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Characterization of magnetic properties in a 316 stainless steel after deformation and irradiation

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ABSTRACT

Specimens of austenitic stainless steel were compressively deformed or irradiated with Xe ions at room temperature. The characterization of the magnetic properties and microstructural variations were done by vibrating sample magnetometer, grazing incidence X-ray diffraction and TEM. The results indicated that higher deformation or irradiation damage causes more significant magnetization phenomenon. The amount of ferromagnetic phase can be deduced from M_s and increases with deformation and irradiation. The stress relief after deformation or irradiation may be responsible for the ferromagnetic phase formation. H_c increases first and then decreases continuously with a turning point at deformed to 40% or irradiated to 2dpa. The different contributions of dislocation density and ferromagnetic precipitates on H_c can explain this variation.

1. Introduction

Austenitic stainless steels (ASS) are essential structural materials that are widely used in light water reactor due to their excellent properties. The reliability and integrity of such stainless steel are of particular importance for the reactors safe operation. The property degeneration of stainless steel has been reported in several nuclear power stations, including the irradiation assisted stress corrosion cracking (IASCC), fatigue and so on. It is well established that a main factor that is responsible for these degradations in ASS is the martensitic transformation of γ (fcc) to α (bcc) by deformation or irradiation [1,2]. This ferromagnetic α-phase can lead to cracks due to the hard and brittle nature of martensite phase and associated microstructural change and chemical element segregation. So the investigation of ferromagnetic phase can provide more information on the IASCC and fatigue.

On the other hand, because magnetic measurement is more sensitive on α-phase formation than any other kind of measurements and may provide various kinds of information about the mechanism of the transformation, it gets more and more attentions on the use of measurements of magnetic properties as a non-destructive evaluation (NDE) tool for monitoring and determining deformation and fatigue damage prior to crack initiation in stainless steels [2,3]. So the characterization of magnetic properties after deformation or irradiation can obtain the basic data on damage evaluation.

At present, the saturation magnetization and coercivity measurements were often used to evaluate the magnetic properties, such as volume fraction of α-phase et al., few studies were conducted to compare the effect of strain and irradiation on the magnetic properties of ASS. Simultaneously, due to the lack of comprehensive knowledge on the coercivity under a continuous deformation or irradiation damage, variations of coercivity have not formed a unified understanding. In this studies, the vibrating sample magnetometer (VSM) was conducted to investigate the magnetic properties under compressively deformation or irradiation. The saturation magnetization and coercive force obtained from hysteresis loops were given a detailed analysis. Moreover, grazing incidence X-ray diffraction (GIXRD) and TEM were also used to give more auxiliary information on ferromagnetic phase formation in austenite.

2. Experiments

The materials used in this study are 316 ASS used for reactor internals. 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 used for compressive deformation tests are cylinder. Then they were compressively deformed at room temperature with a strain rate of 0.5 mm/min using a MTS Landmark k370–500 kn testing machine to obtain different plastic deformation levels. The plastic strain was calculated by the equation of $\varepsilon = \frac{h_0 - h}{h_0} \times 100\%$, where ε is the plastic strain value in%, h_0 and h is the initial and remaining height of

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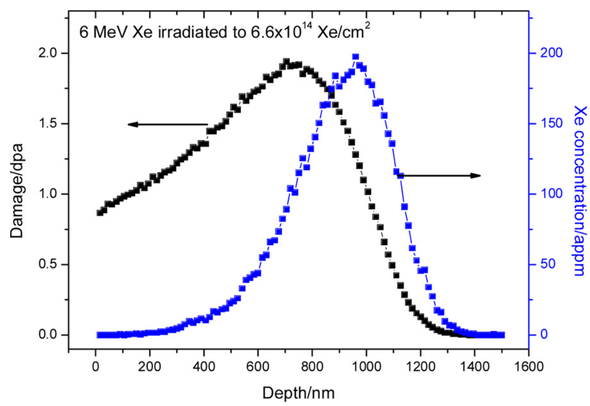

Fig. 1. Distribution of displacement damage versus depth in stainless steel irradiated with Xe ions to 6.6×10^{14} ions/cm^2 according to simulation with SRIM.

cylinder before and after deformation. With this method, four different levels of true strain of 22%, 40%, 50% and 60% were obtained.

The specimens used for irradiation were polished to mirror-like with mechanical method. Then these specimens were irradiated with 6 MeV Xe ions at room temperature to different fluences at the ECR–320 kV High-voltage Platform. The irradiation fluences were 6.6×10^{14}, 2.3×10^{15}, 5×10^{15} and 8.3×10^{15} Xe/cm^2. These fluences correspond to the peak damage levels of 2, 7, 15 and 25 displacement/atom (dpa) calculated by Monte-Carlo code SRIM 2012 [4] (taking the density of 7.8 g/cm^3 and threshold displacement energies of 40 eV for Fe, Cr and Ni sub-lattices [5]). The distribution of displacement damage versus depth in stainless steel irradiated with Xe ions to 6.6×10^{14} ions/cm^2 is shown in Fig. 1.

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 measurements. A 3 mm diameter disk shape specimens with a thickness less than 30 μm was used in measurement to decrease the effect of unirradiated parts.

Grazing incidence X-ray diffraction (GIXRD) was carried out at Beijing Synchrotron Radiation Facility (for irradiated specimens). 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 ° with a resolution of 0.05 °. A conventional XRD measurements (for deformed specimens) were carried out on a four-circle diffractometer with the pure Cu Kα1 line of wavelength 0.154 nm by D8 Discovery of BRUKER. The X-ray scanning range was from 35 to 55 ° with a resolution of 0.02 °.

Thin foils suitable for TEM were prepared by ion milling with 5.5 keV Ar ions after a Φ3 mm diameter cross-sectional discs grinding to about 30 μm. Microstructural characterization was conducted in a FEI Tecnai G^2 F20 analytical TEM with a resolution of 0.19 nm.

3. Results and analysis

To investigate the effect of deformation and irradiation on magnetic properties of ASS, we measured magnetic hysteresis loops by using VSM after deformation and irradiation respectively. As observed in Fig. 2, the magnetization curve of initial specimen (no deformation and irradiation) remains a small magnetization value with a linear increase in the entire field, indicating no hysteresis dependence typical of ferromagnetic materials. The ASS is paramagnetic due to its perfect austenitic microstructure. So there is no hysteresis dependence of initial specimen.

Whereas after deformation or irradiation, it is indicated that the hysteresis dependence typical of ferromagnetic materials with a nonlinear variation. As shown in Fig. 2, a weak magnetization phenomenon can be observed after deformed to 22%. With the increase of deformation after more than 40%, the magnetization increase remarkable. It is indicated that higher deformation causes more significant magnetization. In the case of irradiation, with increasing irradiation, a similar magnetization behavior (continuously increase) can be observed. This indicates that deformation or irradiation will introduce ferromagnetic phase in ASS matrix.

In order to confirm the category of the ferromagnetic phase, GIXRD was used to obtain the phase transformation information. As shown in Fig. 3, it is indicated that a new diffraction peak corresponding to α(110) appears after irradiation and become remarkable gradually with irradiation damage increase. This suggests that a martensitic phase transformation (γ to α phase) occurs. Martensitic phase will also be formed after deformation. But due to a low content (as calculation below), it cannot be observed by GIXRD. Nevertheless, according to our VSM analysis and previous studies, we confirmed that the ferromagnetic phase should be the martensitic phase.

To evaluate the amount of ferromagnetic phase, the saturation magnetization (M_s) was obtained from the horizontal tangent line of the hysteresis loops. According to this method, the magnetization of the deformed and irradiated specimens after subtracting the paramagnetic contribution (equivalent to magnetization of undeformed or unirradiated specimens) is shown in inset of Fig. 2, and the M_s obtained from these plots are shown in Fig. 4.

We usually take the M_s of 100 vol.% martensite to be about 127emu/g for material 316 stainless steel [6]. From the magnetic measurement results above, we can obtain the amount of ferromagnetic phase. But due to the inhomogeneous distribution of irradiation damage for irradiated specimens, the amount of ferromagnetic phase in our calculation is an average value after deducting the unirradiated domain. In fact, irradiation of Xe ions produces a near-uniform distribution of irradiation damage (the ratio of minimum and maximum damage is about 50% from the surface to peak damage region), so this approximate treatment is reasonable. It can be observed in Fig. 4 that the amount of ferromagnetic phase increases with strain and irradiation. However, the amount of ferromagnetic phase obtained here was very small in deformed stainless steel, 1.2 vol.% for strain up to 60%. This low content of ferromagnetic phase cannot be revealed by XRD (as shown in Fig. 3). Whereas in the case of irradiation, the amount of ferromagnetic phase obtained by M_s is about 18 vol.% at irradiation to 25dpa. This volume content is in consistent with previous conversion electron Mossbauer spectroscopy (CEMS) and TEM results [7].

Since ASS is paramagnetic and α-phase has ferromagnetism, it can be supposed that magnetic properties are determined by the volume of ferromagnetic phase and saturation magnetization M_s depends only on the amount of the ferromagnetic phase. In fact, previous studies indicated that the saturation magnetization is used as a structure-insensitive parameter that depends only on the volume amount of ferromagnetic phase [8]. Therefore, M_s is an excellent parameter to evaluate the percentage of ferromagnetic phase in ASS and NDE techniques can be exploited to analyze the saturation magnetization to detect ferromagnetic phase formation in deformed or irradiated stainless steel.

It has been proved that deformation or irradiation can induce ferromagnetic phase transformation. When an austenitic stainless steel is plastically deformed, an austenitic structure transforms into martensites under stress relief by shearing the lattice. As the plastic deformation progresses, martensite particles grow and form larger clusters, accompanying an increase of the volume fraction [9]. Whereas in the case of irradiation, due to the accumulation of radiation damage caused by collision cascades and subsequent defect evolution, additional stress will be introduced in irradiated domain and cause martensitic transformation. Meanwhile, as shown in Fig. 5, it is indicated that a high density of Xe bubbles with a size of about ~1 nm in white and black dots at underfocus and overfoucs mode is observed after Xe irradiated to 2 dpa. Such bubbles, which have a very high internal pressure, will induce extremely high internal stress levels in the surrounding matrix