EVALUATION OF THERMAL EMBRITTLEMENT IN FE-CR ALLOYS BY MAGNETIC AND ELECTROMAGNETIC ACOUSTIC RESONANCE TECHNIQUES

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ABSTRACT

Isothermal ageing was carried out in Fe-20% Cr and Fe-48% Cr alloy to observe the Cr rich α’ phase embrittlement and σ phase embrittlement by magnetic hysteresis loop (MHL) measurement. Isochronal ageing was carried out in Fe-48% Cr alloy to observe the embrittlements by electromagnetic acoustic resonance (EMAR) measurement. Micro Vickers’s hardness measurement was carried out in the samples to compare the change in mechanical properties with the magnetic and acoustic properties. The change in coercivity and resonant frequency showed a good correlation with the change in hardness due to the formation and growth of Cr rich α’ phase. Formation and growth of σ phase decreased the maximum induction. Estimation revealed that the MHL measurement can detect even 1% of σ phase in the alloy.

Key words: Fe-Cr Alloys, Thermal Embrittlement, Magnetic Hysteresis Loop, EMAR, Micro Hardness
PACS: 75.50.Bb, 62.20.mj, 75.60.Ej, 75.80. +q, 62.20.Qp

INTRODUCTION

Fe-Cr alloys are the candidates for the new generation fission and fusion nuclear reactor materials for their excellent mechanical properties, corrosion resistance and resistance to radiation induced swelling [1, 2]. Such materials are suffering from embrittlement due to phase separation of Fe-rich α and Cr-rich α’ phase in the temperature range 300-500°C and formation of σ phase in the temperature range 650-850°C [3-6]. Both the phenomena lead to deterioration of mechanical properties of the materials vulnerable to failure of the components. For the safety of the components there is a need to inspect the health of the components nondestructively. Since the conventional non-destructive evaluation (NDE) techniques are not suitable for the detection of microstructural changes in a very fine scale, advanced NDE techniques like magnetic and electromagnetic acoustic resonance are growing demand [7-12].

In the present investigation, Fe-Cr alloys were subjected to isothermal and isochronal ageing to produce the Cr rich α’ phase embrittlement and σ phase embrittlement. MHL and EMAR measurements were carried out to observe the feasibility of both the techniques for the detection and evaluation of embrittlements in Fe-Cr alloys. Micro-Vickers hardness measurements were carried out at various heat treated conditions to correlate the mechanical properties with the magnetic and acoustic properties.

EXPERIMENTAL

Fe-x% Cr alloys (x = 20, 48) were prepared by arc melting technique in argon atmosphere. The alloys were cut by electric discharge machine in the form of ring (thick: 2.0 mm, ID: 10.0 mm, OD: 15.0 mm) samples for magnetic and flat \((L_1 \times L_2 \times L_3 = 7.0 \, \text{mm} \times 2.0 \, \text{mm} \times 5.0 \, \text{mm})\) samples for EMAR measurement. Isothermal ageing was carried out in Fe-20% Cr alloy at 475 °C and 500 °C up to 1000 hrs and in Fe-48% Cr alloy at 700 °C up to 200 hrs. Isochronal ageing was carried out in Fe-48% Cr alloy between temperature range 400 to 700°C with 50 °C step and 1hr holding time. MHL measurements were carried out at 50 mHz frequency and 6 kA/m magnetizing field using triangular current. The EMAR measurements were carried out at 0.25 to 1.5 MHz. Flat specimens were used for hardness measurement at a load of 0.1 kg. The hardness measurements were carried out on the samples randomly at 10 places for Fe-20% Cr alloy and at 30 places for Fe-48% Cr alloy. The average value of the hardness with the standard deviations is given in this study.
RESULTS AND DISCUSSION

α′ phase embrittlement in Fe-20% Cr alloy

Fig. 1 shows the change in hardness of the Fe-20% Cr alloy aged at 475 °C and 500°C with ageing time. The hardness of the alloy increased with the increase in ageing time and the rate of increase in hardness at 500°C is higher than the hardness at 475 °C due to the accelerated form of embrittlement at higher temperature. The change in the coercivity and remanence of the alloy with ageing time is shown in the Fig. 2. The coercivity increased and the remanence was decreased in the alloy with the progress of ageing time.

\[ \alpha' \] phase embrittlement in Fe-20% Cr alloy

The Fe-20% Cr alloy aged at 475 °C, with the progress of ageing, nucleation of fine Cr rich α′ phase precipitation took place resulted in the production of coherent stress in the alloy. The Vickers hardness depends on the dislocation mobility under the local stress due to the indentation. Precipitates act as obstacle to dislocation displacement and restrict the dislocation mobility. The mobility decreases with increasing the number density and size of precipitates [13]. As the number of precipitates increases with ageing time the hardness of the alloy was increased. After the growth of such precipitates the dislocation experience strong pinning and hence the hardness increase more rapidly. At 500°C, the nucleation and growth process occurs simultaneously and hence the hardness increase with ageing time. At higher ageing periods the rate of increase in hardness becomes slow due to the decrease in coherency between the matrix and the coarser precipitates.

The precipitates also hinder the domain wall motion and increase the magnetic hardness. The coercivity in the alloy increased and remanence in the alloy decreased with the increase in size and number density of the Cr rich α′ phase precipitates. The coercivity increased more sharply in the alloy aged at 500°C, compared to the alloy aged at 475°C due to the increased in size of the precipitates at high temperature. The effect of size of the precipitates is much higher compared to the number density reported earlier [9]. Hence with increase in size of the precipitates the strong pinning observed by the domain wall resulted in rapid increase in coercivity in the alloy.

A plot between the coercivity and hardness of the alloy aged at two different temperatures is shown in Fig. 3. A linear relation between the coercivity and hardness indicates that the MHL can be a powerful NDE tool for the evaluation of thermal embrittlement in Fe-Cr alloys. It can be seen that the alloy aged at 500°C showed a shift to right with the progress of ageing. This can be explained by the increase in size of the precipitates at higher temperature due to the growth mechanism. The size of the precipitates has a strong effect of pinning for the domain wall motion than the dislocation motion. Hence the coercivity is increased more sharply than the hardness of the alloy aged at higher temperature.

α′ phase embrittlement in Fe-48% Cr alloy

The change in hardness of the alloy with isochronal ageing is shown in Fig. 4(a). The hardness of alloy increased with isochronal ageing up to 500 °C due to the formation of coherent Cr rich α′ phase which arises in the alloy by Fe-Cr phase separation. The increase in Cr rich α′ phase enhances the coherent stress in the alloy and hence the hardness of the alloy was increased. Above 500°C, the coherent stress disappears and hence the hardness of the alloy decreased.

The change in resonant frequency of the alloy with isochronal ageing is shown in Fig. 4(b). The resonant frequency increased similar to the change in hardness in the alloy up to 500°C and then decreased. The resonant frequency is related to both the elastic constant and the specimen size. The specimen size measured by micrometer did not change with ageing. Hence,
mechanical properties [14]. In duplex stainless steel 1% of $\sigma$ phase can affect the toughness of the steel and 10% of the $\sigma$ phase can make the steel completely embrittled. The study inferred that the MHL can detect even 1% of $\sigma$ phase in Fe-Cr alloys however up to 20% of $\sigma$ phase is difficult to detect by hardness measurement.

**CONCLUSION**

Fe-Cr alloys were subjected to various thermal treatments to produce the Cr rich $\alpha'$ phase and $\sigma$ phase. The formation and growth of both the phase increased the mechanical hardness of the alloys. An increase in coercivity and resonant frequency were observed with the formation and growth of Cr rich $\alpha'$ phase. A rough estimation indicates that 1% of $\sigma$ phase can be detected by MHL measurement. The results revealed that coercivity and resonant frequency would be suitable parameters for the detection and evaluation of Cr rich $\alpha'$ phase embrittlement whereas maximum induction would be a suitable parameter for the detection and evaluation of $\sigma$ phase embrittlement in Fe-Cr alloys.

**ACKNOWLEDGEMENT**

This work is supported in part by Japan Society for the Promotion of Science and Korea Science and Engineering Foundation under the contract of the Japan-Korea Basic Scientific Cooperation program and the Grants-in-Aid for JSPS Fellows, KAKENHI (22.00390).
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