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Magnetic properties of a stainless steel irradiated with 6 MeV Xe ions

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ABSTRACT

Specimens of austenitic stainless steel were irradiated with 6 MeV Xe ions at room temperature to 2, 7, 15 and 25 dpa. The vibrating sample magnetometer (VSM), grazing incidence X-ray diffraction (GIXRD) and positron annihilation lifetime spectroscopy (PLS) were carried out to analysis the magnetic properties and microstructural variations. The magnetic hysteresis loops indicated that higher irradiation damage causes more significant magnetization phenomenon. The equivalent saturated magnetization M_{es} and coercive force H_c were obtained from magnetic hysteresis loops. It is indicated that the M_{es} increases with irradiation damage. While H_c increases first to 2 dpa and then decreases continuously with irradiation damage. The different contributions of irradiation defects and ferrite precipitates on M_{es} and H_c can explain these phenomena.

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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. The property degeneration of stainless steel internals has been reported in several nuclear power stations, such as the baffle-former bolts cracking at Bugey-2 (CP0 series units) in France, Tihange-1 in Belgium, Point Beach-2 and Ginna in American caused by irradiation assisted stress corrosion cracking (IASCC).

Previous studies have suggested that the phase transformation (ferrite phase formation) and IASCC phenomenon can be detected simultaneously in austenitic stainless steels after irradiation [1]. Takahashi et al. [2] have indicated that the ferrite phase formation in austenitic steels is closely related to the matrix elements segregation because alloying elements like Ni, Mn considerably stabilize the austenite structure while the Cr promotes a ferrite structure. This means the formation of ferrite in stainless steel is affected by radiation induced segregation (RIS) processes. On the other hand, it has been suggested that RIS of chemical elements at grain boundaries plays important roles on IASCC [3,4]. So the investigation of the phase transformation of stainless steel can provide information on RIS and are of great importance for understanding of IASCC mechanism.

The formation of ferrite phase in austenitic steels (which is paramagnetic at room temperature) can be detected by magnetic properties analysis. Assessing the degradation of stainless steel by nondestructive magnetic method is of current interest because magnetic properties are very sensitive to the changes in microstructures. In the present studies, the vibrating sample magnetometer (VSM) was conducted to investigate the magnetic properties under different irradiation damage. The equivalent saturated magnetization and coercive force obtained from hysteresis loops were given a detailed analysis. Moreover, the grazing incidence X-ray diffraction (GIXRD) and positron annihilation spectroscopy (PLS) was also used to give more auxiliary information on ferrite phase formation in austenite and vacancy defects evolution.

2. Experiments

The materials used in this study are the austenite stainless steel Z6CND17.12 used for reactor baffle-former bolts. The specimens used in our experiments were cut from bar with solution treatment at 1060 ± 10 °C for 90 min followed by air cooling. 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

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with mechanical method. The specimens were irradiated with Xe ions at room temperature to different fluences at the ECR-320 kV High-voltage Platform in the Institute of Modern Physics. The irradiation fluences were 6.6 × 10^{14}, 2.3 × 10^{15}, 5 × 10^{15} and 8.3 × 10^{15} Xe/cm^2. According to the theoretical calculation by Monte-Carlo code SRIM 2012 [5] (taking the density of 7.8 g/cm^3 and threshold displacement energies of 40 eV for Fe, Cr and Ni sublattices [6]), there fluences correspond to the peak damage levels of 2, 7, 15 and 25 displacement/atom (dpa) as shown in Fig. 1. The displacement damage rate is about 8.0 × 10^{-4} dpa/s (corresponding to the flux of 2.6 × 10^{11} Xe/cm$^2\cdot$s). In the SRIM calculation process, the vacancy file obtained by the Kinchin–Pease quick calculation model was used to calculate the displacement damage values.

Magnetic hysteresis loops of austenite stainless steel 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 to deviate the demagnetizing effects due to specimen shape and size and decrease the effect of unirradiated parts.

GIXRD was carried out at Shanghai Synchrotron Radiation Facility (for 25 dpa specimen) and Beijing Synchrotron Radiation Facility (for other specimens). X-rays was generated by a bending magnet, focused and monochromated to a wavelength of 1.2398 nm and 0.154 nm. The X-ray scanning range was from 35 to 55° with a resolution of 0.05°. According to the penetration depth of X ray calculation by $t_0 = 1/\mu_m\rho$ (where μ_m is the absorption factor and ρ is the density), the penetration depth of incident angle of 4° was chosen in order to correspond to the microstructure at Xe irradiation damage region.

The PLS of specimens (15 mm × 15 mm × 1 mm) were measured by means of BaF$_2$ lifetime spectrometer with a ^{22}Na positron source. At least 10^6 counts are collected in each spectrum. Besides the source components, all measured positron lifetime spectra were fitted by the PATFIT program with two lifetime components of short lifetime parameter τ_1 and long lifetime parameter τ_2. Usually, the total number of residual defects can be derived from the positron mean lifetime τ_{av} [7] (the τ_{av} can be obtained by the equation of $\tau_{av} = \tau_1 I_1 + \tau_2 I_2$, where I_1 and I_2 are the intensity of τ_1 and τ_2).

3. Results and analysis

To investigate the effect of ions irradiation on the magnetic properties of austenitic stainless steels, we measured magnetic hysteresis loops by using VSM before and after irradiation. Fig. 2 shows the variations of magnetization-magnetic field hysteresis loops of initial specimen and specimens irradiated with Xe ions to 2, 7, 15 and 25 dpa respectively. As observed from Fig. 2, the initial specimen remains a small magnetization value with a linear increase in the entire field, showing up no hysteresis dependence typical of ferromagnetic materials. Nevertheless, after irradiation with Xe, the specimens show the hysteresis dependence typical of ferromagnetic materials with a nonlinear variation. It is indicated that higher irradiation damage causes more significant magnetization phenomenon.

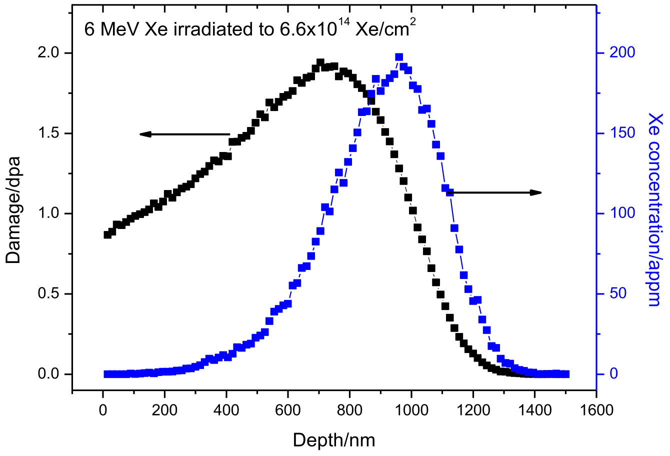

Fig. 1. Distribution of displacement damage versus depth in stainless steel irradiated with Xe ions to 6.6 × 10^{14} ions/cm^2 according to simulation with SRIM.

The austenitic stainless steels are 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, as shown in Fig. 2, austenitic stainless may become ferromagnetic under the condition of irradiation. This phenomenon must be due to the microstructure variations. Therefore, in order to obtain the microstructure information of irradiated specimens, the GIXRD patterns of initial stainless steel and stainless steel irradiated to 2, 7, 15 and 25 dpa were investigated (as shown in Fig. 3). It is observed from GIXRD that the unirradiated specimen shows two face-centered-cubic austenite diffraction peaks of γ(111) and γ(200). No other diffraction peaks are observed. With increasing the irradiation fluence, a new diffraction peak corresponding to the α(110) appears and becomes remarkable gradually with irradiation damage increase to 25 dpa. This indicates that partial austenite phase transforms into ferrite phase during Xe ions irradiation. The ferrite phase is formed and its size and density enlarge with irradiation damage. This can also be proved by Tan et al. [8] who observed ferrite clusters with several nanometers in size and 10^{22} m^{-3} in density by TEM under neutron irradiation.

Fig. 4 is the variations of the relative GIXRD intensity of α(110)/γ(111) to different irradiation damages. The relative GIXRD intensity means the ratio of relative peak intensity between α(110) and γ(111) obtained from Fig. 3. According to Fig. 4, the relative GIXRD intensity increases with irradiation damage, indicating an increase of amount of ferrite phase with the irradiation. Several studies have proved that the formation of ferrite phase will change the magnetic behavior of austenitic stainless steels [1,9]. The more of the ferrite phase precipitates, the stronger of the magnetization. Therefore, based on the martensitic transformation, the austenitic stainless steel will become ferromagnetic dependence and magnetization depends on the irradiation damage directly.

Fig. 4 is the variations of the M_{es} to different irradiation damages. The M_{es} denotes the magnetization at 3000 Oe as equivalent saturated magnetization obtained from Fig. 2. According to Fig. 4, the M_{es} increases with irradiation damage, indicating a gradually increase of magnetization phenomenon. Meanwhile, a similar trend of relative GIXRD intensity and M_{es} can also be observed.

Previously, Jiles [10] believes that defect clusters reduce the energy of domain walls when the domain walls intersect defect clusters and consequently the domain walls are attracted to the defect clusters which effectively impede wall motion, leading to change of magnetic properties of irradiated specimens. Park et al. [11] assume that defects clusters may be associated with the decrease of magnetic domain wall energy induced and cause the increase of saturation magnetization. While Takaya et al. [12] indicated that the M_{es} depends entirely on the amount of the magnetic phase. In our present studies, we assume that the increase of the M_{es} is related to the defect clusters and ferrite precipitates together. When the irradiation damage is less than 2 dpa, the M_{es} is determined by the ferrite precipitates and irradiation

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