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Formation of (B2 + D0₃) phases at $a/2\langle 100 \rangle$ anti-phase boundary in an Fe-23 at.%Al-8.5 at.%Ti alloy

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The as-quenched microstructure of the Fe–23 at.%Al–8.5 at.%Ti alloy was a mixture of $(A2 + D0_3)$ phases. Transmission electron microscopy (TEM) examinations indicated that when the alloy was aged at 900 °C, the size of the D0₃ domains increased with increasing aging time, and an $A2 \rightarrow (A2 + D0_3) \rightarrow (B2 + D0_3)$ transition occurred at $a/2\langle 100\rangle$ anti-phase boundaries (APBs). This feature has never been reported by other workers in the Fe–Al–Ti alloy systems. © 2007 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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In order to improve the high-temperature oxidation and mechanical properties, several authors have added Ti to Fe-Al binary alloys [1-5]. Based on their results, it can be generally concluded that the addition of Ti can effectively improve these properties. In addition, the effects of Ti addition on the microstructures of Fe-Al binary alloys have also been studied by many workers [3-13]. It was reported that the addition of Ti would strongly increase the $D0_3 \rightarrow B2$ and $B2 \rightarrow A2$ transition temperatures [6–13] and expand the $(A2 + D0_3)$ phase field [10–12]. Furthermore, a $(B2 + D0_3)$ two-phase field was claimed to be existent in the Fe-Al-Ti ternary alloys [11–13]. It should be mentioned that the $(B2 + D0_3)$ two-phase field has not been found by previous workers in the Fe-Al binary alloys [14-16]. However, the existence of a (B2 + D0₃) two-phase field in Fe-Al-Ti ternary alloys was confirmed principally by using X-ray diffraction and electron-probe microanalysis [11-13]. In order to clarify the microstructural revolution for the formation of the $(B2 + D0_3)$ phases, a transmission electron microscopy (TEM) study was performed to investigate the phase transition in the Fe-23 at.%Al-8.5 at.%Ti alloy.

The Fe-23 at.%Al-8.5 at.%Ti alloy was prepared in a vacuum induction furnace by using pure Fe (99.9%), Al (99.9%) and Ti (99.9%). After being homogenized at 1250 °C for 48 h, the ingot was sectioned into 2 mm-

thick slices. These slices were subsequently solution heat-treated at 1100 °C for 1 h and then quenched rapidly in room-temperature water. The aging process was performed at 900 °C for various times in a vacuum heat-treated furnace followed by rapid quenching. TEM specimens were prepared by a double-jet electropolisher with an electrolyte of 67% methanol and 33% nitric acid. TEM observation was performed using a JEOL JEM-2000FX transmission electron microscope operating at 200 kV. Elemental concentrations were examined by using a Link ISIS 300 energy-dispersive X-ray spectrometer (EDS). Quantitative analyses of elemental concentrations for Fe, Al and Ti were made using the Cliff-Lorimer ratio thin section method.

Figure 1(a) shows a selected-area diffraction pattern of the as-quenched alloy, revealing the presence of the superlattice reflection spots of the ordered D0₃ phase [17]. Figures 1(b) and (c) are (111) D0₃ and (200) D0₃ (or, equivalently, (100) B2) dark-field (DF) electron micrographs of the as-quenched alloy, exhibiting the presence of fine D0₃ domains with $a/2\langle100\rangle$ APBs and small B2 domains with $a/4\langle111\rangle$ APBs, respectively [15,16]. In Figure 1(c), a high-density of disordered A2 phase showing a dark contrast could also be seen within the B2 domains. Accordingly, the as-quenched microstructure of the alloy was a mixture of (A2 + D0₃) phases. This is similar to what has been reported by other workers in as-quenched Fe–(18–22.5) at.%Al–5 at.%Ti alloys [10].

When the as-quenched alloy was aged at 900 °C for a short time, the D0₃ domains grew with preferred

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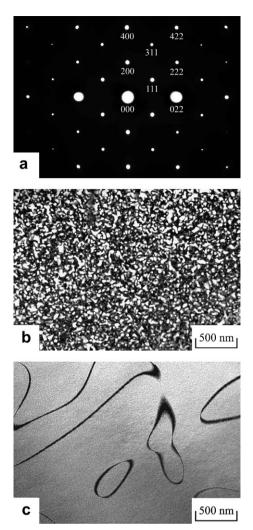


Figure 1. Electron micrographs of the as-quenched alloy: (a) a selected-area diffraction pattern. The foil normal is $[01\bar{1}]$ ($hkl = D0_3$ phase); (b) and (c) (111) and (200) D0₃ DF, respectively.

orientation, as shown in Figure 2. This feature is similar to that reported by other workers in aged Fe–Al–Ti alloys [10]. In Figure 2, it is also seen that the $a/2\langle 100\rangle$ APBs were coated with a continuous layer of the disordered A2 phase. However, after prolonged aging at 900 °C, some fine particles started to appear within the A2 phase. Figures 3(a) and (b) are (111) and (200)

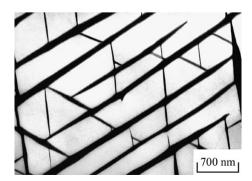


Figure 2. (200) D0₃ DF electron micrograph of the alloy aged at 900 °C for 0.5 h.

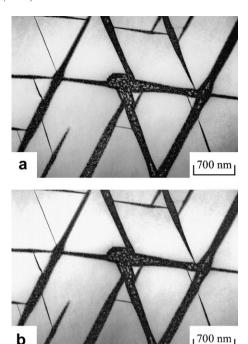


Figure 3. Electron micrographs of the alloy aged at 900 °C for 1 h. (a) (111) and (b) (200) D0₃ DF, respectively.

D0₃ DF electron micrographs of the alloy aged at 900 °C for 1 h, clearly revealing that the (111) D0₃ DF image and (200) D0₃ DF image are morphologically identical. Since the (200) reflection spot comes from both the B2 and D0₃ phases [10,12], while the (111) reflection spot comes only from D03 phase, the fine bright particles at $a/2\langle 100 \rangle$ APBs presented in Figures 3(a) and (b) are considered to be D0₃ phase. TEM examinations indicated that no evidence of the $a/4\langle 111 \rangle$ APBs could be observed. This result seems to imply that the B2 domains would grow up to the whole grains during aging. With increasing the aging time at 900 °C, the amount of the D0₃ particles at $a/2\langle 100 \rangle$ APBs increased and the disordered A2 phase decreased, as illustrated in Figure 4. Figure 5(a), a (111) D0₃ DF electron micrograph of the alloy aged at 900 °C for 24 h, indicates that at $a/2\langle 100 \rangle$ APBs the amount of the D0₃ particles increased considerably and a dark contrast could also be detected between the particles. However, a (200) D0₃DF electron micrograph (Fig. 5(b)) reveals that the whole regions of the $a/2\langle 100\rangle$ APBs were full bright in contrast. This indicates that the dark regions at $a/2\langle 100 \rangle$ APBs presented in Figure 5(a) should be of the B2 phase. Consequently, when the alloy was aged at 900 °C for a longer time, the microstructure at the $a/2\langle 100\rangle$ APBs was a mixture of (B2 + D0₃) phases.

The facts that with increased aging time at 900 °C the size of the D0₃ domains existing in the as-quenched alloy increased and an A2 \rightarrow (A2 + D0₃) \rightarrow (B2 + D0₃) transition occurred at $a/2\langle 1\,0\,0\rangle$ APBs are remarkable features in the present study. This feature has never been reported in an Fe–Al–Ti alloy system before. In order to clarify this feature, quantitative EDS analyses were undertaken. The results are shown in Table 1. It is seen in the table that when the alloy was aged at 900 °C for 0.5 h, the Al and Ti concentrations in the D0₃ domains

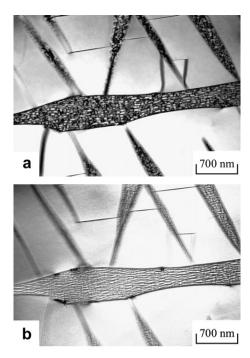
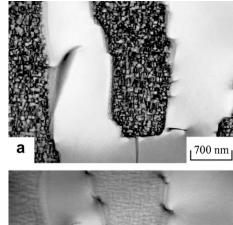


Figure 4. Electron micrographs of the alloy aged at 900 °C for 6 h. (a) (111) and (b) (200) D0₃ DF, respectively.



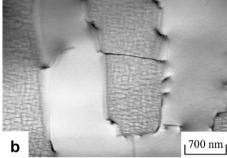


Figure 5. Electron micrographs of the alloy aged at 900 °C for 24 h. (a) (111) and (b) (200) D0₃ DF, respectively.

Table 1. Chemical compositions of the phases revealed by EDS

Heat treatment	Phase	Chemical composition (at.%)		
		Fe	Al	Ti
As-quenched	$A2 + D0_3$	68.3	23.1	8.6
900 °C, 0.5 h	D0 ₃ domain	62.5	25.1	12.4
	APB(A2)	75.9	19.8	4.3
900 °C, 1 h	D ₀₃ domain	63.5	24.9	11.6
	$APB(A2 + D0_3)$	73.6	20.2	6.2
900 °C, 6 h	D ₀₃ domain	64.4	24.3	11.3
	$APB(A2 + B2 + D0_3)$	71.6	21.5	6.9
900 °C, 24 h	D0 ₃ domain	65.1	23.8	11.1
	$APB(B2 + D0_3)$	70.2	22.4	7.4

were much greater than those in the as-quenched alloy, and these concentrations were noticeably lower at $a/2\langle 100\rangle$ APBs. The insufficient concentrations of both Al and Ti would cause the disordered A2 phase to form at $a/2\langle 100\rangle$ APBs. However, along with the growth of the D0₃ domains, partial Al and Ti atoms would proceed to diffuse toward the $a/2\langle 100\rangle$ APBs. EDS analyses indicated that during the early stage of isothermal aging at 900 °C, the increased amount of Ti at $a/2\langle 100\rangle$ APBs was more than that of Al. This implies that, during aging, Ti redistributed first and then Al started to move appreciably. This result is consistent with that found by other workers in Fe-Al-Ti ternary alloys [18], in which they reported that the diffusion of Ti is faster than that of Al. Furthermore, it is well known that a small Ti addition in the Fe-Al binary alloys would strongly enhance the formation of the D0₃ phase [10–12]. Therefore, it is plausible to suggest that the drastic increase in the Ti concentration should be favorable for the formation of the fine D0₃ particles at $a/2\langle 100 \rangle$ APBs, which is consistent with the observation in Figure 3. With increased aging time at 900 °C, both Al and Ti

concentrations at $a/2\langle 100\rangle$ APBs continued to increase significantly. It is thus expected that, owing to these increases, the amount of fine D0₃ particles at $a/2\langle 100\rangle$ APBs would increase and the microstructure of the remaining regions would transform from the disordered A2 phase to B2 phase, as observed in Figures 4 and 5.

Finally, it is interesting to note that, compared with the previously established isothermal sections of Fe–Al–Ti ternary alloys at 900 °C, the chemical compositions of Fe–23.8 at.%Al–11.1 at.%Ti and Fe–22.4 at.%Al–7.4 at.%Ti obtained from the D0₃ domain and (B2 + D0₃) region in the present alloy aged at 900 °C for 24 h are just located in the D0₃ and the (B2 + D0₃) regions, respectively [12].

The as-quenched microstructure of the Fe–23 at.%Al–8.5 at.%Ti alloy was a mixture of (A2 + D0₃) phases. When the alloy was aged at 900 °C, the D0₃ domains existing in the as-quenched alloy grew and an A2 \rightarrow (A2 + D0₃) \rightarrow (B2 + D0₃) transition occurred at $a/2\langle 100 \rangle$ APBs of the D0₃ domains. The microstructural revolution has not previously been reported by other workers in Fe–Al–Ti alloy systems.

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