Grain Boundary Precipitation in an Fe-28Mn-8Al-1C-1.25Si Alloy

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Abstract

The aging of fully austenitic FeMnAlC alloys at temperatures ranging from 500°C to 750°C for a short period of time produces a fine structure of $(Fe,Mn)_3AlC$ carbide (κ-phase) precipitation within the austenitic matrix, but not on grain boundaries. However, with increasing aging times a α+κ, or α+κ+β-Mn transition has been detected on grain boundaries. In order to improve the strength, corrosion resistance and high-temperature oxidation, silicon has been added to Fe-Mn-Al-C alloys. In the present work, the grain boundary precipitation in an austenitic Fe-28Mn-8Al-1C alloy with 1.25%Si was investigated by transmission electron microscopy (TEM) and atomic and magnetic force microscopy (AFM and MFM). Isothermal holding at temperatures above 800°C results, in the initial stages, in a mixture of α+κ-phase in the grain boundaries, followed by a gradual α→B2 transition. At 700°C, isothermal holding gives origin to κ-phase and B2, which gradually transforms to DO_3, detected by magnetic force microscopy. Therefore, it is concluded that Si addition stabilizes the DO_3 phase only at temperatures below 700°C.

Keywords: FeMnAlC alloys, microstructure, grain boundary precipitates

Introduction

Alloys of the FeMnAlC system (usual compositions 25-30% Mn, 8-10% Al and 0.8-1.1% C) have been studied for over 40 years (1-4). They are 15% lighter than stainless steels, present good oxidation resistance and good mechanical characteristics. Such alloys have been developed for different uses, from cryogenic temperatures up to 673K, with specific composition (which may include small additions of refractory elements) recommended for each specific application. More recently, the possibility of adopting such alloys for soft-magnetic applications (5) and for structural purpose (6) has attracted considerable attention, due to the possibility of controlling phase transformation reactions of the supersaturated austenite during thermal and thermomechanical processing.

Phase transformations in fully austenitic FeMnAlC alloys have been extensively studied by many workers (7-13). When aged at temperatures ranging from 500°C to 750°C for short times, fine $(Fe,Mn)_3AlC$ carbides (κ-phase, with an $L'1_2$ structure) are observed within the austenitic matrix, but not on grain boundaries. However, with increasing aging times, two more phases may appear, α(bcc) and β-Mn, in such a way that a α+κ, or α+κ+β-Mn transitions have been detected on grain boundaries. Within the temperature range 700°C-850°C, three phases may coexist, namely γ (fcc), κ phase particles and α (bcc). Above 850°C, steels with 28-30%Mn, 8-10%Al and 1%C are fully austenitic.

In order to improve mechanical strength, corrosion resistance, and high-temperature oxidation resistance, silicon has been added to Fe-Mn-Al-C alloys. The available information concerning the effects of Si addition points to the fact that such addition results in the appearance at austenitic grain boundaries of segments in which α-phase (1, 14), ordered B2-phase (15), and a mixture of (α + DO_3) are present. Increasing the temperature, the DO_3 phase can further decompose successively as $DO_3 \rightarrow (DO_3 + \kappa) \rightarrow B2 \rightarrow \alpha$ (16, 17).

In the present work, the grain boundary precipitation in an austenitic Fe-28Mn-8Al-1C alloy with 1.25%Si was investigated by transmission electron microscopy (TEM) and atomic and magnetic force microscopy (AFM and MFM) and X-ray diffraction pattern.

Materials and Methods

A 5 kg ingot of Fe-28Mn-8.5Al-1C steel with 1.25% Si was prepared in a vacuum induction furnace under controlled argon ambient. It was further homogenized for 6 hours at 1150°C and then hot-rolled to the final thickness of 13 mm. After cutting into billets, the parts were solution treated at 1050°C for 2 hours and water quenched. Subsequent aging was performed in salt baths followed by water quenching.

Metallographic specimens were subjected to etching in a 5%-solution of nitric acid in ethyl alcohol. An Olympus BX60 optical microscope and a MEV-ZEISS 940-A electron scanning microscope were applied for microstructural observation. A 200kV JEOL 2000 FX microscope was used for transmission electron microscopy. Observations based on atomic force and magnetic force were performed with a TMX 2010 Discoverer Topometrix Scanning Probe Microscope operating in the noncontact mode. X-ray diffraction patterns were obtained using Co-κ_α radiation with a graphite monochromator in a Siemens diffractometer, operated at 40KV and 20 mA.

Electron microscopy specimens were prepared by means of a double-jet electric polisher with an electrolyte containing 60% of ethanol, 30% of acetic acid, and 10% of perchloric acid. The polishing temperature and the electric-current density were maintained within the limits from –10°C to +10°C and from 1.5×10^4 A/m^2 to 2.0×10^4 A/m^2, respectively.

Results and Discussion

The grain boundary phase transformations in an alloy with composition Fe-9.8Al-28.6Mn-1.0C-0.8Si have been studied (13), and a $\gamma \rightarrow DO_3 + \kappa$ transition was reported to occur after aging at 750°C. When aging at 800°C, the grain boundary precipitates were a mixture of (B2+κ) or (α+κ) phases. In the present work a preliminary study by x-ray diffraction was conducted in samples aged at 650°C and 800°C for 75 hours (figure 1), which correspond to regions of different reactions on grain boundaries. At 650°C it is observed well-defined superlattice reflections of DO_3 phase, while at 800°C the phases present are (κ+α) or (κ+B2).

In order to better understand the transformations at 800°C, a transmission electron microscopy study was carried on. Figure 2 shows a bright field electron micrograph of the alloy aged at 800°C for 20 minutes and then quenched, revealing the presence of grain boundary precipitates.

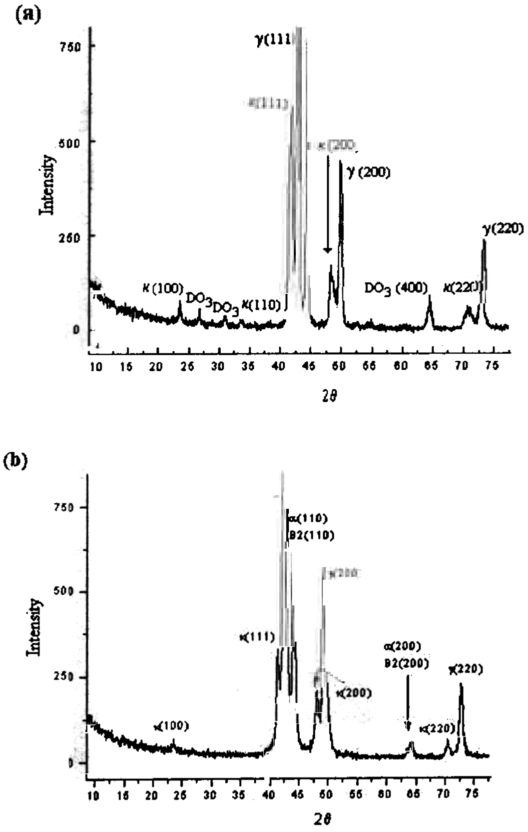

Figure 1. X-ray diffraction profiles of (a) alloy aged at 650°C and (b) at 800°C for 75 hours.

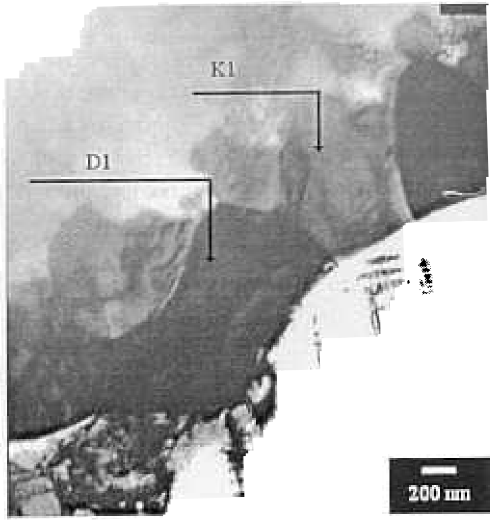

Figure -2 Electron micrograph after aging at 800°C for 20 min. Bright field electron micrograph.

The particles marked by K_I and D_I were selected for characterization. The analysis of selected-area diffraction pattern (figure 3) of the K_I particle indicates that it is the ordered κ phase. The same analysis performed in the D_1 area (figure 4) reveals that the diffraction pattern could be interpreted as a single DO_3 phase. However, as the DO_3 reciprocal lattice contains all the B2 and disordered α reflections, a more detailed analysis was realized. The contrast condition required for revealing the ordered phases and antiphase boundary structures in the B2 and DO_3 superlattices were first detailed by Marcinkowski and Brown (18). They have shown that the antiphase boundaries of the ordered B2 and DO_3 phases may be observed in dark field electron microscopy by imaging a superlattice reflection for which h, k and l are all odd. On the other hand, only the antiphase boundaries of B2 are observed when a superlattice reflection is imaged for which (h+k+l)=4n+2. The strongest reflections (figure 4) correspond to the families of plans {400} and {220} of the DO_3 phase and do not produce contrast of anti-phase DO_3 and B2 (18), but the {200} reflections presented produce contrast B2, so the domains in the area D_1 correspond to the B2 phase.

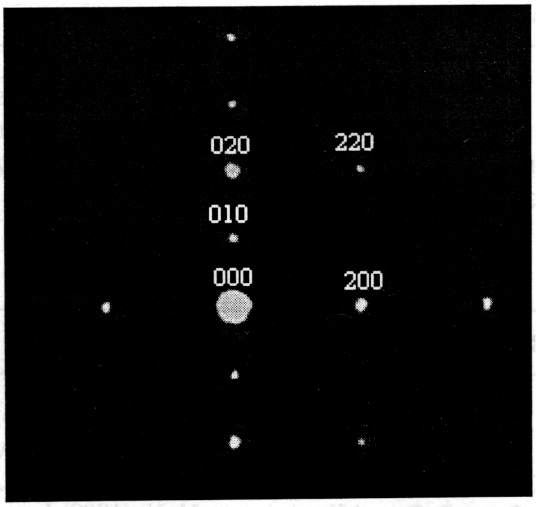

Figure -3 Electron micrograph after aging at 800°C for 20 min. Selected area diffraction pattern of the K_1 particle.

Figure 5 is a dark field electron micrograph of the same area of figure 1, obtained with {200} reflection, showing the contrast of small domains of the B2 phase. The DO_3 phase had also been observed in the Fe-Al, Fe-Al-Ti, Fe-Al-Mn and Fe-Al-Mn-C alloys (19). In these alloys, it has been observed that the DO_3 phase was formed by continuos ordering transition during quenching, through a $\alpha \rightarrow B2 \rightarrow DO_3$ transition. This indicates that up to 20 min at 800°C, therefore during the initial stages of austenite decomposition, we have a mixture of α + κ-phase in grain boundaries, followed by a gradual $\alpha \rightarrow B2$ transition. The DO_3 phase is formed during cooling to room temperature by the transformation $B2 \rightarrow DO_3$.

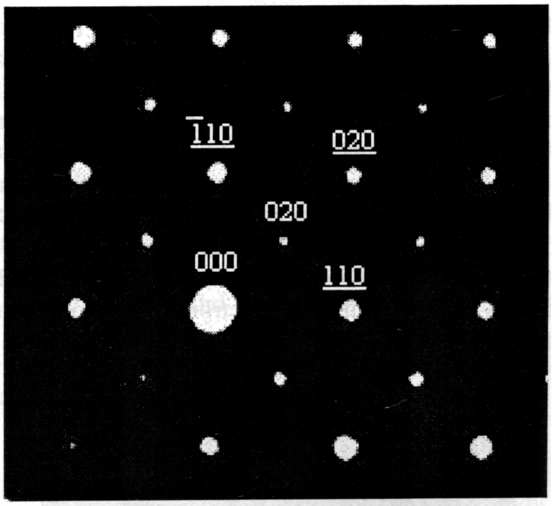

Figure -4 Electron micrograph after aging at 800°C for 20 min. Selected area diffraction pattern of the D_1 particle.

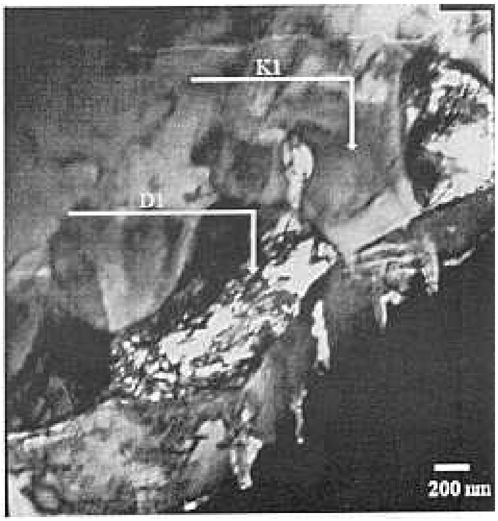

Figure -5 Electron micrograph after aging at 800°C for 20 min. Dark field microscopy using the {200} reflection.

In alloys of the same composition but with 0.8% Si, it has been reported that the addition of silicon not only enhances the formation of the ordered DO_3 phase but also stabilizes the DO_3 phase up to 750°C (19), which is much higher than the 550°C found in Fe-Al binary alloy. However, it is well-known that a small amount of silicon addition in binary Fe-Al alloy pronouncedly increases the energy of the a/4<111> antiphase boundaries of the ordered B2 phase (20,21). Figures 6 and 7 show the topography of Atomic Force Microscopy (AFM) and the image of magnetic domains by Magnetic Force Microscopy (MFM), of the alloy aged at 700°C for 10 hours. At 700°C, isothermal holding gives origin to κ-phase and B2, which gradually transforms to DO_3, leaving after 10 hours a residual fraction of B2, detected by magnetic force microscopy (figure 7). In this figure, the white areas are magnetic phases, while the dark ones are non-magnetic phases. In the matrix, it is noted the presence of magnetic phase, which is the coherent κ-phase (22). In the grain boundary the non-magnetic phases are the non-coherent κ-phase and DO_3 and the magnetic phase is B2. Therefore, it is concluded that Si addition stabilizes the DO_3 phase only at temperatures bellow 700°C. This result is in contradiction with other authors (19, 23), who, based only on transmission electron microscopy, have concluded that DO_3 is stable up to 750°C.

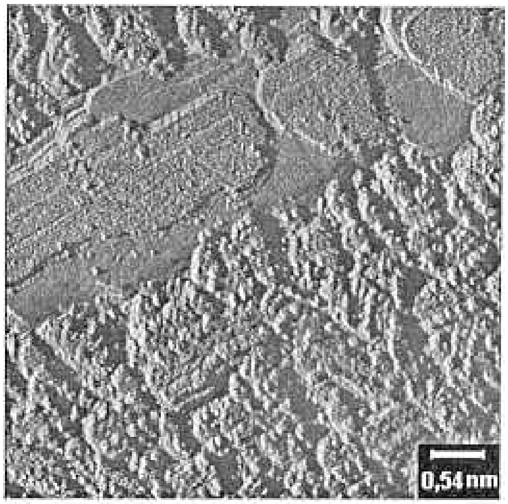

Figure -6. Grain boundary precipitation after aging at 700°C for 10 hours. Topography by AFM.

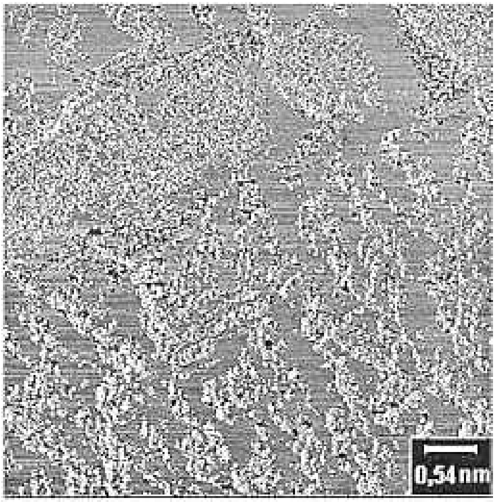

Figure -7. Grain boundary precipitation after aging at 700°C for 10 hours. Image of magnetic domains by MFM.

Conclusions

The grain boundary precipitation in an austenitic Fe-28Mn-8Al-1C alloy with 1.25%Si was investigated by transmission electron microscopy (TEM) and atomic and magnetic force microscopy (AFM and MFM). At 800°C, we have shown that grain boundary precipitates are κ-phase and B2. During the initial stages of austenite decomposition, we have a mixture of α + κ-phase in grain boundaries, followed by a gradual $\alpha \rightarrow B2$ transition. The DO_3 phase is formed during cooling to room temperature by the transformation $B2 \rightarrow DO_3$.

At 700°C, isothermal holding gives origin to κ-phase and B2, which gradually transforms to DO_3, leaving after 10 hours a residual fraction of B2. In the grain boundary the non-magnetic phases are κ and DO_3 and the magnetic phase is B2. Therefore, it is concluded that Si addition stabilizes the DO_3 phase only at temperatures bellow 700°C.

Acknowledgments

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