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ScienceDirect
JOURNAL OF IRON AND STEEL RESEARCH, INTERNATIONAL. 2016, 23(9):981-987

Magnetic Properties of Thermally Aged Fe-Cu Alloys with Pre-deformation

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Abstract: Magnetic properties of thermally aged Fe-Cu alloys with pre-deformation have been evaluated to improve the understanding of using magnetic technology for the nondestructive evaluation (NDE) of irradiation embrittlement in reactor pressure vessel (RPV) steels. Fe-Cu alloys with and without pre-deformation were thermally aged at 500 ℃ and the changes in microstructure, mechanical properties and magnetic properties were determined. It is found that the strain-induced dislocations recover and the Cu-rich particles precipitate during the aging process, and the magnetic properties variation depends on the combined influence of these two factors. From the point of view of NDE, a fully tempered or annealed microstructure is favorable before RPV is put into service. These results improve the understanding of magnetic property evolution in actual RPV steels and help to develop NDE theory for irradiation embrittlement.
Key words: RPV steel; nondestructive evaluation; magnetic property; Cu-rich precipitate; dislocation

Irradiation embrittlement in reactor pressure vessel (RPV) due to nano-sized Cu-rich precipitates (CRPs) is one of the main concerns impacting the safe operation of nuclear reactors[1-3]. Charpy impact samples are prevailingly used to evaluate the embrittlement, but this procedure suffers from some disadvantages such as the uncertain dose rate effect[4-6] and the diminishing stock of the Charpy impact samples after life extension of nuclear power plants. Reactor-component-like RPV is irreplaceable during its service life; thus, it is of great meaning to monitor the embrittlement situation of RPV online and nondestructively. Magnetic nondestructive evaluation (NDE) technology could be a good candidate method because of its sensitivity to the evolution of lattice defects such as dislocations, precipitates and grain boundaries[7-9]. In view of the high radioactivity of the neutron irradiated RPV steels, thermally aged Fe-Cu alloys are commonly used to simulate the irradiated RPV steels[10-15]. It has been found that the coercivity and hardness of Fe-Cu alloys increase with CRPs precipitation and decrease with CRPs ripening[10,11]. However, the effects of other lattice defects, mainly dislocations, on magnetic properties of RPV steels after irradiation have been neglected. In fact, actual RPV steels possess bainite or martensite microstructure with dislocations, and these preexisting dislocations may rearrange their substructure[16] or decrease in their number density[17,18] during their service lives. Therefore, it is of crucial importance to study the combined influences of these lattice defects on magnetic properties. This work aimed to study the combined effect of dislocations and CRPs using thermally aged Fe-Cu alloys with pre-deformation, and to improve the fundamental understanding of NDE for irradiation embrittlement in actual RPV steels.

1 Experimental

The material used for this study was binary Fe-0.97%Cu alloy, which was made through melting of pure iron and copper in a vacuum induction furnace, followed by hot forging of the ingot into a plate. The plate was solution treated at 850 ℃ for 5 h and then water quenched. Uniaxial tensile test samples were cut from this solution treated plate and deformed at a strain rate of 6.7×10^{-4} s^{-1} up to a strain of 5% and 20%, respectively. Samples for Vickers hardness and electrical resistivity measurement were cut

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from the deformed tensile samples and the water quenched plate, and then isothermally aged at 500 ℃ for various time. Vickers hardness was measured on the electro-polished surface under a load of 3 N at eleven points and then averaged. Electrical resistivity was measured to characterize the volume fraction of CRPs. It was conducted at 25 ℃ using a source meter (Keithley, Model 6220) and a voltmeter (Keithley, Model 2182) in accordance with ASTM F76-08. Transmission electron microscope (TEM) was used to check the microstructure of the samples. Magnetic hysteresis loops (HLs) were measured using vibrating sample magnetometer (VSM) and the initial magnetization curves were measured using SQUID-VSM. Specimens with sizes of 6 mm×4 mm×0.8 mm were cut from the water quenched plate and the deformed tensile samples along the tension direction, carefully ground to a thickness of about 160 μm, electro-polished on both sides to a thickness of about 130 μm to ensure stress-free surfaces, and finally thermally aged at 500 ℃ in vacuum. A maximum magnetic field of 1000 Oe (79600 $A \cdot m^{-1}$) was applied along the tension direction to measure the HLs. The variation of the applied field between −3980 and +3980 $A \cdot m^{-1}$ was set as small as 79.6 $A \cdot m^{-1}$ to acquire a more accurate coercivity.

2 Results and Discussion

2.1 Microstructure evolution

Fig. 1 shows the stress-strain curves of the uniaxial tensile samples deformed up to strains of 5% and 20%, respectively. It is noted that uniaxial tension can produce a uniform microstructure without introducing macro residual stress compared with other deformation modes such as rolling and bending[19]. The samples in Fig. 1 were in the uniform elongation region far away from necking, which indicates that the specimens cut from the deformed samples are of the same and uniform original microstructure. The inset of Fig. 1 shows the TEM images of the solution treated and 20% pre-deformed Fe-Cu alloys. No signs of dislocation or precipitate can be found in the solution treated alloy, while after pre-deformation, the plastic strain produces a large number of dislocations tangled with each other.

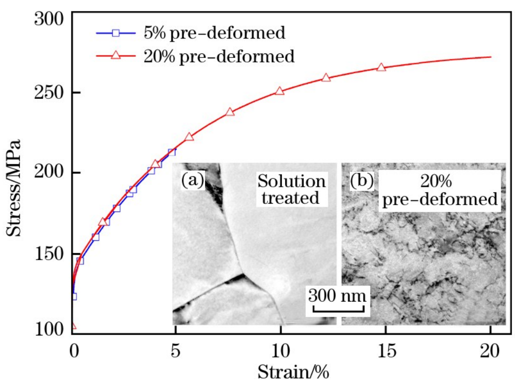

Fig. 1 Stress-strain curves of Fe-Cu alloys pre-deformed up to strains of 5% and 20% respectively; inset TEM images show microstructures of solution treated (a) and 20% pre-deformed (b) alloys

Fig. 2 shows the microstructure evolution of 20% pre-deformed alloy when thermally aged at 500 ℃. After thermally aging, recovery process occurs as demonstrated in Fig. 2. The dislocation density drastically decreases after aging for 60 min, and at the same time, the dislocations slide to rearrange into loose dislocation walls, as shown in Fig. 2(b). Clear dislocation walls form with increasing the aging time, leaving very few dislocations within the regions between the walls, as shown in Fig. 2(c). The mean spacing between the walls increases with a further increase in aging time, which can be found by comparing Figs. 2(c) and 2(d). Recovery happens when the annihilation and restructuring of dislocations could drive the dislocation clusters into energetically more favorable configurations.

The variation of electrical resistivity (ER) was used to characterize the change in volume fraction of CRPs with aging time. It is reported that ER varies linearly with volume fraction (or mass fraction) of CRPs, i.e. one percent mass fraction of CRPs could reduce ER by 4×10^{-8} $\Omega \cdot m$[20,21]. Fig. 3 shows the change in ER with aging time. Both the CRPs and dislocations could affect the ER; however, the influence of dislocations can be ignored, which can be seen from the very little change in ER when 20% pre-deformation is introduced (see the points at 0.01 min in Fig. 3). ER decreases with increasing aging time, reflecting the precipitation of CRPs. Compared with the un-deformed alloy, ER decreases much faster in the initial aging stage for the 20% pre-deformed alloy, indicating that pre-deformation can accelerate the precipitation process. The reason for this may lie in the larger number of nucleation sites and faster diffusion velocity produced by the strain-induced dislocations. Fig. 4 shows the TEM images of Fe-Cu alloys aged for 30 min with or without pre-deformation. It can be found that more CPRs appear in the alloy with pre-deformation (Fig. 4(b)) and the mean size of CRPs seems to be a little larger than that in the alloy without pre-deformation (Fig. 4(a)). The dis-