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Original Article

The Studies of Irradiation Hardening of Stainless Steel Reactor Internals under Proton and Xenon Irradiation

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ABSTRACT

Specimens of stainless steel reactor internals were irradiated with 240 keV protons and 6 MeV Xe ions at room temperature. Nanoindentation constant stiffness measurement tests were carried out to study the hardness variations. An irradiation hardening effect was observed in proton- and Xe-irradiated specimens and more irradiation damage causes a larger hardness increment. The Nix-Gao model was used to extract the bulk-equivalent hardness of irradiation-damaged region and critical indentation depth. A different hardening level under H and Xe irradiation was obtained and the discrepancies of displacement damage rate and ion species may be the probable reasons. It was observed that the hardness of Xe-irradiated specimens saturate at about 2 displacement/atom (dpa), whereas in the case of proton irradiation, the saturation hardness may be more than 7 dpa. This discrepancy may be due to the different damage distributions.

1. Introduction

Austenitic stainless steels are essential structural materials that are widely used in light-water reactor internals due to their excellent strength, ductility, and corrosion-resistance properties. The reliability and integrity of such stainless steel internals are of particular importance for the safe operation of reactors. Irradiation hardening has been a concern for reactor internals in radiation environments during long-term service, and is considered as an important reason for various phenomena, such as irradiation-assisted stress corrosion cracking [1]. Therefore, it is essential to understand the hardening behavior of stainless steel internals.

Because of the difficulties associated with conducting neutron irradiation studies, charged particles (protons and heavy ions) were chosen to simulate the irradiation hardening behaviors of neutron irradiation. However, compared with the nearly uniform distribution of neutron irradiation damage, the shallow penetration depth and nonuniform irradiation damage distribution by ions irradiation create difficulties for hardness results analysis. It is therefore critical to extract the bulk hardness at corresponding irradiation damage from the

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nonuniform damage distribution. Moreover, many researchers choose protons or heavy ions to study irradiated hardening, mainly due to their accelerated effects, which are regarded as advantageous. However, only a few studies have been carried out to investigate the mechanical properties under different ion (protons or heavy ions) irradiation, and little is known about any additional hardening effects caused by different damage rates. Therefore, before analyzing the effect of hardening, the first thing we should do is to clarify hardening behaviors by different ion irradiation.

In the past decades, the nanoindentation technique was used to investigate mechanical properties after ion irradiation. These nanoindentation hardness data are valuable for scientific discussion. Moreover, the nanoindentation hardness data can be converted into the macroscopic Vickers hardness using the methods developed by Takayama et al. [2] and Kang et al. [3] based on the equation:

$$H_0 = 0.01\mathrm{HV} + 0.025 \qquad (1)$$

This method extends the discussion scope of nanoindentation hardness and can provide more references for engineering issues.

In this work, the irradiation hardening behaviors of the stainless steel reactor internals after proton and xenon irradiation were investigated by nanoindentation tests. The hardness of the ion-irradiated damage region was characterized by the Nix-Gao model. The hardening effect of protons and Xe ions irradiation was discussed and then the probable reason was given.

2. Materials and methods

The material used in this study was austenite stainless steel (Z6CND17.12) used for reactor baffle-former bolts. The specimens used in our experiments were cut from bars by solution treatment at 1,060 °C for 90 minutes, followed by air cooling. The chemical composition of this material is presented in Table 1.

The plate specimens ($10 \times 10 \times 1\ \mathrm{mm}^3$) were polished until they become mirror-like before irradiation. The specimens were irradiated with 240 keV protons and 6 MeV Xe^{26+} ions at room temperature in a chamber with a vacuum of 10^{-5} Pa at the ECR-320-kV High-Voltage Platform in the Institute of Modern Physics (Lanzhou, Gansu province). The specimens were irradiated to 5×10^{17} ions/cm^2, 1×10^{18} ions/cm^2, and 3.5×10^{18} ions/cm^2 with protons and 6.6×10^{14} ions/cm^2, 2.3×10^{15} ions/cm^2, and 5×10^{15} ions/cm^2 with Xe ions. According to the Monte Carlo code *SRIM 2012* [4], these fluences correspond to the peak damage levels of 1 displacement/atom (dpa), 2 dpa, and 7 dpa for proton irradiation and 2 dpa, 7 dpa, and 15 dpa for Xe irradiation (density of 7.8 g/cm^3 and threshold displacement energies of 40 eV for Fe, Cr, and Ni sublattices [5]), as shown in Fig. 1. In the SRIM calculation process, the vacancy file obtained by the Kinchin–Pease quick calculation model was used to calculate the displacement damage values. The displacement damage rate for H and Xe irradiation is about 1.1×10^{-4} dpa/s and 8.0×10^{-4} dpa/s, respectively.

Table 1 – Chemical composition of the stainless steel Z6CND17.12.

Elements[a]	C	Si	Co	P	S
Weight (%)	0.038	0.340	0.010	0.008	0.003
Elements	Cr	Ni	Cu	Mo	Mn
Weight (%)	17.28	11.65	0.46	2.49	1.24

[a] Balance of composition is Fe.

Nanoindentation measurements of the specimens were carried out using a diamond Berkovich indenter in a Nano Indenter G200 (Agilent Technologies) at Suzhou Institute of Nano-Tech and Nano-Bionics (Suzhou, Jiangsu province). The continuous stiffness measurement mode was chosen to obtain a hardness (*H*) versus depth (*h*) profile. The hardness was calibrated using a fused silica reference material to 2.0 μm depth. Specimens were mounted onto aluminum stubs with hot wax and indents were produced in a direction normal to the specimen surface. The maximum penetration depth and applied load were about 2.0 μm and 330 mN, respectively. Each specimen was tested five times at different points, and average values were taken for analysis. The distance between indentations was ~50 μm. Because of the deviation from the ideal shape of the diamond indenter tip geometry and the surface effect of the specimens, the data from the surface to 50 nm were not accurate. Therefore, we did not use the hardness data from the surface to 50 nm in this study.

3. Results and Discussion

Fig. 2 shows the hardness versus the penetration depth of unirradiated specimens, specimens irradiated to 1 dpa, 2 dpa, and 7 dpa by protons and 2 dpa, 7 dpa, and 15 dpa by Xe. It can be clearly seen that the hardness of both proton- and Xe-irradiated specimens is larger than that of unirradiated specimens. This indicates an irradiation-hardening phenomenon of stainless steel. The irradiation hardness is fluence dependent, and higher fluence causes a greater hardness increment. It is well-known that hardening of irradiated specimens is mainly due to the formation of irradiation defects. Previous studies have proved that more irradiation defects will be produced by higher irradiation fluence [6,7]. Thus, it is reasonable that more significant hardening appears in higher fluence specimens.

The gradual decrease of hardness curves with indenter depth from around 50 nm to 2,000 nm was observed, as shown in Fig. 2. This decrease is caused by the indenter size effect. This effect can be explained by the model developed by Nix-Gao based on the geometrically necessary dislocation theory [8]. Using the Nix-Gao model, the hardness–depth profile is expressed as:

$$H = H_0(1 + h^*/h)^{0.5} \qquad (2)$$

where H_0 is the hardness at infinite depth (i.e., bulk hardness) and h^* is a characteristic length that depends on the material and shape of the indenter trip. According to this model, with the increases in indentation depth (*h*), the