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Evaluation of hardening behaviors in ion-irradiated Fe–9Cr and Fe–20Cr alloys by nanoindentation technique

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ABSTRACT

The ion irradiation hardening behaviors of Fe–9 wt% Cr and Fe–20 wt% Cr model alloys were investigated by nanoindentation technique. The specimens were irradiated with 3 MeV Fe^{11+} ions at room temperature up to 1 and 5 dpa for Fe–9Cr alloy and 1 and 2.5 for Fe–20Cr alloy. The ratio of average hardness in the same depth of irradiated and unirradiated (Hirr. av/Hunirr. av) was used to determine the critical indentation depth hcrit to eliminate the softer substrate effect. The Nix–Gao model was used to explain the indentation size effect. Irradiation hardening is clearly observed in both Fe–9Cr alloy and Fe–20Cr alloy after ion irradiation. The differences of ISE and irradiation hardening behaviors between Fe–9Cr and Fe–20Cr alloys are considered to be due to their different microstructures and microstructural evolution under ion irradiation.

1. Introduction

Although having many differences with neutron irradiation, heavy ion irradiations (especially self-ion, e.g., Fe ion for steels) are widely used, because of their advantages: rapid damage production, no induced-radioactivity, easier control of irradiation conditions, and co-implantation with helium and/or hydrogen [1–3]. The nanoindentation technique has been widely used to investigate the hardening behaviors of ion-irradiated materials, such as austenitic steels [4], Fe–Cr alloys [5], ferritic/martensitic (F/M) steels [6], reduced activation ferritic/martensitic (RAFM) steels [7], and Fe–Cu alloys [8]. The hardness depth profiles after self-ion irradiation could intrinsically include three different depth dependent effects: the indentation size effect (ISE), the softer substrate effect (SSE), and the possible damage-gradient effect (DGE) [8–11]. The most commonly applied model to describe the ISE is the Nix-Gao model [12]. Kasada et al. proposed a composite hardness model for SSE of the non-irradiated region beyond the irradiation range, and reported that the critical depth of SSE depended on the amount of irradiation hardening in the irradiated layer [3].

Fe–Cr and Fe–Cr–X based alloys are the most widely used materials in the nuclear industry. High chromium (7–12 wt%) F/M steels, such as HT9, T91, F82H, CLAM, NF616, Eurofer 97, etc, are widely considered as candidates of structural materials in Generation IV fission and fusion reactors because of their low swelling, higher thermal conductivity and reduced creep rate [13–15]. The irradiation-induced microstructural evolution and mechanical property changes of F/M steels become the research hot spot recently [3,5–7,13,14]. In addition, ferrite phases in duplex stainless steels (DSS), cast austenitic stainless steels (CASS) or weld of austenitic stainless steels, generally contain >20 wt% Cr, are well known to be sensitive to thermal aging embrittlement, which is induced by spinodal decomposition of ferrite into Cr-rich α' and Cr-depleted α domains [16,17]. Neutron/ion irradiation was found to induce spinodal decomposition in the ferrite of as-cast microstructure, and also affect the spinodal decomposition in the thermally aged cast alloys [18–20]. In the present paper, two kinds of model alloys Fe–9 wt% Cr and Fe–20 wt% Cr were investigated to study the effects of ion irradiation on the hardening behaviors of Fe–Cr alloys by nanoindentation technique.

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2. Experimental

Fe–9Cr and Fe–20Cr alloys were prepared by melting of pure Fe (99.9%) and Cr (99.99%) with a vacuum induction melting furnace (ZG-0.01). Alloys were remelted several times in order to obtain the homogenous composition. The ingots were homogenized at 1100 °C for 20 h and subsequently quenched in water. Fig. 1 shows the EBSD orientation maps for the martensitic structure in Fe–9Cr alloy and the single-phase ferritic structure in Fe–20Cr alloy. Table 1 lists the chemical compositions of the two alloys. Prior to irradiation, the irradiation surfaces were mechanically polished, and then finished by electro-polishing to remove any deformation layer.

Plate specimens of 5 × 5 mm and 2 mm in thickness were irradiated with 3 MeV Fe^{11+} ions at room temperature at a flux of 3.34×10^{15} ions/m^2/s on the 320 kV multi-discipline research platform for highly charged ions in Institute of Modern Physics (IMP) in Lanzhou, China. The damage profile of 3 MeV Fe ion irradiations in the Fe–Cr alloys was calculated with SRIM-2008 [21] using the quick damage mode with average displacement energies of 40 eV Fig. 2 shows the depth profiles of the displacement damage in Fe–Cr alloys irradiated with 3 MeV Fe ions, and the calculated peak damage appears at about 870 nm in depth. The peak damages were taken as their nominal displacement damages, which were up to 1 and 5 dpa for Fe–9Cr alloy and 1 and 2.5 for Fe–20Cr alloy.

The irradiation hardening behaviors of Fe–Cr alloys were measured by a nanoindenter (Nano Indenter DCM, MTS, now Agilent Technologies) with a Berkovich type indentation tip, which was calibrated against a fused silica standard. The continuous stiffness measurement (CSM) was carried out with an indentation depth of 2000 nm, an amplitude of 2 nm, and a frequency of 45 Hz, and more than eight indents were tested for each sample.

3. Results and discussion

Fig. 3 shows the indentation-depth dependence of the nanoindentation hardness of Fe–9Cr alloy before and after the ion irradiation. Each plot symbol represents data of a single indent. For both of the unirradiated and irradiated samples, the decrease in hardness with increasing indent depth was observed. This depth-dependent hardness behavior has been noticed as ISE [22], which is well explained by the Nix-Gao model based on a concept of geometrically necessary dislocation [12]. Fig. 4 shows the indentation-depth dependence of the averaged nanoindentation hardness with error bars on each specimen of Fe–9Cr alloy. After Fe-ions irradiation, the hardness of Fe–9Cr alloy has an obvious increase, especially in the near surface region. The specimens irradiated to 1 dpa and 5 dpa have the almost same value in hardness.

Kasada et al. [8] reported that the ion-irradiated sample usually had an inflection of SSE at a critical indentation depth (h_{crit}), over which a contribution of softer unirradiated region (substrate) beyond the harder ion-irradiated surface to the measured hardness

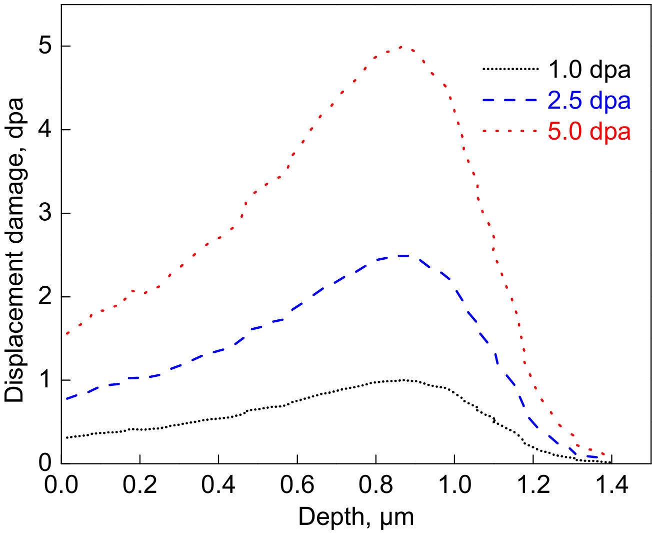

Fig. 2. Depth profiles of the displacement damage in Fe–Cr alloys irradiated with 3 MeV Fe ions, calculated by SRIM.

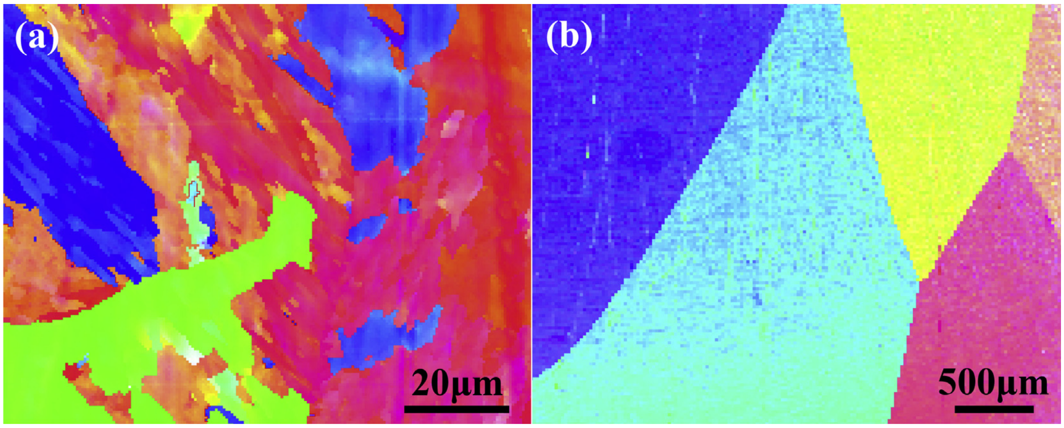

Fig. 1. EBSD orientation maps for (a) the martensitic structure in Fe–9Cr alloy and (b) the single-phase ferritic structure in Fe–20Cr alloy.

Table 1
Chemical compositions of Fe–9Cr and Fe–20Cr alloys.

Alloy	C	Cr	Ni	Al	Mn	Si	S	P	Fe
Fe–9Cr	0.0128	9.05	0.015	0.016	0.023	0.008	<0.005	<0.005	Balance
Fe–20Cr	0.0106	19.98	0.014	0.018	0.024	0.008	<0.005	<0.005	Balance