JOURNAL OF MATERIALS SCIENCE **34** (1999) 4039–4042

Influence of Mn addition on the ordering of DO_3 Fe-28%Al intermetallics

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The DO_3-type ordering in Fe-28Al and Fe-28Al-1.5Mn alloys are investigated by TEM and XRD. The results show that Mn addition into DO_3-ordered Fe_3Al alloy could decrease degree of ordering. Two major factors are considered to have effect of Mn on ordering behaviour of the alloy: reducing grain size and reducing antiphase domain size. The further investigation of deformed antiphase boundaries and slip lines in the alloy with Mn addition suggests that Mn could promote slip and cross slip in DO_3 Fe_3Al alloy during deformation.

1. Introduction

Fe_3Al intermetallic compound, with a DO_3 long rang ordering, has excellent oxidation resistance, relatively low density and cost, and good high temperature strength. These advantages have led to consideration of Fe_3Al alloy for structural applications at elevated temperature [1]. Previous investigations [2, 3] have indicated that the temperature-strength anomalous behaviour of Fe_3Al alloy in the DO_3 region is associated with the degree of ordering. The strength peak will be observed at some intermediate degree of DO_3-type order below $DO_3 \rightleftharpoons B_2$ order-type transformation temperature, Tc and can be rationalised by a mechanism involving a transition of glide from superdislocations to unit dislocations. However, similar to other intermetallics, Fe_3Al is brittle at room temperature which limits its engineering applications, though the alloy is not intrinsically brittle and the reason for its low ductility is the humidity-induced hydrogen embrittlement [4]. A major effect in recent years has been directed toward the improvement of room-temperature ductility of Fe_3Al, especially in the addition of some alloying elements such as Cr, Ti, Mg, B and Ce in the alloys [5–8]. Recently some attentions have been paid to addition of manganese into FeAl and Fe_3Al alloys and it is reported that Mn could improve mechanical property of alloys [9, 10]. Our recent studies have also shown that Mn is an effective element for improving room temperature ductility [11–13]. The purpose of the present work is to investigate the effect of Mn on the ordering of hyper-stoichiometric Fe_3Al alloy, and to elucidate the dominant factors responsible for the ordering degree. In this work, the transmission electron microscope (TEM), accompanied by X-ray diffraction (XRD) and optical microscope, has been used. The mechanism by which Mn affects the ductility of DO_3 Fe_3Al alloy has been further investigated.

2. Experimental

The DO_3 Fe_3Al intermetallics studied were Fe-28%Al and Fe-28%Al-1.5%Mn (atomic percent) alloys. Both alloys were smelted in a vacuum induction furnace and cast into iron moulds. After homogenising for 4 h at 950 °C, the alloys were hot-rolled at 950 °C and warm-rolled at 650 °C from 7 to 0.7 mm. The samples were punched from the rolled sheet, and then heat treated in air for 1 h at 800 °C (for recrystallization) plus 50 h at 450 °C (for DO_3 ordering).

Thin foil samples were prepared from undeformed and deformed tensile samples to examine the state of order, antiphase domain (APD) and antiphase boundary (APB). These thin foil samples were prepared by spark cutting 3 mm disks from the 0.7 mm thick samples, gliding to 0.1 mm, and electropolishing in a fresh solution of one part nitric acid to four parts methanol in a Tenupol twin jet system at about −5 °C and 8 V. TEM examination was carried out using a philips CM12 electron microscope operating at 120 kV.

X-ray diffraction samples for ordering study were cut from the sheets by spark machining, with gauge dimension of 10 mm width, 14 mm length, and 0.7 mm thickness, following mild abrasion to remove the oxide coating. X-ray diffractometer traces were obtained from these samples using a Rigaku D/MAX-3BX operated at 50 kV, 25 mA, producing MoK_α radiation. Measurements were performed by step scanning 2θ from 10° to 90° with 0.3 step size. A count time of 1 min per step was used.

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3. Results

It will be helpful at first to review briefly the crystal structure in alloy.

Fe_3Al intermetallics has two types of ordering structure: one is B_2 (P_{m3m}) existing between 540 and 760 °C, the other is DO_3 (F_{m3m}) existing below about 540 °C. Above 760 °C exists disordered α(I_{m3m}). A unified description of all three structures is obtained by the introduction of a new larger elementary cell which comprises eight standard b.c.c. cell and thus contains 16 atoms. The coordinates of their corners are (0,0,0), (1/2,0,0), (1/4,1/4,1/4), and (3/4,1/4,1/4). These four sublattices will be referred to the letters I, II, III and IV, respectively. For this structure only those Bragg reflections are allowed when Miller indices are either all odd or all even. Al atoms occupy only IV sites in the DO_3 structure, but occupy both in III and IV sites in the B_2 structure. In the α structure Al could occupy all sites. According to Bragg reflection rule, the fundament α phase reflections occur for $h+k+l=4n$ (where n is an integer), the B_2 superlattice reflections occur for $h+k+l=4n+2$, and DO_3 reflections occur for h, k and l are unmixed. When h, k and l are even, the reflections coincide with either fundamental α or B_2 reflections. When h, k, and l are odd, the reflections belong to DO_3 alone.

In our investigation, both alloy have DO_3-type ordering structure after 50 h ordering treatment at 450 °C. Fig. 1 shows their superlattices in the [$\bar{1}$10] diffraction pattern. As Fig. 1 shown, strong fundamental spots are (004), (220) and weak superlattice spots are (111), (113). The degree of DO_3 ordering was determined by the following relation [14]:

$$S = K\sqrt{\frac{I_{(111)}}{I_{(220)}}} \tag{1}$$

where $I_{(111)}$ is integral strength of 111 superlattice diffraction, $I_{(220)}$ is integral strength of 220 fundamental diffraction, and K is a constant related to $I_{(111)}$ and $I_{(220)}$. Compared with Fig. 1a and b, it could been seen that the relative strength between superlattice diffraction spots and fundamental diffraction spots in Fe-28Al alloy is stronger than in Fe-28Al-1.5Mn alloy. In order to illustrate further that effect of Mn on ordering in the alloy, both alloys are analysed by XRD in the same measurement condition. The results of superlattice diffraction strength $I_{(111)}$ and fundamental strength $I_{(220)}$ by XRD are shown in Table I.

TABLE I XRD measurements on (111) and (220) planes

Alloy	2θ	I	hkl	$I_{(111)}/I_{(220)}$
Fe-28Al	12.220	262	111	3
	19.900	10252	220	
Fe-28Al-1.5Mn	12.220	403	111	2
	19.960	23581	220	

In Table I, θ is scanning angle, I is integral strength and hkl is diffract face. It is obvious from Table I that the alloy without Mn addition has higher ratio $I_{(111)}$ to $I_{(220)}$ than the alloy with Mn addition. The above results suggest that Mn could weaken superlattice diffraction of DO_3 Fe_3Al and decrease the degree of DO_3 ordering.

4. Discussion

It is shown by TEM and XRD analysis that DO_3-ordered Fe_3Al alloy could remain original ordering structure but has lower degree of ordering after Mn addition. There maybe many reasons that Mn reduces the ordering degree in the alloy. From the view of microstructure, there exists two main causes which give rise to decrease in ordering degree of the alloy after Mn addition, as discussed below:

(1) *grain size*: DO_3-ordered Fe_3Al-based alloy consists of many equiaxed grains. The atoms in grain boundaries arrange at random, reducing long rang order in the alloy. The smaller the grain, the more the grain boundaries and thus the lower the degree of ordering in the alloy. It is found from investigation of grains in both ordered alloys that addition of small Mn element into Fe-28Al ordered alloy can make its grain fine. The optical micrographs of the alloy with and without Mn are shown in Fig. 2. Obviously, the average grain size is much smaller in the alloy with Mn (about 50 μm) than that in the alloy without Mn (about 95 μm), resulting in decrease in ordering degree in the alloy after Mn addition. The effect of Mn on grain size is needed to research further.

(2) *APD size*: The DO_3 Fe_3Al alloy exists typically two types of APD: thermal APD and deformed APD. In general, the thermal ordered domains are formed and grow during ordering and at last the alloy consists of many fine thermal APDs which are separated by APBs. The atoms in the APBs arrange at disorder, reducing long rang order in the alloy. The smaller the APD, the more the APBs and thus the lower the ordering degree.

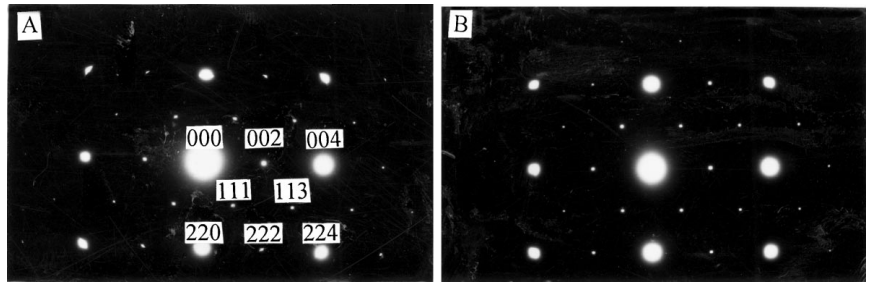

Figure 1 Electron Diffraction Patterns at [$\bar{1}$10] in (A) Fe-28Al-1.5Mn alloy and (B) Fe-28Al alloy.

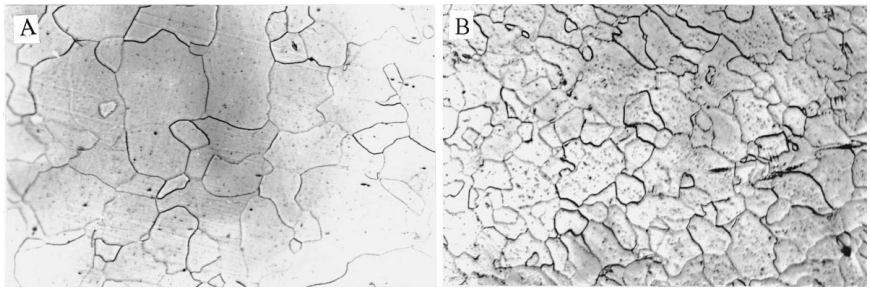

Figure 2 Optical micrographs showing grain structures of Fe-28Al (A) and Fe-28Al-1.5Mn (B).

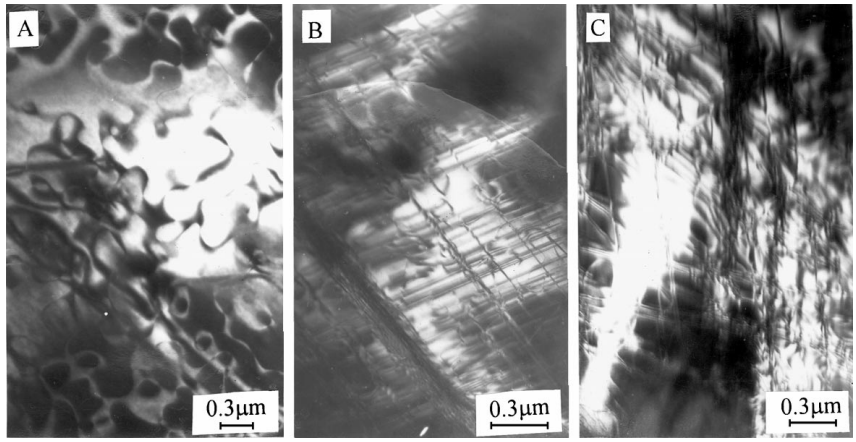

Figure 3 111 dark field micrographs showing antiphase boundaries in DO_3 Fe-28Al-1.5Mn alloy before deformation (A), after 0.3% plastic deformation (B) and after fracture (C).

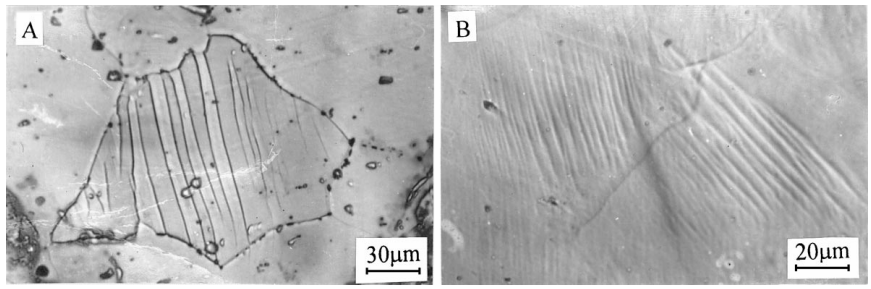

Figure 4 Slip lines produced in tension at room temperature showing (A) coarse, straight slip in Fe-28Al alloy and (B) wavy and finer slip in Fe-28Al-1.5Mn alloy.

The electron diffraction pattern with [110] axis of Fe_3Al alloy with Mn addition has shown 111 superlattice reflections of DO_3 type, as shown in Fig. 1a, suggesting that Mn addition does not change the DO_3 type of ordered phase of the alloy. Dark field electron micrograph using above reflection shows thermal APBs in the alloy with Mn addition corresponding to the states before and after deformation respectively (Fig. 3). An average diameter of 150–250 nm for the thermal APD (Fig. 3a) is observed in the alloy with Mn, which is smaller than the value of 350–400 nm reported for Fe_3Al alloy without Mn [15, 16]. Obviously, the higher density of APBs leads lower ordering in the alloy after Mn addition. After deformation, the morphology of APBs are completely change from random curves into crossed lines, deformed APBs. It is indicated from Fig. 3b and c that the APBs get more and more as deformation increases, suggesting that superdislocations induce more deformed APBs in the specimen as they move. The existence of zigzag APBs (Fig. 3c) also suggesting that cross slips of dislocation took place frequently when the alloy deformed. To examine further the increase in slip and cross slip of the alloy after Mn addition, deformed sample (1.5% plastic strain) were prepared under tension at room temperature. Slip lines on the surface of both alloys are shown in Fig. 4. In Fig. 4, straight slip lines are parallel to each other and aligned in the same direction within individual grains on the surface of the Fe-28Al alloy, suggesting that little cross slipping happened and only one slip system is generated in this alloy. In contrast, slip lines in the Fe-28Al-1.5Mn alloy become finer, denser and apparently wavy (Fig. 4b), and some slip lines even pass through grain boundaries, indicating that not only slip is easier to take place but cross slip is also generated during deformation in the alloy after Mn addition, leading to increase in ductility of DO_3 Fe_3Al. Wang *et al.* [17] found that slip behaviour of superdislocations is major reason for difficult cross slip in DO_3 Fe_3Al alloy. There are main four-fold superdislocations in DO_3 Fe_3Al alloy, which slip very

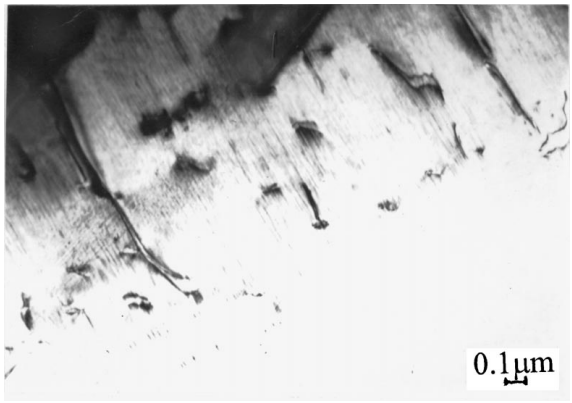

Figure 5 Structure of superdislocations in Fe-28Al-1.5Mn alloy.

difficulty in the alloy when deformed. Our research shows that two-fold superdislocations exist mainly in DO_3 Fe_3Al alloy with Mn addition, as shown in Fig. 5. Two-fold superdislocations slip easily than four-fold superdislocations because of low APB energy.

5. Conclusions

Mn could decrease degree of DO_3 ordering in Fe_3Al, which is caused by decrease in grain size and antiphase domain size after Mn addition into the alloy. Besides, Mn could promote slip and cross slip of superdislocations in the alloy because of existed mainly two-fold superdislocations, which may be one of reasons for improving ductility of the DO_3 Fe_3Al alloy.

Acknowledgements

The authors would like to thank professor Lu Guanglie for X-ray diffraction measurements.

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Received 20 March
and accepted 22 December 1998