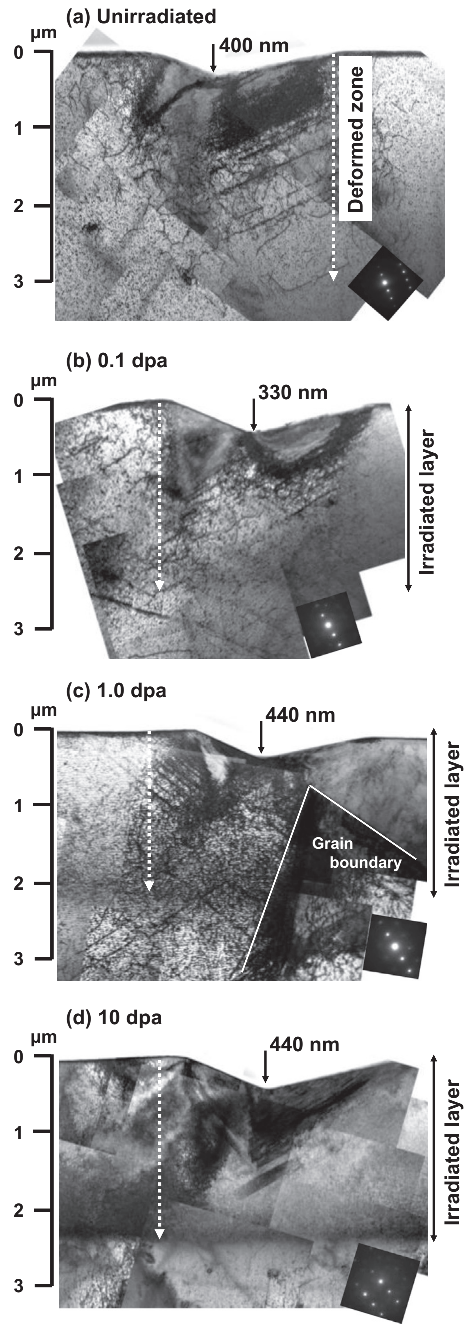

Fig. 2. Deformation zone (indicated by the white arrow) of (a) the unirradiated specimen and the irradiated specimen up to (b) 0.1, (c) 1.0, and (d) 10 dpa where the indentation depth is h_c.

indent with penetration depth of 400 nm, reached to 3.0 μm from the specimen surface. Note that the observed black dots in Fig. 2 are artifacts induced by Ga^+ ion beam. For Fig. 2b–d, prior to discussing the deformation zone, the distribution of defect clusters introduced by Fe^{3+} ion irradiation must be mentioned. As seen in Fig. 2b–d, the defect clusters were distributed in the region from the surface to 2.5 μm depth. Especially the 10 dpa specimen clearly exhibited the defect cluster distribution and the area with the maximum number density of the defect cluster appeared to be at 2.3 μm depth. The plastically deformed zone produced by the indent where the indentation depth is h_c was examined with respect to its range in irradiated specimens. In Fig. 2b–d, the deformed zone appeared to extend to the peak damage region in