Proceedings of the Korean Nuclear Society Autumn Meeting
YongPyung, Korea, October 2003

Investigation for deformation of ion-irradiated RPV steel using nanoindentation hardness test

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Abstract

To evaluate deformation depending on depth under ion irradiation, nanoindentation test was applied to heavy ion-irradiated SA508 CL.3 RPVSs. The specimens were irradiated with 8MeV Fe^{+4} ions to 0.15dpa and 1.5dpa at below 60℃. The derivative of the load-depth ratio, *d(L/D)/dD*, was estimated the depth dependent formation of plastic and elastic deformation in the irradiated specimens. In ion-irradiated SA508 CL.3 RPVS, the peak of deformation was observed at about 20nm from the surface, but the one of radiation damage was appeared at 1500nm from the surface when TRIM98 was simulated damage depth profile. In order to study the effect of deformation depth under ion irradiation rate, we evaluated nanoindentation hardness test. The hardness was larger for the irradiated than for the non-irradiated, and also larger for 1.5dpa than for 0.15dpa one.

1. Introduction

Lifetime extension of reactor pressure vessel (RPV) requires a more refined estimation of irradiation embrittlement of reactor pressure vessel steel (RPVS) during reactor operation because irradiation embrittlement is assumed to be due to radiation hardening [1]. From such a practical point of view, irradiation-induced hardening of RPV steel is one of the important phenomena concerning the nuclear reactor safety. The hardness usually implies a resistance to deformation for metals [2]. Therefore, the measurement of the hardness profile through depth of SA508 CL.3 RPVS is a good method to study deformation of irradiation induced in RPV steel.

In the present study, the irradiation hardening of SA508 CL.3 RPVS has been

measured after ion irradiation at different fluence and flux conditions in order to investigate the deformation depth with dose variation. Nanoindentation hardness test was used to estimate the depth dependence of the irradiation hardening to evaluate deformation characteristic within those irradiated specimens.

2. Experimental

2.1 Material and specimen

The material in the present study was the SA508 CL.3 RPVS. The chemical compositions of the metal and the heat treatment conditions are shown in Table 1. The specimens were nanoindentation specimens with a thickness of 0.5mm and gauge length and width of 5mm and 5mm, respectively. All the specimen surfaces for ion-irradiation and nanoindentation tests were mechanically polished up to 1 ㎛. After polishing, the specimens were cleaned with ethanol to remove residual diamond particles.

2.2 Ion-irradiation and TRIM simulation

Specimens were irradiated with 1.6MV terminal voltage and 8MeV Fe^{+4} ions to 0.15dpa and 1.5dpa using a Tandem Vande-Graaff accelerator (model: NEC 5SDH-2) in Korea Institute of Geology and Mining (KIGAM). Beam homogeneity was achieved by the electric field scanning. The beam current was 250nA, the flux was 3.9×10^{15} ions $\cdot$ $m^{-2} \cdot sec^{-1}$ and the fluence was 4.24×10^{18} ions $\cdot$ m^{-2} at 0.15dpa and 4.24×10^{19} ions $\cdot$ m^{-2} at 1.5dpa. The irradiation period was 1086sec at 0.15dpa and 10860sec at 1.5dpa. The beam diameter was 10mm (Quadrangle Beam). The irradiation chamber was kept in a vacuum of 10^{-6} Torr and the irradiation temperature was not over 60℃. TRIM98 was used to determine the irradiation dose and accelerator condition with 40eV Fe atom displacement energy.

2.3 Nanoidentation test

Nanoindentation test was performed on a Nanoindentation Continuous Stiffness Measurement (CSM). Nanoindentation CSM technique is used to measure the load and hardness profile through depth of the SA508 CL.3 RPVS [3]. Figure1 shows the difference between basic nanoindentation test and nanoindentation CSM test. In a basic nanoindentation test, the physical property (or stiffness, $S = dP/dH$) of the maximum

depth is only obtained due to the fact that stiffness comes from the slope unloading [4]. But Nanoindentation CSM test utilizes low frequency oscillation during control of indentation depth to get continuous stiffness. As a result, we can get continuous stiffness. The oscillation amplitude should be kept small enough not to disturb original indentation process. In a review of the available experimental results Hahn, Lee and Kim [5] concluded that the test result of the modulus and hardness, measured using nanoindentater, was within the 5% deviation to the reported data. This means the reliability of the result from nanoindentation test and it can be compared directly with the result from basic nanoindentation test.

In this investigation, the indentation load was not fixed, but the indentation depth was fixed at 500㎚. Uncertainty in the indenter displacement increases at larger load speed especially at the beginning stage of its contact on the surface of the specimen, while vibration of the indentation machine can affect load-displacement results at lower speed. The loading rate was set at $0.05sec^{-1}$. To avoid any effects of grain boundaries, the deformed regions were carefully chosen.

3. Results and discussion

3.1 Deformation and damage depth profile

Figure 2 shows load versus indentation depth curves in SA508 CL.3 RPVS irradiated with 8MeV Fe^{+4} ions to 0.15dpa and 1.5dpa at below 60℃. The load increases with the depth both in un-irradiation and in irradiation condition. In addition, it has larger value for the case of irradiation than for un-irradiation. Note that the load is always larger with irradiation. Depth dependence of the load-displacement ratio (*L/D*) is shown in Figure 3. Irradiation hardening by 8MeV Fe^{+4} ions is most clearly observed for the initial part of the *Load/Depth* curve up to about 25-35nm.

The correlation between load and indentation depth during nanoindentation CSM in uniform specimens can be expressed as follows [6, 7];

$$L = (A_p + A_e)D^2 + BD\,, \qquad (1)$$

where A_p and A_e can be taken as characterizing plastic and elastic contributions to deformation, and B is a constant depending on the shape of indenter, respectively. It should be noted that A_p depends on the depth in the ion-irradiated samples with non-uniform formation and evolution of defects. The *Load/Depth* can be written by the following equation;

$$L/D = (A_p + A_e)D + B, \qquad (2)$$

$$\frac{d(L/D)}{dD} = A_p + A_e. \qquad (3)$$

The parameter *B* in Eq. (2) can be estimated by extrapolating the *Load/Depth* versus Depth to zero Depth. No effect of irradiation on the parameter was detected for all the tested materials as the ratio of load-depth.

Figure 4 shows the indentation depth dependence of the derivative value of the load-depth. This *d(L/D)/dD* is plastic and elastic deformation with respect to the depth. The peak in this deformation is clearly observed at an indenter depth between 15 and 25nm. Figure 5 shows the results of TRIM98 calculation for the damage depth profile. It is shown that the displacement damage peak appears at 1500nm from the surface. Because the effect of elastic deformation should not be considered except for estimating very low load indentation testing, the derivative value of the load-depth can be simply thought of plastic deformation. From this point of view, the plastic deformation peak with respect to the depth is 0.01 ~ 0.02 of the calculated displacement damage peak with respect to the depth because the shearing probability is much larger at near the surface than in the bulk, owing to the elastic dipole interaction in the bulk between dislocation loops of opposite character; that is, between loops of vacancies or interstitials [8].

3.2 Hardening depth profile

To estimate deformation in SA508 CL.3 RPVS, we tried to test the hardness change plotted against the indentation depth by nanoindentation CSM. Figure 6 shows the profile of the hardness change in SA508 CL.3 RPVS both un-irradiated and irradiated with 8MeV Fe^{+4} ions to 0.15dpa and 1.5dpa at below 60℃. In the figure, the hardness depends on the indentation depth. When the specimens are irradiated, hardness is larger than the un-irradiated. The reason why the hardness increases at irradiation is that the formation of the various point and clustered defects causes an increase in hardness. And, from the fact that hardness increases with irradiation rate, we can conclude that a resistance to deformation is larger for the irradiated than for the non-irradiated, and also larger for 1.5dpa than for 0.15dpa one.

4. Summary

A nanoindentation CSM technique has made it possible to evaluate irradiation deformation in SA508 CL.3 RPVS. It is found that the differential value of load-depth ratio, *d(L/D)/dD*, is estimated to evaluate the depth dependent formation of deformation features in the irradiated specimen. The depth distribution of the radiation-enhanced hardening in SA508 CL.3 RPVS is found to change with dose. The hardness of the irradiated specimens was always larger with that of the non-irradiated one. Also, the hardness was larger for 1.5dpa than for 0.15dpa in the same specimens under ion irradiation.

References

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Table 1 The chemical compositions and the heat treatments of SA508 CL.3 RPVS (in wt.%).

Element	C	Mn	Ni	Mo	Cu	Al	P
	0.17	1.35	0.82	0.5	0.03	0.015	0.006

Element	S	Si	Cr	N	V	Fe
	0.002	0.1	0.16	0.0055	0.004	Bal.

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Heat Treatments:

1) Quenching: 880℃, 8hr, water quench. 2) Tempering: 660℃, 10.5hr, air cooled.

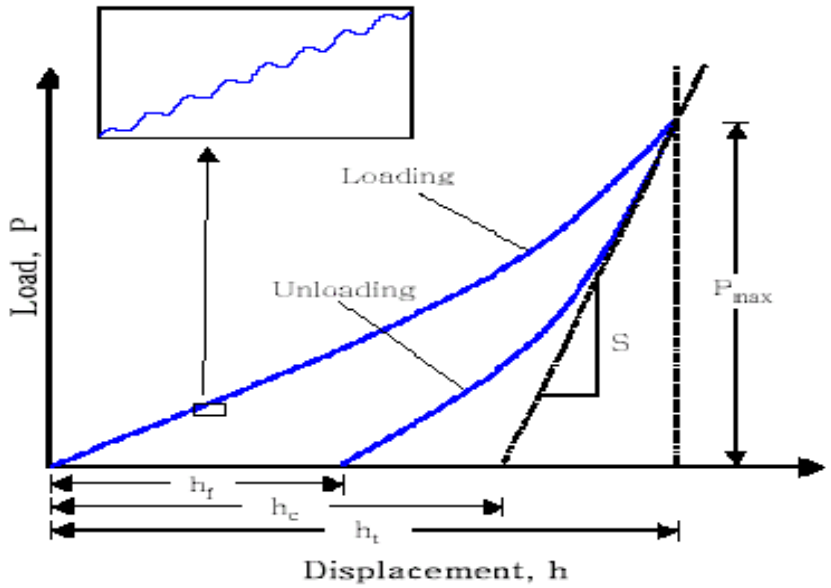

Figure 1 Schematic representation of load versus indenter displacement showing a comparison between the normal nanoindentation and the nanoindentation CSM. P_{max} represents the load of the maximum indentation depth, and S(stiffness) is calculated from the slope at the end of the unloading curve. Note that h_t is total displacement, h_s is elastic displacement and constant depth, h_c, is h_t - h_s. Also, h_f is depth from surface after load removal [3].

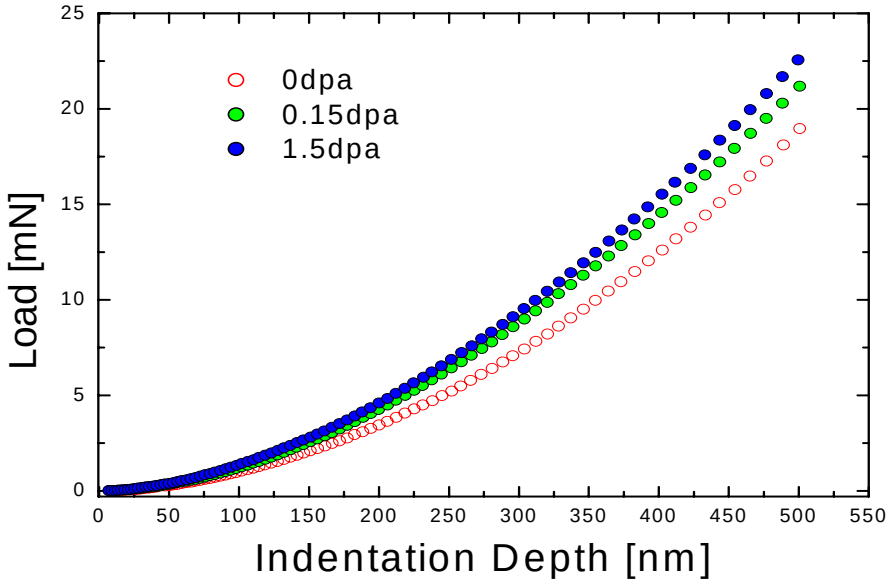

Figure 2 Load vs. indentation depth curves in SA508 CL.3 RPVS both un-irradiated and irradiated with 8MeV Fe^{+4} ions to 0.15dpa and 1.5dpa at below 60℃.

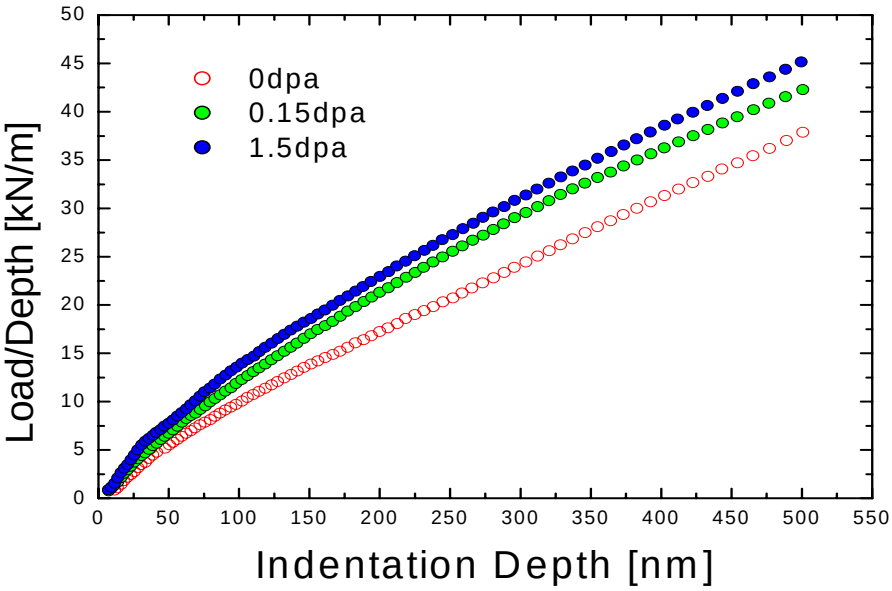

Figure 3 *Load/depth* vs. indentation depth curves in SA508 CL.3 RPVS both un-irradiated and irradiated with 8MeV Fe^{+4} ions to 0.15dpa and 1.5dpa at below 60℃.

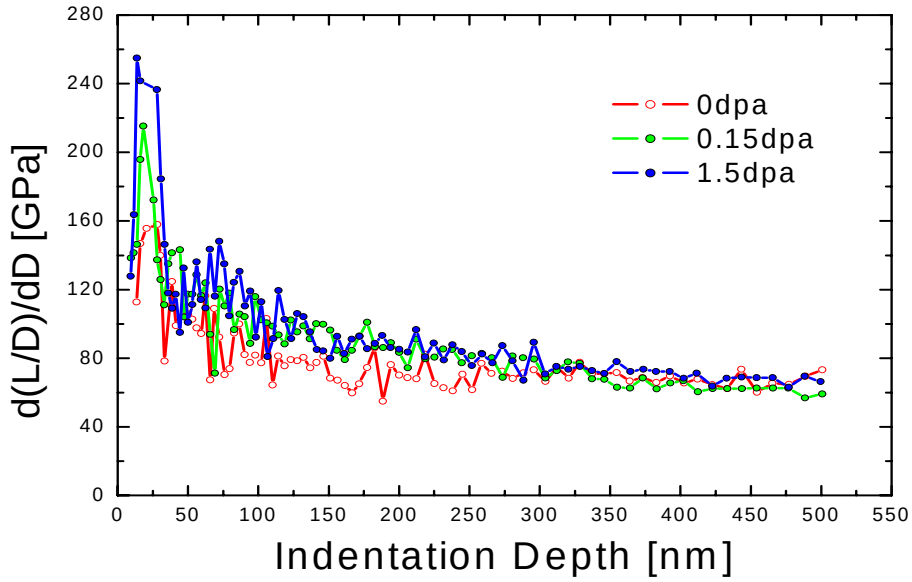

Figure 4 Derivative value of *Load/Depth* (*L/D*) as a function of indentation depth (*D*) in SA508 CL.3 RPVS both un-irradiated and irradiated with 8MeV Fe^{+4} ions to 0.15dpa and 1.5dpa at below 60℃.

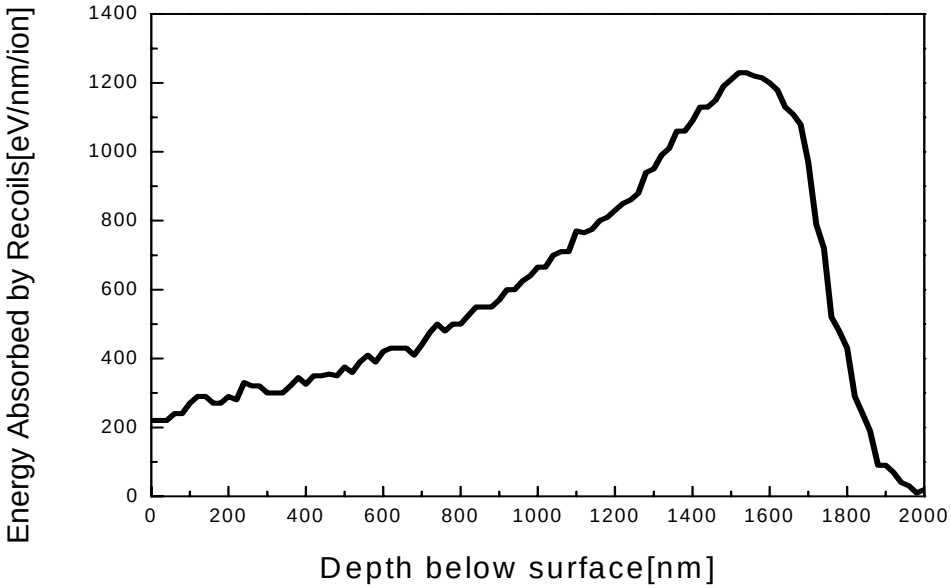

Figure 5 The calculated depth distribution of displacement damage by the TRIM98 simulation of the energy profile from 8MeV Fe ions.

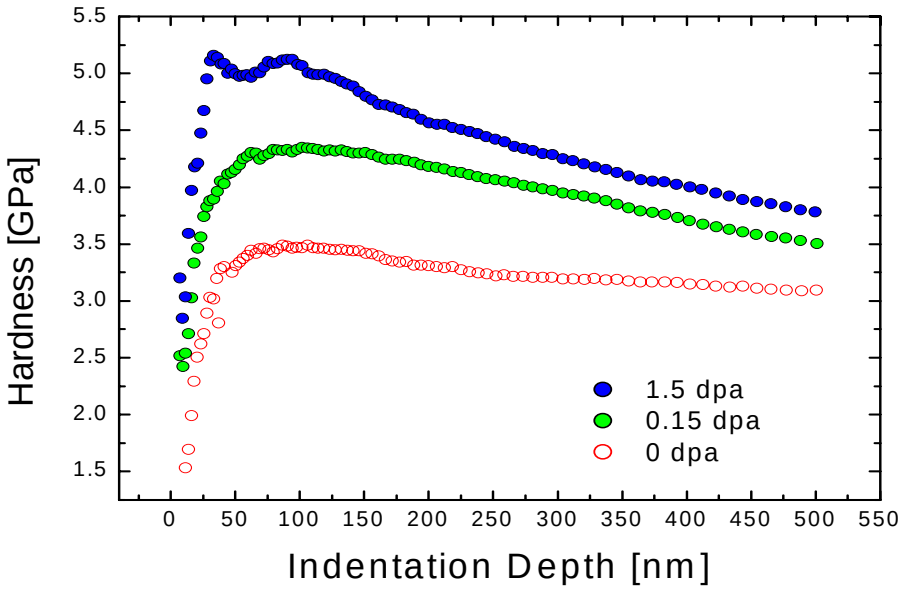

Figure 6 Dependence of hardness change on indentation depth for SA508 CL.3 RPVS both un-irradiated and irradiated with 8MeV Fe^{+4} ions to 0.15dpa and 1.5dpa at below 60℃.