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Comparison of magnetic properties of austenitic stainless steel after ion irradiation

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ABSTRACT

Specimens of austenitic stainless steel (ASS) were irradiated with H, Fe and Xe ions at room temperature. The vibrating sample magnetometer (VSM) and grazing incidence X-ray diffraction (GIXRD) were used to analyze the magnetic properties and martensite formation. The magnetic hysteresis loops indicated that higher irradiation damage causes more significant magnetization phenomenon. Under the same damage level, Xe irradiation causes the most significant magnetization, Fe irradiation is the second, and H irradiation is the least. A similar martensite amount variation with irradiation can be obtained. The coercivity H_c increases first to 2 dpa and then decreases continuously with irradiation damage for Xe irradiation. At the same damage lever, H irradiation causes a largest H_c and Xe irradiation causes a minimal one.

1. Introduction

Austenitic stainless steels (ASS) are generally used as incore structural components in light water reactors as internals, neutron flux thimble tube and so on due to its excellent corrosion resistance in combination with high ductility and toughness at elevated temperature. The reliability and integrity of such ASS components are of particular importance for reactors safe operation. However, in-service inspection of the core internals has revealed a susceptibility of baffle to former bolts known as Irradiation Assisted Stress Corrosion Cracking (IASCC). IASCC corresponds to irradiation induced or enhanced intergranular stress corrosion cracking of the material. It is known that corrosion resistance along grain boundaries can be degraded with Cr depletion caused by radiation induced segregation (RIS), and this is considered one of the principle mechanisms of IASCC on stainless steels [1]. Previously Kodama et al. [2] suggested that the IASCC susceptibility was related to the austenite stability in ASS. Simultaneously many studies [3] have indicated that RIS will result in the formation of magnetic phase in austenitic steels. So the investigation of the magnetic phase in ASS can provide information for understanding of IASCC mechanism.

Due to the difficulty on neutron irradiation studies, charged particles (protons and heavy ions) were chosen to simulate the irradiation behaviors of neutron irradiation. So it is critical to understand the difference of magnetic properties and austenitic phase stability in ASS under different ions irradiation. But so far few studies have been carried out due to the limitation of shallow penetration depth of ions irradiation which leads to a difficulty to obtain the magnetic properties from a thicker unirradiated layer.

In the present studies, the vibrating sample magnetometer (VSM) was conducted with a TEM specimen to investigate the magnetic properties under H, Fe and Xe. The saturated magnetization and coercivity obtained from hysteresis loops were give a detailed analysis and comparison subsequently with the irradiation damage and damage rate. Moreover, in order to give more auxiliary information of martensite formation in austenite, the grazing incidence X-ray diffraction (GIXRD) was also used.

2. Experiments

The materials used in this study are a Z6CND17.12 ASS. The specimens used in experiments were cut from a bar with solution treatment at 1060 ± 10 °C for 90 min. The chemical composition is Cr (17.28%), Ni (11.65%), Mo (2.49%), Mn (1.24%), Cu (0.46%), Si (0.340%), C (0.038%), Co (0.010%), P (0.008%), S (0.003%) and Fe (the balance).

The specimens before irradiation were polished to mirror-like with mechanical method. Then they were irradiated with proton, Fe and Xe ions separately at room temperature to different fluences (as shown in Table 1) at the ECR-320 kV High-voltage Platform in the Institute of Modern Physics. The theoretical results of the displacement damage are calculated by the Monte-Carlo code SRIM 2012 [4] as shown in Table 1, taking the density of 7.8 g/cm^3 and threshold displacement energies of 40 eV for Fe, Cr and Ni sub-lattices [5]. In the SRIM calculation process, the vacancy file obtained by the Kinchin-Pease quick calculation model was used to calculate the displacement damage values.

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Table 1
The irradiation parameters of ASS specimens.

Ions species	Ions energy	Fluence (ions/cm^2)	Peak displacement damage (dpa)
proton	240 keV	3.5×10^{18}	7
Fe	3.5 MeV	6.7×10^{15}	7
Xe	6 MeV	6.6×10^{14}	2
		2.3×10^{15}	7
		5.0×10^{15}	15
		8.3×10^{15}	25

Magnetic hysteresis loops of ASS were measured with the vibrating sample magnetometer (VSM) 7407 produced by Lake Shore. The maximum magnetic field intensity is 3000 Oe in measurement. A 3 mm diameter disk shape specimens with a thickness of about 30 μm was used in measurement to deviate the demagnetizing effects due to specimen shape and size and decrease the effect of unirradiated parts. GIXRD was carried out at Beijing Synchrotron Radiation Facility, Institute of High Energy Physics. X-rays was generated by a bending magnet, focused and monochromated to a wavelength of 0.154 nm. The X-ray scanning range was from 35 to 55 degree with a resolution of 0.05 degree.

3. Results

Fig. 2 shows the variations of magnetization-magnetic field hysteresis loops of initial specimen and specimens irradiated with H, Fe and Xe ions separately to different damage. As shown in Fig. 2, the initial specimen remains a small magnetization in the entire field, showing up no hysteresis dependence typical of ferromagnetic materials. The ASS is paramagnetic due to its perfect austenitic microstructure. The dependence of the specific magnetization M on the external magnetic field H for the unirradiated specimen is a straight line described by the dependence of $M(H) = \chi_p H$, where χ_p is the paramagnetic susceptibility. So there is no hysteresis dependence of unirradiated specimen.

Nevertheless, after irradiation with H, Fe or Xe, the specimens show the hysteresis dependence typical of a ferromagnetic property with a nonlinear variation. For the Xe irradiated specimen, higher irradiation damage causes more significant magnetization. Under the same damage level, Xe irradiation causes the most significant magnetization, Fe irradiation is the second, and H irradiation is the least.

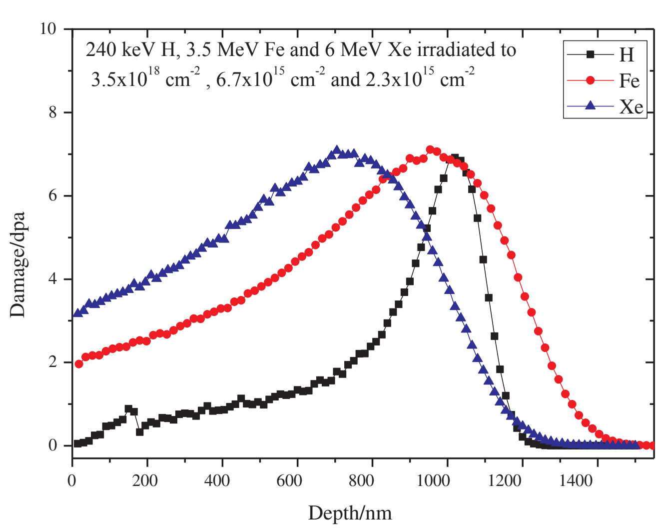

Fig. 1. Distribution of displacement damage versus depth in stainless steel irradiated with H, Fe and Xe ions separately to different fluences according to simulation with SRIM.

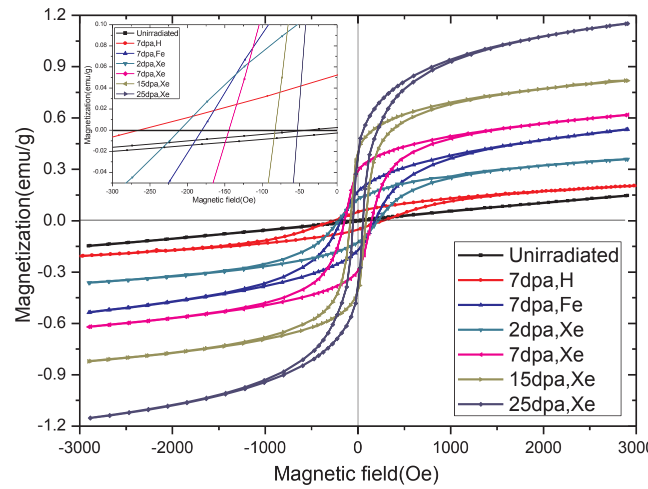

Fig. 2. Variations of hysteresis loops of ASS irradiated with H, Fe and Xe to different damage.

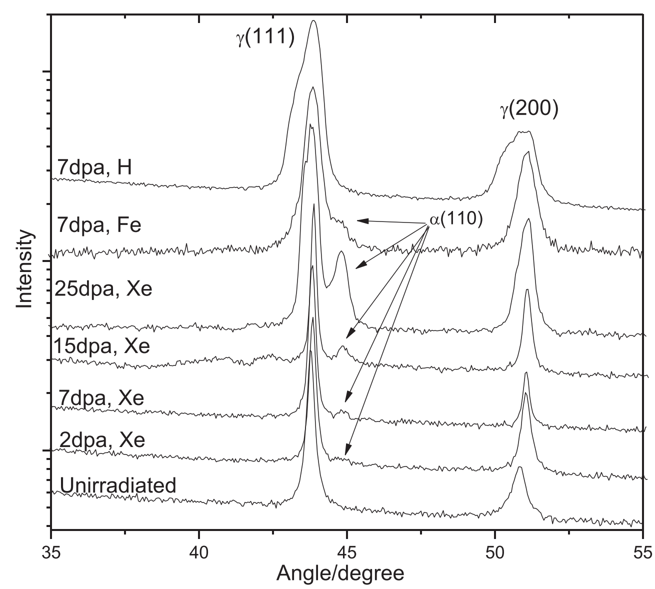

Fig. 3. GIXRD patterns of the ASS irradiated with H, Fe and Xe to different damage.

In order to obtain further information of magnetic phase after irradiation, the GIXRD patterns of unirradiated specimen and specimens irradiated to different damage level with H, Fe and Xe were investigated (as shown in Fig. 3). It is observed that the unirradiated specimen shows two face-centered-cubic austenite diffraction peaks of γ(1 1 1) and γ(2 0 0). No other diffraction peaks are observed. With increasing the irradiation fluence by Xe, a new diffraction peak corresponding to the α(1 1 0) appears and then become remarkable gradually with irradiation damage increased to 25 dpa. This indicates the formation of α martensite phase during Xe ion irradiation and the amount of the martensite shows a continuous increase with irradiation. A similar diffraction peak of α(1 1 0) can also be observed after irradiated to 7 dpa by Fe, which also indicate the formation of the martensite. This α martensite phase has been proved in ASS after ions irradiation by GIXRD [6,7], TEM [8,9] and Mössbauer [10]. Whereas, in the case of H irradiation, compared to the Xe and Fe irradiation, no remarkable new diffraction peak was observed. But the initial γ(1 1 1) and γ(2 0 0) become broadening.

The austenitic phase exhibits paramagnetic properties and magnetic phase is ferromagnetic. The formation of α magnetic phase in austenitic