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Evaluation of irradiation hardening of proton irradiated stainless steels by nanoindentation

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ABSTRACT

Ion irradiation experiments are useful for investigating irradiation damage. However, estimating the irradiation hardening of ion-irradiated materials is challenging because of the shallow damage induced region. Therefore, the purpose of this study is to prove usefulness of nanoindentation technique for estimation of irradiation hardening for ion-irradiated materials. SUS316L austenitic stainless steel was used and it was irradiated by 1 MeV H^+ ions to a nominal displacement damage of 0.1, 0.3, 1, and 8 dpa at 573 K. The irradiation hardness of the irradiated specimens were measured and analyzed by Nix–Gao model. The indentation size effect was observed in both unirradiated and irradiated specimens. The hardness of the irradiated specimens changed significantly at certain indentation depths. The depth at which the hardness varied indicated that the region deformed by the indenter had reached the boundary between the irradiated and unirradiated regions. The hardness of the irradiated region was proportional to the inverse of the indentation depth in the Nix–Gao plot. The bulk hardness of the irradiated region, H_0, estimated by the Nix–Gao plot and Vickers hardness were found to be related to each other, and the relationship could be described by the equation, $HV = 0.76H_0$. Thus, the nanoindentation technique demonstrated in this study is valuable for measuring irradiation hardening in ion-irradiated materials.

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1. Introduction

Stainless steels are essential industrial materials, which show excellent features such as strength, ductility, corrosion resistance, weldability, and workability. Of course, stainless steels are also used as nuclear materials in many components of fusion and fission reactors. Nuclear materials are used under neutron irradiation, and that is the unique points against other industrial materials. Various phenomena such as irradiation assisted stress corrosion cracking (IASCC) and swelling occur in irradiated stainless steel [1–4], and the precise elucidation of the mechanisms and the prediction of such degradations are essential.

The degradation caused by neutron irradiation is attributed to microstructural changes. Under irradiation, many point defects such as interstitial atoms as well as vacancies and their clusters are produced by the collision cascade. The point defects themselves and the interaction among the point defects and the alloying element cause microstructural changes [5–7]. These microstructural changes depend on irradiation conditions such as the irradiation flux, irradiation temperature, and by the chemical composition and lattice structure of the target materials. Therefore, to elucidate the mechanisms of degradation, it is necessary to investigate the microstructural changes of various materials under various irradiation conditions. Neutron irradiation experiments are typically of long durations, which involve irradiation of materials, cooling down of radioactivity, unloading specimens from the reactor core, and post-irradiation experimentation (PIE) under radiation control. Furthermore, in these experiments, the handling of the irradiated specimens is a challenge because of the high radioactivity. In contrast, ion irradiation experiments involve short irradiation period because of the high damage rate and cause less or no activation and transmutation effects depending on mass and energy of accelerated ions, which eases specimen handling during PIE. In addition, the irradiation conditions such as the irradiation dose, irradiation flux, and irradiation temperature can be controlled more precisely and more flexibly in the case of ion irradiation experiments than in neutron irradiation experiments. Hence, ion irradiation experiments are useful for investigating the mechanisms of irradiation damage.

However, ion irradiation also has some demerits such as limited beam penetration depth. Hence, the irradiated area obtained is limited to the surface region of the irradiated specimens, which is in the micrometer scales. Therefore, to assess the irradiated area, micromechanical testing techniques are required. Nanoindentation techniques have been used for the evaluation of irradiation hardening [8,9]. However, in the case of hardness measurement in

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the micrometer scales of indentation depth, indentation size effect (ISE), which is a unique phenomenon involving an increase in the measured hardness with decrease in the indentation depth, is observed [10]. The ISE is described using the geometrically necessary dislocation theory proposed by Nix and Gao [11]. Some studies evaluate the irradiation hardening using the Nix–Gao model [12]. Recently, Kasada et al. developed a process to evaluate irradiation hardening by the Nix–Gao model using Fe-based alloys [13].

In the present study, to prove usefulness of nanoindentation technique for evaluation of irradiation hardening of ion irradiated materials, irradiation hardening of proton irradiated stainless steels were investigated by nanoindentation. Moreover, the irradiation hardening estimated by Nix–Gao model was compared with Vickers hardness and the relationship between nanoindentation hardness and Vickers hardness was discussed.

2. Experimental

SUS316L stainless steel, with the chemical composition shown in Table 1, was the material used in the present study. Disk shaped specimens with a diameter of 3 mm and thickness of 0.2 mm were punched out. The disk shaped specimens were annealed at 1323 K for 0.5 h in vacuum and were quenched in iced water. The average grain diameter after heat treatment was about 30 μm.

The specimens were irradiated using the Dynamitron accelerator at Tohoku University [14]. Irradiation was carried out with 1 MeV H^+ ions to a nominal displacement damage of 0.1, 0.3, 1, and 8 dpa at a nominal rate of 3.5×10^{-6} dpa/s. The typical depth profile of the displacement damage calculated by the SRIM code is shown in Fig. 1 [15]. The nominal dpa is defined as the average displacement damage at a depth ranging from 1 to 4 μm. The irradiation temperature was measured by thermography and controlled at 573 K within ±10 K.

To investigate the irradiation hardening, a nanoindentation test was conducted using a nanoindenter (Agilent Technologies Inc. Model NanoIndenter G200) with a Berkovich type indentation tip. The indentation was performed on the irradiation surface in the direction parallel to the incident H^+ beam. The constant stiffness measurement (CSM) technique was used to obtain the depth profile of hardness [16]. The test temperature was 298 K and the nominal strain rate was 0.05 s^{-1}. To eliminate the effects of grain orientation on measured hardness, each indentation was performed on different grains and then the standard deviation and average of hardness was also evaluated. In order to compare with the nanoindentation hardness, the Vickers hardness test was performed with a 98 mN load for 15 s.

3. Results and discussion

Fig. 2 shows the indentation depth profiles of the nanoindentation hardness of SUS316L before and after irradiation. ISE was observed for all the specimens, and the measured hardness decreased with increase in indentation depth. For the irradiated specimens, points of variation in hardness, indicated by arrows in Fig. 2(b)–(e), were observed. These points indicated that the deformation region originating from the indentation reached the boundary between the irradiated and unirradiated regions. In order to explain the normal ISE, Nix and Gao developed a model based on

Table 1
Chemical composition of SUS316L.

(wt.%)	C	Si	P	S	Cr	Mn	Ni	Mo	Fe
SUS316L	0.005	0.51	0.014	0.001	17.36	0.83	12.13	2.12	Bal.

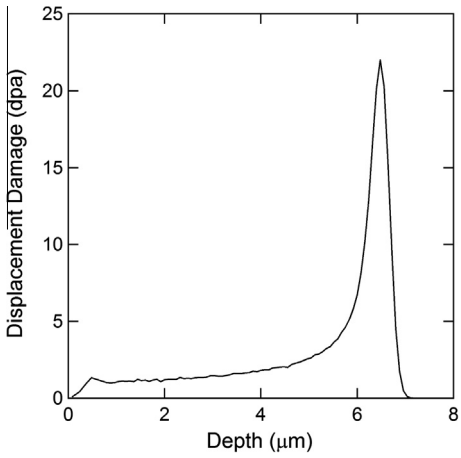

Fig. 1. Depth profiles of the displacement damage (dpa) at H^+ ion irradiation of 1 MeV.

the concept of geometrically necessary dislocation [11], which is described in the following equations:

$$\frac{H}{H_0} = \sqrt{1 + \frac{h^*}{h}} \tag{1}$$

$$h^* = \frac{81}{2} b\alpha^2 \tan^2\theta \left(\frac{\mu}{H_0}\right)^2 \tag{2}$$

In the above equations, H is the measured hardness at the depth of h, H_0 is the hardness at infinite depth (i.e., macroscopic hardness), which will be referred to as the bulk hardness hereafter, h^* is a characteristic length which depends on the material and the shape of indenter tip, b is the Burgers vectors, α is a constant, θ is the angle between the surface of the indenter and the specimen surface, and μ is the shear modulus. Using the Nix–Gao model, the hardness depth profile is given by the following equations:

$$H^2 = H_0^2 + \frac{h^{**}}{h} \tag{3}$$

$$h^{**} = \frac{81}{2} b\alpha^2 \mu^2 \tan^2\theta \tag{4}$$

In Fig. 3, the square of the nanoindentation hardness is plotted against the reciprocal of indentation depth ($1/h$) to compare with the Nix–Gao model. As expected from Eq. (3), the square of nanoindentation hardness was found to be proportional to the reciprocal of indentation depth. The unirradiated SUS316L showed a good linearity, and the bulk hardness H_0, which is the square root of the intercept value, was estimated as 1.5 GPa. However, in the case of the irradiated SUS316L samples, the plots showed linearity over an inflection point indicated by an arrow in the plot. The inflection point could be more clearly identified with increasing dpa. Fig. 4 shows the typical Nix–Gao plot of SUS316L irradiated to (a) 8 dpa and (b) 0.3 dpa. As shown in Fig. 4(a), the data over the inflection point, which is indicated by the arrow 1, could be interpreted as the hardness of the irradiated region. The bulk hardness H_0 estimated from this irradiated region was 5.3 GPa. The data below the inflection point indicated by arrow 2 included the hardness of both the irradiated and unirradiated regions. For the SUS316L sample irradiated to 0.3 dpa shown in Fig. 4(b), the data was divided into three regions. In the region indicated by arrow 1, the square of the nanoindentation hardness, H^2, was proportional to the reciprocal of the indentation depth, $1/h$. The data in this region could be interpreted as the hardness of the region irradiated to 0.3 dpa. In the region indicated by arrow 2, the hardness increased with increase in the indentation depth. The data in this region included

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