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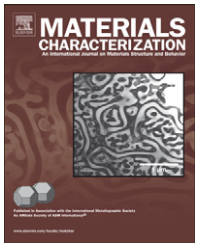

Phase decomposition in an Fe-40 at.% Cr alloy after isothermal aging and its effect on hardening

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ABSTRACT

The phase decomposition process of an Fe-40 at.% Cr alloy was studied after isothermal aging at 475 and 500 °C using a high-resolution transmission electron microscope, as well as hardness measurements. High-resolution transmission electron microscope observations showed that the hardening behavior is associated with the formation of the nanometric coherent decomposed Cr-rich and Fe-rich phases with irregular shape and interconnected as expected for a spinodally-decomposed alloy. As the aging progressed, coherent rounded Cr-rich phase precipitates were observed in the Fe-rich phase matrix. The coarsening process of the Cr-rich phase was observed for aging times up to 750 h. Nevertheless, no decrease in hardness with time was observed because of the nanometric size of the Cr-rich phase, less than 10 nm. Aging hardening was higher at 500 °C because of the higher decomposition kinetics.

1. Introduction

Fe–Cr alloys are susceptible to embrittlement when they are aged at temperatures in the range of 300 and 500 °C [1]. This embrittlement has been known as 475 °C-embrittlement. This is attributed to the phase decomposition of the solid solution into an ultrafine mixture of Cr-rich and Fe-rich phases. This type of microstructure can also be carried out by phase separation via the nucleation and growth mechanism (between ~10 and 20 at.% Cr) [2,3]. As a result of the aging process, Fe–Cr alloys are also hardened due to the phase separation of the supersaturated solid solution into a mixture of the Cr-rich and Fe-rich phases. Thus, the analysis of phase decomposition seems to be an important issue to understand the embrittlement process in this type of alloys. Several studies have been conducted to analyze the phase decomposition process in this alloy system by several characterization techniques [4,5]. For instance, Miller et al. [4] studied the phase decomposition by atom-probe field ion microscope in an Fe-45 at.% Cr alloy aged at 400 and 500 °C. They observed that the phase decomposition took place via the spinodal decomposition mechanism. Besides, the growth kinetics of phase decomposition was slow at 400 °C. Ustinovshikov et al. [2,3] reported that the spinodal decomposition was observed to occur in Fe-20 to 50 at.% Cr alloys after aging at 475 °C. The aging process of these alloys at 550 °C caused the phase separation via the nucleation and growth mechanism. Thus, the objective of present work is to characterize the phase decomposition by HR-TEM in an Fe-40 at.% Cr alloy after isothermal aging and its effect on the hardening behavior.

2. Experimental Procedure

An Fe-40 at.% Cr alloy was prepared by melting of pure Fe (99.99%) and Cr (99.99%) with a mini-arc melting furnace. Alloy was remelted several times in order to obtain a homogenous composition. The ingot was homogenized at 1100 °C for 240 h. The chemical composition of alloy was confirmed by chemical analysis. Specimens were encapsulated in quartz tubes under an argon atmosphere. These specimens were solution treated at

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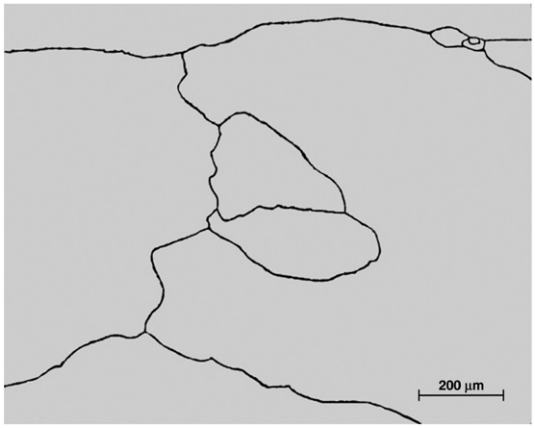

Fig. 1 – Optical micrographs of the as-cast Fe-40 at.% Cr alloy.

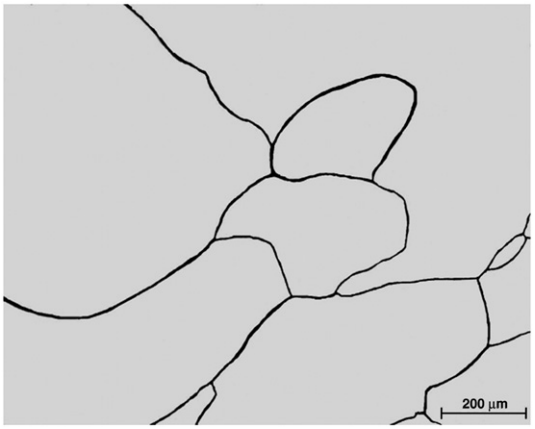

Fig. 3 – Optical micrographs of the Fe-40 at.% Cr alloy solution treated and subsequently aged at 500 °C for 750 h.

1000 °C for 2 h and subsequently quenched in water. The aging treatments were performed at 475 and 500 °C for times from 10 to 500 h. Heat treated specimens were observed with a HR-TEM at 300 kV and optical microscope. TEM specimens were prepared by electropolishing with an electrolyte composed of 33 vol.% nitric acid in methanol at −35 °C. Whereas the specimens for optical microscope observation were prepared metallographically and then etched with a solution of 5 g $FeCl_3$ in 50 ml HCl and 100 ml distilled water. Etched specimens were observed in an optical microscope equipped with a commercial image analyzer. Vickers hardness testing with a load of 100 g for 12 s, HV0.1/12 s, was used to follow the hardening behavior. Hardness measurements were conducted ten times for each heat treated specimen.

3. Results and Discussion

3.1. *Microstructural Characterization*

Figs. 1–3 show the optical micrographs for the Fe-40 at.% Cr alloy in the conditions of as-cast, solution treated and subsequently quenched, and then aged at 500 °C for 750 h, respectively. Columnar grains of ferrite, bcc Fe, can be seen instead of the typical dendritic microstructure. This fact is attributed to the melting process, which involved several steps of heating and slow cooling. The solution treated specimen only shows a coarse polycrystalline α–Fe phase, as expected from the equilibrium Fe–Cr phase diagram [6], shown in Fig. 4. The aging process of the solution treated specimen is expected to cause the phase decomposition into Fe-rich and Cr-rich phases, as shown in Fig. 4; however, the size of the decomposed phase is of the nanometric order, as shown later, and therefore only a polycrystalline microstructure is observed in the aged specimen.

Fig. 5 shows the HR-TEM micrograph of the specimen aged at 500 °C for 25 h and its corresponding simulated electron diffraction pattern. The morphology of the decomposed phases is irregular and interconnected. This type of morphology is a characteristic of a spinodally-decomposed alloy system [7–9]. Additionally, the same type of morphology was observed to occur in the aged Fe–Cr alloys by Miller et al. [4]. This micrograph also indicates that there is a coherent interface between the decomposed phases, indicated by the continuity of atomic planes. This coherent interface has been also observed during the early stages of decomposition of several spinodally-decomposed alloys [7–9]. The electron diffraction pattern corresponds to a bcc crystalline structure with a zone axis [11 $\bar{1}$]. Likewise, the diffraction spots

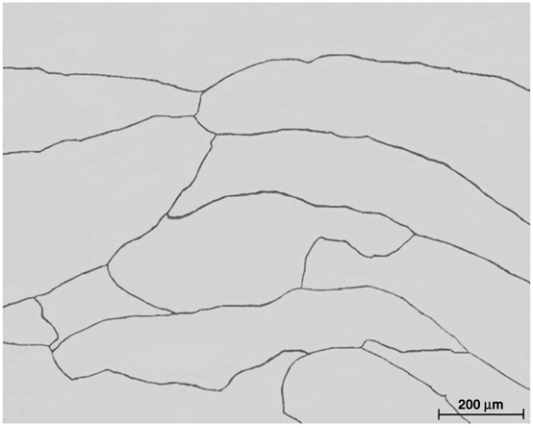

Fig. 2 – Optical micrographs of the solution treated Fe-40 at.% Cr alloy.

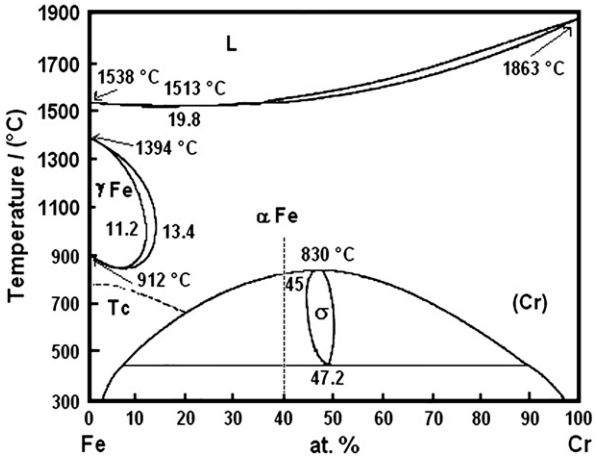

Fig. 4 – Equilibrium Fe–Cr phase diagram [6].