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Evaluation of Embrittlement in Isochronal Aged Fe-Cr Alloys by Magnetic Hysteresis Loop Technique

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Fe-Cr alloys with different Cr contents were prepared by an arc melting technique. The alloys were isochronally aged in the range from 400 °C to 900 °C with 50 °C steps with a holding time of 100 hours. The ageing produced embrittlement in the alloys due to either the formation of a Cr-rich α' phase or a σ phase at high temperatures. Magnetic Hysteresis Loop (MHL) and Micro-Vickers hardness were measured at each step to correlate the magnetic and mechanical properties. Coercivity and hardness of the alloys were increased and remanence decreased up to 500-550 °C due to formation of a Cr-rich α' phase. Beyond 500-550 °C range, the coercivity and hardness decreased and remanence increased due to the coarsening or dissolution of the Cr-rich α' phase. In the Fe-48% Cr alloy, formation of the σ phase at 700 °C reduced the maximum induction of the alloy significantly.

Keywords: Fe-Cr alloys, embrittlement, magnetic hysteresis loop, hardness

1. Introduction

Fe-Cr alloys are important engineering materials for nuclear and chemical industries for their superior mechanical properties, resistance to radiation induced swelling, and corrosion resistance [1-3]. Such materials experience 475 °C embrittlement due to phase separation of the solid solution into a Fe-rich α phase and a Cr-rich α' phase [4-7]. For the alloys with a Cr content of more than 20%, there is a possibility of forming a σ phase above 550 °C, which also depends on the ageing time and impurity level in the alloy [8]. The formation of the σ phase is detrimental as it makes the material brittle and affects the creep and corrosion properties [9]. The 475 °C embrittlement arises in structural components during service whereas the σ phase embrittlement is produced during heat treatment, welding, or casting. Both the embrittlements are undesirable and responsible for failure of components. Therefore, attempts are being made by researchers to develop the Non-Destructive Evaluation (NDE) of such microstructural changes for the safety of components. Since magnetic properties of ferromagnetic alloys are very sensitive to microstructural modifications and the stress state, the popularity of magnetic techniques is growing for the NDE of deformations by creep, fatigue, thermal ageing, and irradiation [10-14]. In our earlier paper we found a good correlation between changes in magnetic properties, particularly with Cr contents of 15% and 20% with thermal ageing at 475 °C [15]. In this investigation, Fe-Cr model alloys with higher Cr contents were prepared systematically by the arc melting technique and isochronally aged to produce the 475 °C embrittlement and σ phase embrittlement. MHL measurements were carried out in specimens to find the feasibility of the technique for the two kinds of embrittlement. Micro-Vickers hardness measurement was also carried out and correlated with the MHL parameters with the aim of using MHL as a NDE tool for the evaluation of embrittlement in structural components made of Fe-Cr alloys.

2. Experimental

Fe-x wt.% Cr ($x = 20$, 30, 40 and 48) alloys were prepared by an arc melting furnace. Such alloys were solution annealed at 1000 °C/2 hours (hrs) in a high vacuum and then water quenched. The alloys were cut by an electric discharge machine in a ring shape (ID: 10 mm, OD: 15 mm, thickness: 2 mm) for magnetic measurement.

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Flat specimens were also prepared for hardness measurement and microstructural observation. All the specimens were subjected to isochronal ageing in the range from 400 to 900 °C with 50 °C steps with a holding time of 100 hrs. The microstructural observations and hardness measurements were carried out by optical microscopy and a micro-Vickers hardness tester with a 0.1 kg load, respectively. Seven data points for hardness were recorded at each step and averaged to see the scattering. The chemical compositions of the alloys were determined by electron probe micro analyzer (EPMA). Magnetic measurements were carried out using the MHL technique with ring specimens where the primary magnetizing coil and the secondary pick up coils were wound. Triangular wave currents were used for magnetization. The maximum field applied for the measurement was 6 kAm^{-1} and at 50 mHz frequency.

3. Result and Discussion

Fig. 1 shows the change in hardness of Fe-Cr model alloys with isochronal ageing. The first data points (at 20 °C) were the hardness of the as quenched specimens. Hardness of all the alloys increased with ageing temperatures up to 500 °C and then decreased, however the hardness of the Fe-48% Cr alloy was again increased abruptly at 700 °C and dropped at 850 °C. At 650 °C, the hardness of the Fe-48% Cr alloy was found to be completely different; higher values for the σ phase and lower values for the α phase (ferrite) were observed. The regime of isochronal ageing temperatures was divided into two parts: Regime-I at 20-600 °C and Regime-II at 600-900 °C. In Regime-I, the increase in hardness of all the alloys was due to the coherent stress growth for the formation of the Cr-rich α' phase [1]. With increases in ageing temperature, the Cr-rich phases increased and hence their volume fraction also increased in the alloys that were responsible for the increase in hardness. At higher ageing temperatures (above 500 °C), the Cr-rich phases were either dissolved in the Fe-20% Cr alloy or coarsened in higher Cr content alloys. The coherent stress completely vanished due to the dissolution of the Cr-rich phase or decreased when the coarsening of such a phase occurred due to the lack of coherency between the Cr-rich phase and the matrix.

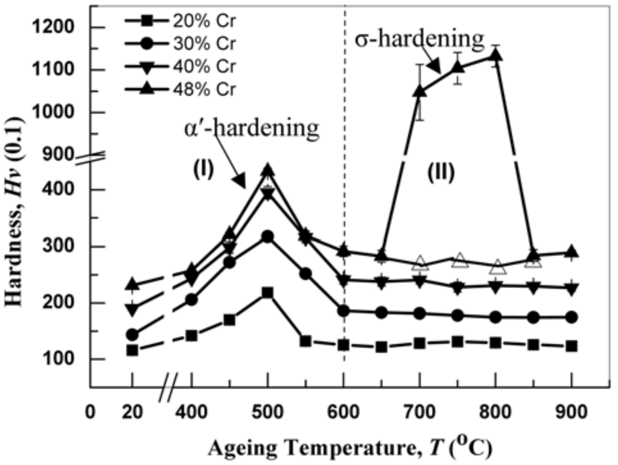

Fig. 1. Change in micro-Vickers hardness of the Fe-Cr alloys with isochronal ageing.

In Regime-II, there was almost no change in hardness observed for all the alloys except for the Fe-48% Cr alloy. For the Fe-48% Cr alloy, the hardness increased abruptly at 700 °C for the formation of the σ phase [1]. At 850 °C, the dissolution of the σ phase resulted in a sudden drop in hardness of the alloy. Fig. 2 shows the optical microstructure of the Fe-48% Cr alloy that was aged at 700 °C/ 100 hrs. Fig. 2a shows the low magnification image revealing the σ phase formation that covered most of the region and Fig. 2b shows the high magnification image where the indentation mark in the σ phase and α phase

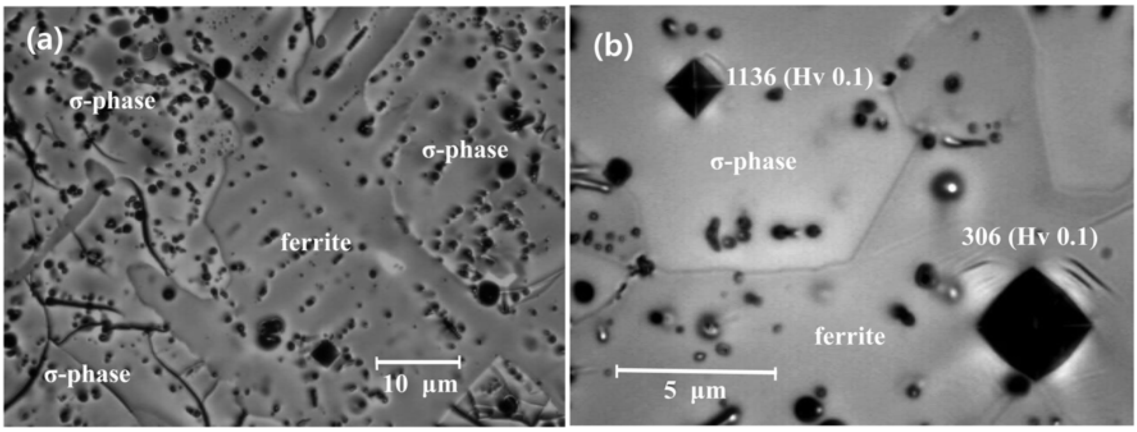

Fig. 2. Optical microstructure of the Fe-48% Cr alloy: (a) low and (b) high magnification images respectively.

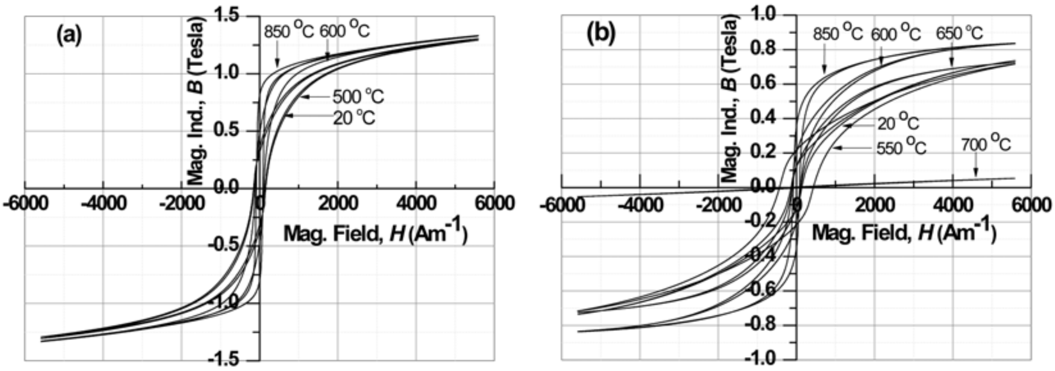

Fig. 3. Hysteresis loop of (a) Fe-20% Cr alloy and (b) Fe-48% Cr alloy at different ageing temperatures.

are drastically different. The higher value (1136 Hv) of hardness was for the σ phase and the lower value (306 Hv) was for the α phase. In alloys with lower Cr contents, no increase in hardness was found, indicating that the σ phase did not form in the alloys. Although earlier results revealed the formation of the σ phase in the alloy containing a lower Cr content (34.9%), such formation occurred over a longer period of ageing (1000 hrs) [1]. In the present study, due to the inadequate ageing time, a σ phase was not produced in the alloys with lower Cr contents.

Fig. 3a and 3b show the magnetic hysteresis loop at different steps of ageing of Fe-20% Cr alloy and Fe-48% Cr alloy, respectively. The α' phase embrittlement and σ phase embrittlement can be clearly distinguishable from the loop. The α' phase embrittlement resulted in the magnetic hardening with an increase in the area of the hysteresis loop whereas the σ phase embrittlement was representative of a drastic fall of maximum induction. Fig. 4a shows the change in coercivity of the alloys with isochronal ageing temperatures. Very similar to the change in hardness in the alloys, the coercivity also increased in Regime-I. The coherent stress growth for the precipitation of the Cr-rich phase also hinders the domain wall motion to enhance the coercivity of the alloys. This shows that coercivity would be a good parameter for the evaluation of 475 °C embrittlement. However, the peak of the coercivity value for Fe-(30-48) % Cr alloys shifted to 550 °C, although the hardness peak was at 500 °C. At high temperatures, the Cr-rich phases were coarsened along with the change in their composition and morphology. With coarsening, spherical shape Cr-rich α' phases are transformed to plate-like structures in the Fe-20% Cr alloy [6]. Such plate like phases are expected to be interconnected due to the higher volume fraction in (30-48) % Cr alloys and act as the pinning sites for the domain wall motion to enhance the magnetic hardening in high Cr alloys. The Cr-rich α' phase is poorly magnetic, which transformed to non-magnetic after changing the alloy composition (80-90% Cr) [16]. Therefore, an increase in volume fraction of the Cr-rich α' phase resulted in a decrease in remanence and maximum induction of the alloys shown in Fig. 4b and 4c, respectively, due to the demagnetizing field produced by such a phase. There was a deviation between the hardness and coercivity of the high Cr (30-48%) alloys at 550 °C. Such discrepancy has to be taken into account during actual applications of the technique as a NDE tool. However, with proper calibration of the hardness and coercivity of the alloys the MHL technique would be a better tool for the evaluation of 475 °C embrittlement. The coercivity of all the alloys dropped at 600 °C due to the dissolution of the Cr-rich α' phase as can be inferred by the recovery of the maximum induction of the alloys. The dissolution of such a phase was earlier in the Fe-20% Cr alloy and resulted in an early (550 °C) decrease in coercivity and an increase in remanence or maximum induction of the alloy.

In Regime-II, a small decrease in coercivity and increase in remanence in lower Cr alloys were observed. Such magnetic relaxation was due to recoveries of dislocations at high temperatures. In the Fe-48% Cr alloy, the coercivity increased at 700 °C, and there was a sudden drop of maximum induction for the formation of a paramagnetic σ phase (Fig. 4c). At 650 °C, the maximum induction of the alloy was already starting to decrease, although the hardness did not change. Such a decrease in maximum induction was also found in the Fe-40% Cr alloy at 700 °C. This showed that a small volume fraction of the σ phase was difficult to detect by hardness measurement, whereas the magnetic technique would be a good tool for the evaluation. At 850 °C, the dissolution of the paramagnetic σ phase recovered the maximum induction and remanence of the alloys. The hardness and coercivity of the Fe-48% Cr alloy decreased with the disappearance of

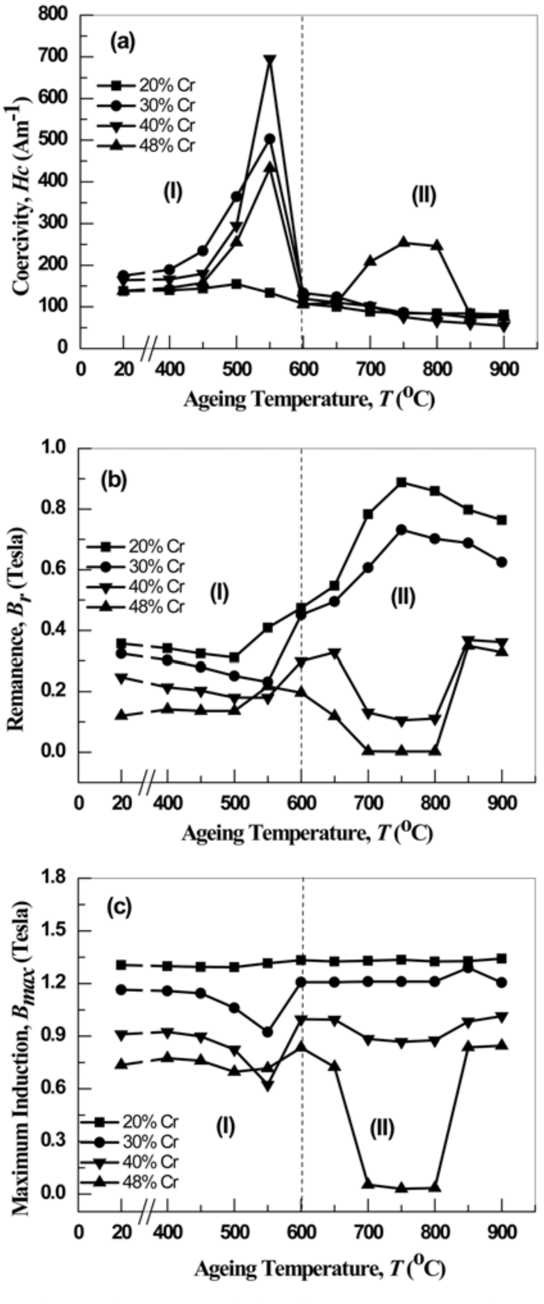

Fig. 4. Change in (a) coercivity, (b) remanence, and (c) maximum induction of the Fe-Cr alloys with isochronal ageing.

the σ phase.

4. Conclusion

Magnetic properties and hardness measurements were carried out in isochronally aged Fe-Cr alloys. Coercivity and hardness of all the alloys increased with the formation of a Cr-rich α' phase. In Fe-48% Cr alloy, the formation of the σ phase after 650 °C drastically reduced the maximum induction for its paramagnetic nature where coercivity and hardness increased. A very good correlation between the coercivity and hardness was found with the formation of the Cr-rich α' phase and σ phase, indicating that the MHL technique would be a good tool for the evaluation of 475 °C embrittlement and σ phase embrittlement in structural components made of Fe-Cr alloys.

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