

Formation of (B2+D0₃) Two-Phase Microstructure in a Fe-23 Al-7 Ti Alloy

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As-quenched microstructure of the Fe-23 at% Al-7 at% Ti alloy was a mixture of (A2+D0₃) phases. When the as-quenched alloy was aged at 1073 K for moderate times, D0₃ domains grew preferentially along $\langle 100 \rangle$ directions and extremely fine B2 particles occurred at $a/2\langle 100 \rangle$ anti-phase boundaries (APBs). After prolonged aging at 1073 K, the B2 particles would grow to occupy the whole $a/2\langle 100 \rangle$ APBs. Consequently, the stable microstructure of the alloy at 1073 K was a mixture of (B2+D0₃) phases. [doi:10.2320/matertrans.MER2007133]

(Received June 14, 2007; Accepted August 24, 2007; Published October 11, 2007)

Keywords: phase transformation, electron microscopy, microstructure

1. Introduction

Effects of Ti addition on the microstructures of the Fe-rich Fe-Al binary alloys have been extensively studied by many workers.¹⁻⁹ Based on these studies, it can be generally concluded that the addition of Ti in the Fe-Al binary alloys would not only pronouncedly raise the A2+D0₃ (or D0₃) \rightarrow B2 \rightarrow A2 transition temperatures but also significantly expand the (A2+D0₃) phase field.³⁻⁹ In addition, a (B2+D0₃) two-phase field was reported to be detected in the Fe-Al-Ti ternary alloys.⁷⁻⁹ Interestingly, the (B2+D0₃) two-phase field has not been found by previous workers in the Fe-Al binary alloys before.¹⁰⁻¹² However, to date, the existence of the (B2+D0₃) two-phase field in the Fe-Al-Ti ternary alloys was determined principally by means of X-ray diffraction and electron-probe microanalysis (EPMA).⁷⁻⁹ Little transmission electron microscopy (TEM) information concerning the formation of the (B2+D0₃) two-phase microstructure has been provided in the literature. Therefore, the purpose of this work is an attempt to clarify the microstructural development for the formation of (B2+D0₃) phases in the Fe-23 at% Al-7 at% Ti alloy by TEM observation.

2. Experimental Procedures

The Fe-23 at% Al-7 at% Ti alloy was prepared in a vacuum induction furnace by using high purity (99.99%) constituent elements. After being homogenized at 1523 K for 48 h, the ingot was sectioned into 2-mm-thick slices. These slices were subsequently solution heat-treated at 1373 K for 1 h and then rapidly quenched into room-temperature water. The aging processes were performed at 1073 K for various times in a vacuum heat-treated furnace and then quenched rapidly. TEM specimens were prepared by means of double-jet electropolisher with an electrolyte of 67% methanol and 33% nitric acid. TEM observation of microstructure was performed on a JEOL JEM-2000FX TEM operating at 200 kV. This microscope was equipped with a Link ISIS 300 energy-dispersive X-ray spectrometer (EDS) for chemical analysis.

Quantitative analyses of elemental concentrations were made with a Cliff-Lorimer Ratio Thin Section method.

3. Results

An optical micrograph of the as-quenched alloy is shown in Fig. 1(a). Figure 1(b) is a selected-area diffraction pattern (SADP) of the as-quenched alloy, exhibiting the superlattice reflection spots of the ordered D0₃ phase.^{13,14} Figure 1(c) is a (1 $\bar{1}1$) D0₃ dark-field (DF) electron micrograph, revealing the presence of fine D0₃ domains with $a/2\langle 100 \rangle$ APBs. Figure 1(d), a (200) D0₃ DF electron micrograph, shows the presence of small B2 domains with $a/4\langle 111 \rangle$ APBs. Since the sizes of both D0₃ and B2 domains are small, it is suggested that these domains were formed during quenching.¹¹⁻¹⁷ In Fig. 1(d), it is also seen that a high density of disordered A2 phase (dark contrast) was present within the B2 domains; otherwise there would be no dark contrast within these domains by using a (200) superlattice reflection. Therefore, it is concluded that the as-quenched microstructure of the alloy was a mixture of (A2+D0₃) phases which were formed by an A2 \rightarrow B2 \rightarrow (A2+D0₃) transition during quenching. This result is similar to that reported by Mendiratta *et al.* in the Fe-(18~25)at% Al-5 at% Ti alloys quenched from 1373 K.³

When the as-quenched alloy was aged at 1073 K, the D0₃ domains grew rapidly, as illustrated in Fig. 2. Figure 2(a) is a DF electron micrograph obtained by use of the (200) superlattice reflection in [001] zone, revealing that the D0₃ domains grew preferentially along $\langle 100 \rangle$ crystallographic directions. This feature is also similar to that observed by Mendiratta *et al.*³ Figure 2(b), (1 $\bar{1}1$) D0₃ DF electron micrograph of the same area as Fig. 2(a) with a higher magnification, shows that the $a/2\langle 100 \rangle$ APBs are fully dark in contrast. Figure 2(b), a (200) D0₃ DF electron micrograph, reveals that a high density of extremely fine particles could be observed at the $a/2\langle 100 \rangle$ APBs. Since the amount of the particles was very small, the reflection spots of the particles were very faint. In order to carry out an unambiguous identification about the particles, prolonged aging at 1073 K was performed.

Figure 3(a) is a bright-field (BF) electron micrograph of the alloy aged at 1073 K for 16 h. In this figure, it is clear that the domains had grown to be very large and the morphology

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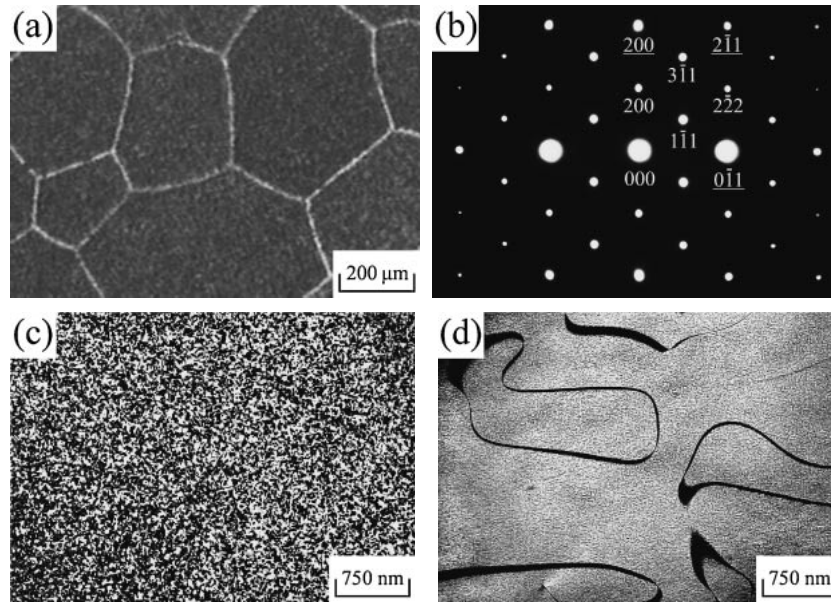


Fig. 1 (a) An optical micrograph of the as-quenched alloy, (b) through (d) electron micrographs of the as-quenched alloy: (b) an SADP. The foil normal is $[011]$. (hkl : disordered A2, hkl : $D0_3$ phase.), (c) and (d) $(\bar{1}\bar{1}1)$ and (200) $D0_3$ DF, respectively.

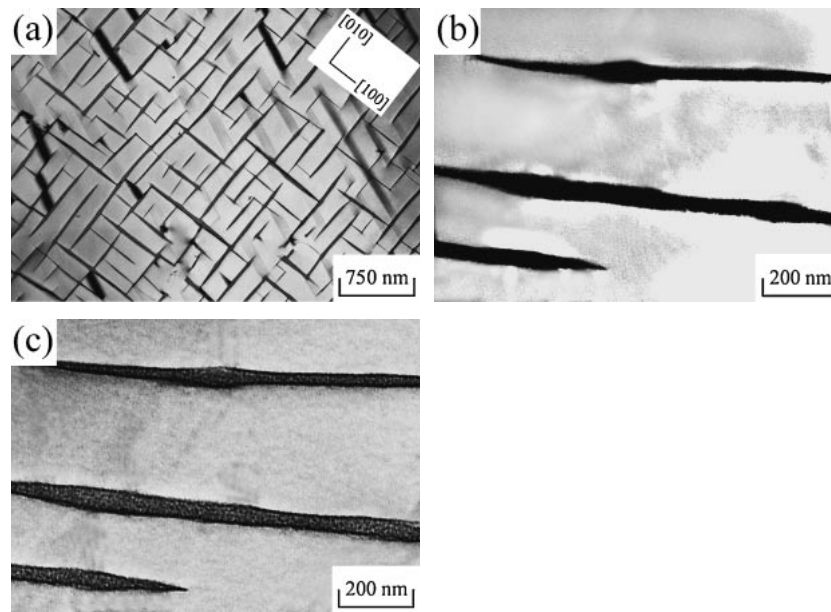


Fig. 2 Electron micrographs of the alloy aged at 1073 K for 1 h: (a) (200) $D0_3$ DF, (b) and (c) $(\bar{1}\bar{1}1)$ and (200) $D0_3$ DF with a higher magnification of (a), respectively.

changed from cubic to granular shape. Figures 3(b) and 3(c) are two SADPs taken from the areas marked as “D” and “B” in Fig. 3(a), respectively. In our previous study,¹⁷⁾ it was found that the intensity of the $(\bar{1}\bar{1}1)$ and (200) reflection spots of a single $D0_3$ phase should be almost equivalent. Therefore, it seems to be deduced that the reflection spots present in Fig. 3(b) should be of a single $D0_3$ phase. However, it is clearly seen in Fig. 3(c) that the (200) and $(\bar{2}\bar{2}2)$ reflection spots are much stronger than the $(\bar{1}\bar{1}1)$ reflection spot. Therefore, it is strongly suggested that the (200) and $(\bar{2}\bar{2}2)$ reflection spots should derive from not only $D0_3$ phase but also the B2 phase, since the $(\bar{1}\bar{1}1)$ reflection spot comes from the $D0_3$ phase only; while the (200) and $(\bar{2}\bar{2}2)$ reflection spots

can come from both the $D0_3$ and B2 phases (the (200) and $(\bar{2}\bar{2}2)$ $D0_3$ reflection spots are equal to the (100) and $(\bar{1}\bar{1}1)$ B2 reflection spots, respectively).^{11,12)} Figures 3(d) and (e) are $(\bar{1}\bar{1}1)$ and (200) $D0_3$ DF electron micrographs of the same area as in Fig. 3(a). It is obviously seen that at the regions marked as “D”, the $(\bar{1}\bar{1}1)$ DF image and the (200) DF image are morphologically identical, and these domains are fully bright in contrast. It means that these domains are of single $D0_3$ phase; whereas, at the regions marked as “B”, the B2 particles are much larger than those observed at $a/2\langle 100 \rangle$ APBs in Fig. 2(b), and the $D0_3$ particles are very extremely fine. This indicates that at the regions marked as “B”, the B2 particles were existent at the aging temperature, and the

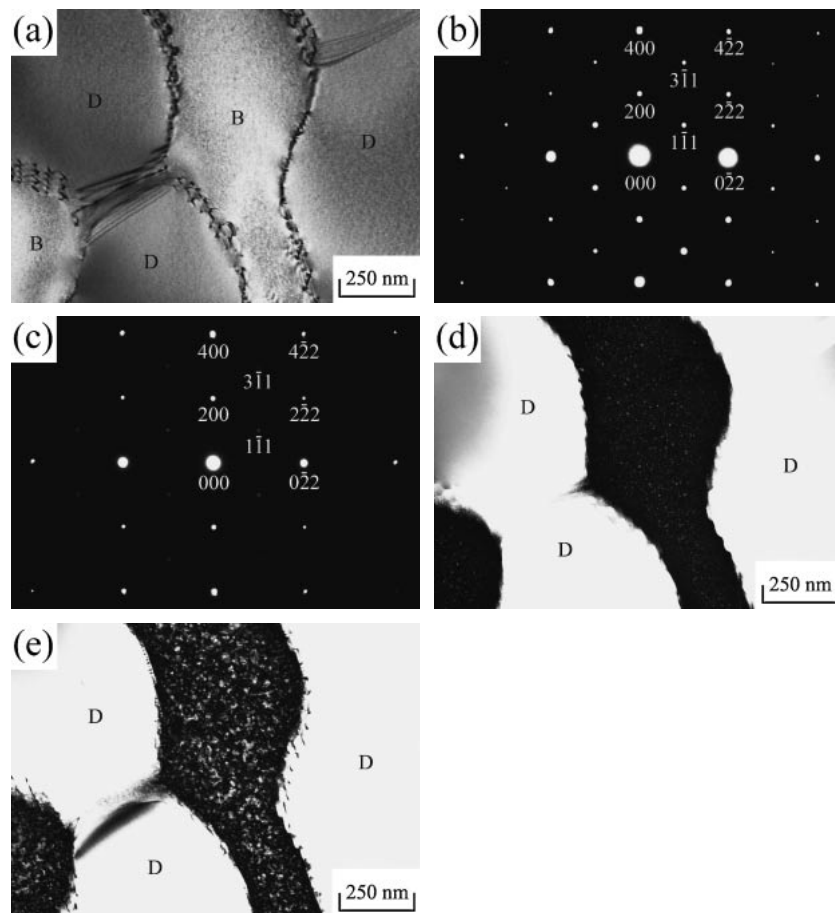


Fig. 3 Electron micrographs of the alloy aged at 1073 K for 16 h: (a) BF, (b) and (c) two SADPs taken from the areas marked as “D” and “B” in (a), respectively. (*hkl*: D₀₃ phase), (d) and (e) (111) and (200) D₀₃ DF, respectively.

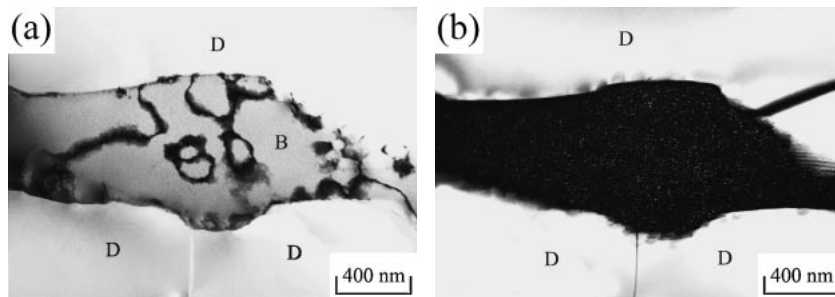


Fig. 4 (a) and (b) (200) and (111) D₀₃ DF electron micrographs of the alloy aged at 1073 K for 72 h.

extremely fine D₀₃ particles were formed during quenching from the quenching temperature by a B2 → D₀₃ ordering transition.^{11–17} With increasing the aging time at 1073 K, besides the presence of the well-grown D₀₃ domains, the B2 particles would grow to occupy the whole a/2<100> APBs and extremely fine quenched-in D₀₃ particles could be also detected within the B2 particles. A typical microstructure is illustrated in Fig. 4. Accordingly, the stable microstructure of the alloy present at 1073 K was a mixture of (B2+D₀₃) phases.

4. Discussion

Based on the preceding results, it is clear that when the

present alloy was aged at 1073 K for longer times, the D₀₃ domains existing in the as-quenched alloy grew and the B2 phase started to occur at the a/2<100> APBs. This transition behavior has never been observed by other workers in the Fe-Al and Fe-Al-Ti alloy systems before. In order to clarify this feature, EDS analyses were undertaken. The average concentrations of the alloying elements were obtained from at least ten different EDS profiles of each phase. The results are summarized in Table 1.

Obviously, it is seen in Table 1 that both the Al and Ti concentrations in the D₀₃ phase are much greater than those in the as-quenched alloy. It is thus expected that along with the growth of the D₀₃ domains, the concentrations of both Al and Ti at a/2<100> APBs would be lacked. The EDS

Table 1 Chemical compositions of the phases revealed by Energy-Dispersive X-ray Spectrometer (EDS).

Heat Treatment	Phase(s)	Chemical compositions (at%)		
		Fe	Al	Ti
as-quenched	A2+D0 ₃	69.90	23.08	7.02
1073 K, 72 h	D0 ₃	65.76	24.15	10.09
1073 K, 72 h	B2	76.35	20.13	3.52

examinations indicated that the elemental concentrations of Al and Ti in the B2 phase are 20.13 and 3.52 at%, respectively. According to the phase diagram of Fe-Al binary alloys,^{11,12)} it is seen that the microstructure of an Fe-20.13 at% Al alloy existing at 1073 K should be a single disordered A2 phase, and no evidence of B2 phase could be observed. Therefore, it is plausible to suggest that the existence of 3.52 at% Ti at a/2⟨100⟩ APBs would be favorable for the formation of the B2 phase, rather than the A2 phase.

Finally, it is worthwhile to point out that the B2 → D0₃ ordering transition could be found to occur in the Fe-Al binary alloys with Al > 25 at%.¹²⁾ However, it is clear in Fig. 4 that the B2 → D0₃ ordering transition could be detected and the Al content in the B2 phase was examined to be 20.13 at% only. This result implies that the existence of Ti would significantly lead the B2 → D0₃ ordering transition to occur with lower Al content.

5. Conclusion

The as-quenched microstructure of the Fe-23 at% Al-7 at% Ti alloy was a mixture of (A2+D0₃) phases. The (A2+D0₃) phases were formed by an A2 → B2 → (A2+D0₃) transition during quenching. When the alloy was aged at 1073 K for moderate times, the D0₃ domains grew preferentially along ⟨100⟩ directions and extremely fine B2 particles

occurred at a/2⟨100⟩ APBs. After prolonged aging at 1073 K, the B2 particles would grow to occupy the whole a/2⟨100⟩ APBs. Consequently, the stable microstructure of the alloy present at 1073 K was a mixture of (B2+D0₃) phases.

Acknowledgments

The authors are pleased to acknowledge the financial support of this research by the National Science Council, Republic of China under Grant NSC95-2221-E-009-086-MY3.

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