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A TEM Study on α-Al(Mn,Fe)Si Dispersoids Precipitated in AA3003 Alloy

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The type, size and distribution of α-Al(Mn,Fe)Si dispersoids have strong influences on the deformation behaviour, recrystallization behaviour and mechanical properties of non heat treatable AA3xxx alloys. A better understanding of the precipitation behaviour of dispersoids is crucial to the optimization of the chemical compositions and homogenization processes of the alloys. In the present work, TEM, HRTEM and EDX have been applied to investigate the dispersoids precipitated at the early stage of precipitation during homogenization treatment. The orientation relationships and the interface structures between the dispersoids and the aluminium matrix have been studied. It was observed that the dispersoids show specific orientation relationships and are partially coherent with the aluminium matrix.

Keywords: *Aluminium; Precipitation; Homogenization; Orientation relationship; TEM*

1. Introduction

AA3xxx alloys are widely used in package and architecture applications. For DC cast ingots, homogenization must be conducted before hot rolling or extrusion, to reduce the concentration of Mn in solid solution and obtain the right size, type and distribution of constituent particles, which have a strong influence on the formability and recrystallization behavior of the materials. Many investigations have been done to study the precipitation behavior of dispersoids in pure and commercial Al-Mn alloys. In the pure Al-Mn binary alloys, different dispersoids, of body centered cubic (BCC) G1 ($Al_{12}Mn$) and orthorhombic G2 (Al_7Mn) and Al_6Mn phases, will form at different temperatures during homogenization [1-3], where the Al_6Mn phase is the equilibrium phase. When there are trace levels of Fe and Si impurities in Al-Mn alloy, simple cubic (SC) G' phase, hexagonal G'' phase and SC α-Al(Mn,Fe)Si phase will also precipitate in the alloy [4].

In commercial 3xxx alloys, cubic type α-Al(Mn,Fe)Si and orthorhombic type $Al_6(Mn,Fe)$ are the two main types of dispersoids [5,6]. At the earliest stage of precipitation, trigonal α′-Al(Mn,Fe)Si phase [7,8] and quasi crystal icosahedral Al(Mn,Fe)Si phase [5,7], both of which have the same composition as α- Al(Mn,Fe)Si phase, have also been found to precipitate in 3xxx alloys. It has been found that α- Al(Mn,Fe)Si dispersoids precipitate nearly in all commercial AA3xxx alloys, especially during heating at low and intermediate temperatures. However, when the concentration of Si is low, $Al_6(Mn,Fe)$ will be the stable dispersoids at high temperatures [6], which is similar to the binary Al-Mn alloys.

The α-Al(Mn,Fe)Si dispersoid is one of the most important intermetallic particles in 3xxx alloys. Usually, α-Al(Mn,Fe)Si dispersoids are considered to be incoherent with the Al matrix [4,9]. However, some electron diffraction studies show that the α-Al(Mn,Fe)Si dispersoids may be coherent or partially coherent with the Al matrix [5,10] at the early stage of precipitation. In order to elucidate this problem, a systematic study on orientation relationships (OR's) and the interface

structures between the dispersoids and the Al matrix has to be carried out. The objective of this work is to shed some more light on these aspects.

2. Experimental

The material used in this experimental work was a DC cast AA3003 extrusion billet with a diameter of 178 mm produced by Hydro Aluminium. The chemical composition of the alloy was (wt.%): Mn 1.15, Fe 0.58, Si 0.20 and Cu 0.08. Samples were taken from the half radius locations of the billet. Heat treatment was conducted in an air circulation furnace with a temperature accuracy of ±2 K. Samples were heated to 500 °C with a heating rate of 50 K/h. The samples were quenched into water when they were heated to different temperatures. TEM foils were prepared by electropolishing in an electrolyte containing two parts methanol and one part nitric acid at –20 °C. Foils were observed in a JEOL 2010 TEM at 200 kV. EDX attached to the TEM was used to measure the composition of the dispersoids.

3. Results and discussion

3.1 OR's between α-Al(Mn,Fe)Si dispersoids and Al matrix

Fig. 1 shows the morphology of α–Al(Mn,Fe)Si dispersoids along the [100] zone axis of Al matrix in the sample as-heated to 500 °C . The dispersoids either show a block-shaped (marked as 1 to 6) or plate-like (marked as 7 to 9) morphology. The size of most of the dispersoids is in the range of 50 to 200nm. It is interesting to note that dislocations and Moiré fringes can be seen on most of the dispersoids. Around the edge-on plate-shaped dispersoid 8, an obvious strain field surrounding the dispersoid can be observed. These results strongly suggest that the dispersoids may be partially coherent with the Al matrix.

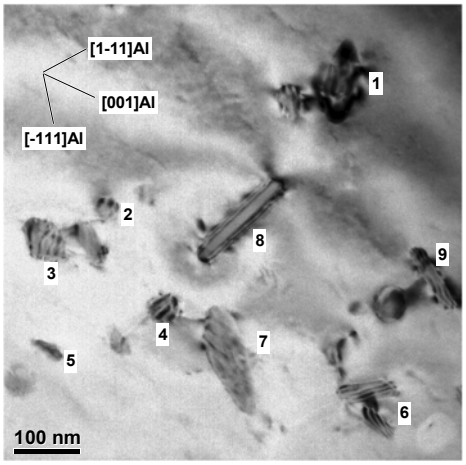

Fig. 1. Morphology of dispersoids precipitated at 500 °C, along [110] zone axis of aluminium matrix.

Fig. 2 shows the selected area electron diffraction patterns (SADP) taken along the [100] and [111] zone axes of the Al matrix. In each selected area, several dispersoids have contributed to the diffraction patterns. As can be seen, the reflections of the dispersoids are not randomly distributed; instead, they are composed of regular patterns, distributed around the reflection spots of Al. This suggests that most of the dispersoids have some common reflections and therefore, the dispersoids have some specific OR's with the Al matrix. Many SADPs of single dispersoids along [100] and [110] zone axis of Al have also been taken (not shown here). They show that the dispersoids' zone axes, which are parallel to the [100] or [110] zone axes of the Al matrix, have high indices. Due to the

double reflections and the overlapping of Zero Order Laue Zone (ZOLZ) and Higher Order Laue Zone (HOLZ) reflections, it is very difficult to determine the zone axes of the dispersoids.

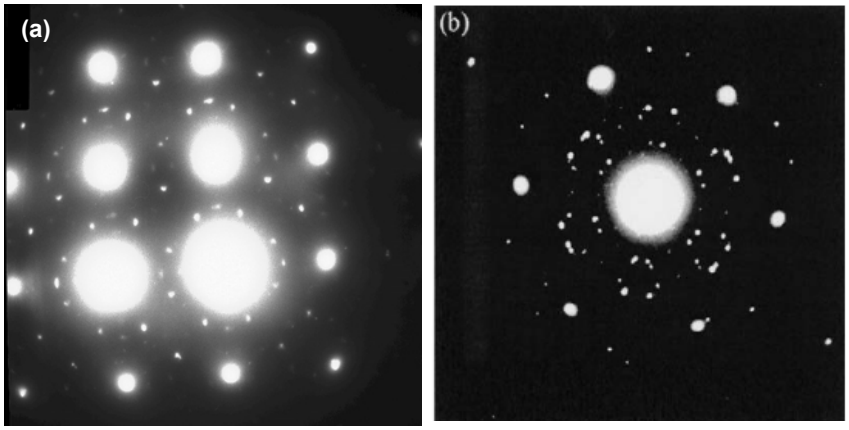

Fig. 2. Selected area diffraction patterns taken along [001] zone axis in (a) and [111] zone axis in (b) of aluminium matrix.

Fig. 3 shows the SADPs of single dispersoids along the [100] and [110] zone axes of the dispersoids. The dispersoids have been determined to have a SC crystal structure, with a = 1.265 nm. As can be seen in Fig. 3(b), the *{h k l }* reflections, with the value of *(h+k+l)* being odd, are slightly weaker than other reflections. EDX measurements show that the Mn content in the α-Al(Mn,Fe)Si dispersoids is much higher than the Fe content. The Mn/Fe concentration ratio in the dispersoids is larger than 3. It is well known that there is a transition from BCC structure to SC structure with increasing Mn content in the α-Al(Mn,Fe)Si phase [12]. At the early stage of precipitation, the α-Al(Mn,Fe)Si dispersoids have a very high Mn content, therefore the dispersoids show a SC crystal structure, which is close to the SC α-AlMnSi phase[11].

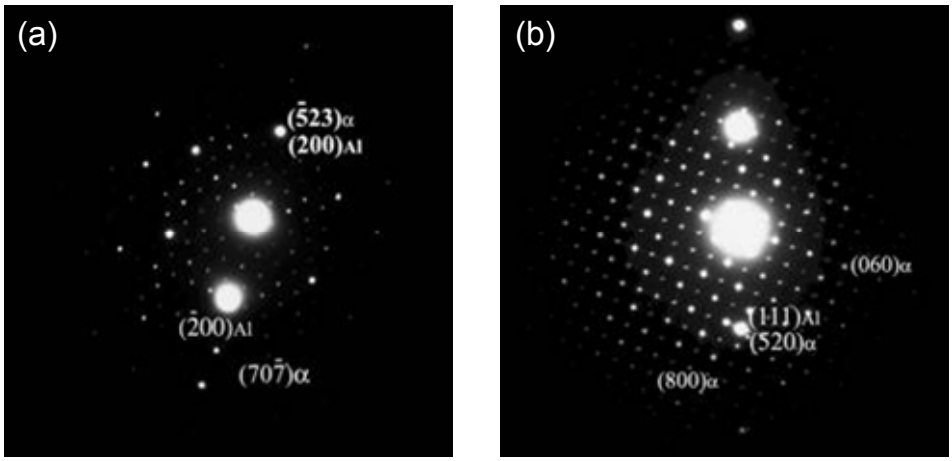

Fig. 3. Selected area diffraction patterns of single dispersoids along [111] in (a) and along [001] zone axis in (b) of dispersoids.

Similar to the zone axes of the dispersoids in the SADPs taken along the [100] and [110] zone axes of the Al matrix, the zone axes of the Al matrix in Fig. 3 also have high indices. However, it is interesting to see from the SADPs of Fig. 4(a) and (b) that coincidence exists between the $\{520\}_p$ and $\{111\}_m$ and between the $\{-253\}_p$ and $\{200\}_m$, where the subscripts *p* and *m* represent α-Al(Mn,Fe)Si

dispersoid and Al matrix, respectively. These coinciding plane pairs have been observed in most SADPs taken along the<100>$_p$,<111>$_p$ and <110>$_p$ zone axes.

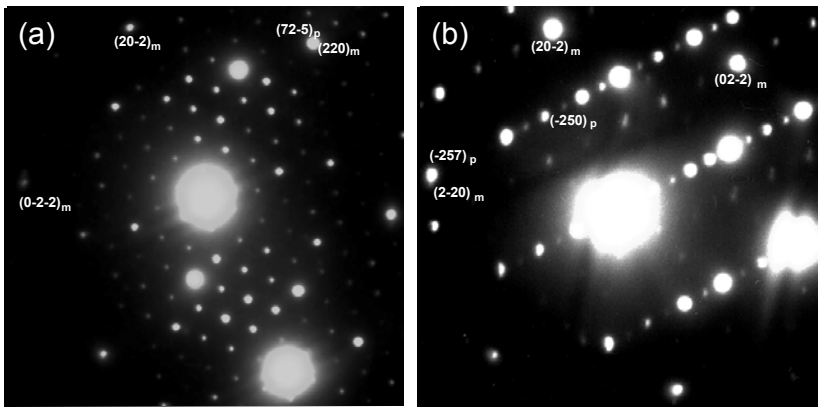

Fig. 4. Selected area diffraction patterns of single dispersoids with the electron beam parallel to $[1\text{-}11]_p$ and $[1\text{-}11]_m$ in (a), and $[520]_p$ and $[111]_m$ in (b).

A detailed study on the SADPs of dispersoids taken along the <111> zone axes of the Al matrix shows that clear OR's between dispersoids and Al matrix can be determined. Mainly two types of diffraction patterns of single dispersoids have been obtained, as shown in Fig. 4(a) and (b). The OR between the dispersoid and the matrix in Fig. 4(a) can be determined as

$$[1\text{-}11]_p \,//[1\text{-}11]_m \text{ and } (572)_p \,//(110)_m. \quad (1)$$

The OR in Fig. 4(b) can be written as

$$[520]_p \,//[111]_m \text{ and } (\text{-}257)_p//(\text{-}110)_m. \quad (2)$$

In both diffraction patterns, the $\{572\}_p$ are found to be coincident with $\{220\}_m$ lattice planes. There are 6 coinciding plane pairs in Fig. 4(a) and 2 pairs in Fig. 4 (b).

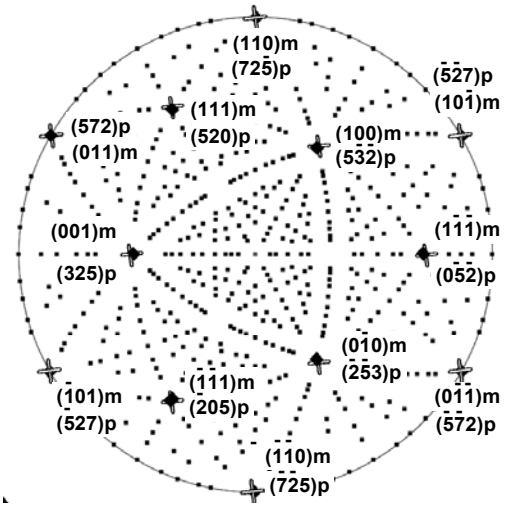

Fig. 5. A composite stereographic projection along [1-11] zone axis of aluminium matrix to show the OR between dispersoids and Al matrix (the reflections of dispersoid matrix are shown as X symbols and square buttons, respectively).

The OR (1) shown in Fig. 4(a) was drawn in a stereographic projection along the [1-11] direction of the Al matrix, as shown in Fig. 5. With this OR, many planes of dispersoids are found to be coincident or nearly coincident with planes of the Al matrix. All the coinciding plane pairs shown in Fig. 3 and 4 can be found in Fig. 5. From the stereographic projection, it can be seen that the OR (2) shown in Fig.

4(b) is equivalent to OR (1) shown in Fig. 4(a). The rotation angle from $[1\text{-}11]_m$ to $[111]_m$ is 70.53° and the rotation angle from $[1\text{-}11]_p$ to $[520]_p$ is 71.24°. The diffraction pattern shown in Fig. 3(b) can also be reproduced by overlapping of several SADPs shown in Fig. 4(a) and (b).

Table 1. Planar spacing and misfit between coinciding planes of α-Al(Mn,Fe)Si phase and Al matrix

Coinciding pairs	Dispersoid plane	Spacing (nm)	Matrix plane	Spacing (nm)	Misfit (%)
{572}p//{220}m	{572}	0.14323	{220}	0.14319	0.031
{532}p//{200}m	{532}	0.20521	{200}	0.02025	0.031
{520}p//{111}m	{520}	0.23490	{111}	0.23382	0.461

The planar spacing of planes of the most common coinciding plane pairs and the misfit between the coinciding plane pairs are shown in Table 1. The misfit was calculated by $\delta=(D_p-D_m)/D_m$, where D_p and D_m are planar spacing of dispersoid and matrix, respectively. As can be seen, the misfit between each coinciding plane pair is less than 0.5%. Although the real shape and habit plane of the alpha dispersoids at the earliest stage of precipitation are not known yet, we believe that the energy of the interface between the dispersoids and the matrix will be very low when the α-dispersoids precipitate with the OR shown in Fig. 5.

3.2 Interface between dispersoids and aluminium matrix

Fig. 6(a) shows a high resolution (HR) TEM image of an edge-on plate like dispersoid along the [111] zone axis of the dispersoid. By tilting the dispersoid, it has be determined that the broad surface and therefore the habit plane of the dispersoid is $(\text{-}12\text{-}1)_p$. If we assume that the dispersoid has an OR as shown in Fig. 5 with the Al matrix, the zone axis of Al matrix will be $[520]_m$, and the plane parallel to $(\text{-}12\text{-}1)_p$ will be $(2\text{-}54)_m$. Although it is difficult to determine the habit plane of the Al matrix directly from the HRTEM image, the angle between the habit trace of the dispersoid and the $(002)_m$ plane is measured to be 53.5°, which is consistent with the angle of 53.4° between $(002)_m$ and $(2\text{-}54)_m$. Hence the corresponding habit plane of the matrix is most probably $(2\text{-}54)_m$. Fig. 6(c) shows an inverse fast Fourier transformation (IFFT) image of the TEM image in Fig. 6(b) filtered by the reflections of the coincident planes $(002)_m$ and $(\text{-}532)_p$. As can be seen, the $(002)_m$ and $(\text{-}532)_p$ planes are continuous through the dispersoid/matrix interface, indicating a one-dimensional coherence between the dispersoid and the matrix.

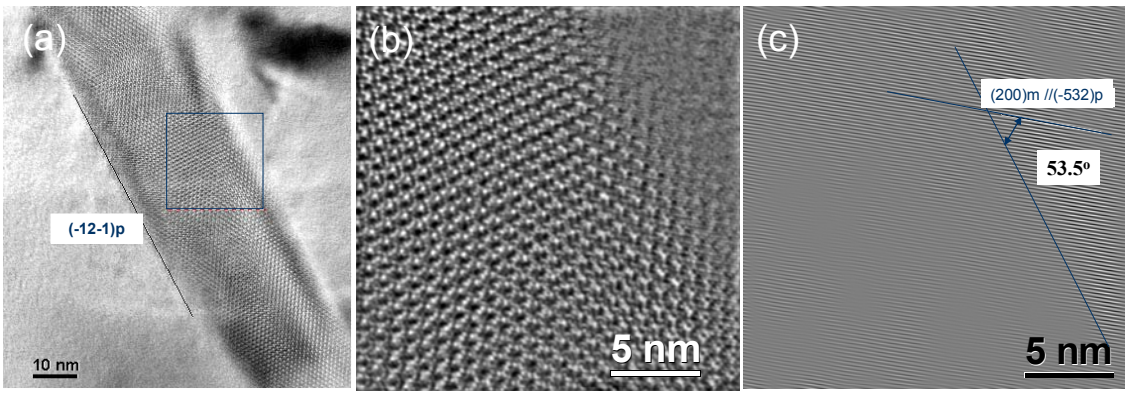

Fig. 6. HRTEM image of an α-Al(Mn,Fe)Si dispersoid along [111] zone axis. (a) HRTEM image; (b) enlarged HRTEM image in the frame inserted in (a); (c) Inverse fast Fourier transformed image of (b) after filtration by reflections of coinciding plane pair of $(200)_m$ and $(\text{-}532)_p$.

By the OR shown in Fig. 5, the dispersoids can also have other low energy habit planes. For example, $(100)_p$ and $(110)_p$(not shown here) have been observed as well. It has to be noted that the present work just showed one of the most common OR's between the dispersoids and the matrix. Some other OR's have also been observed in this work.

4. Conclusions

We investigated the OR's between the aluminium matrix and the α–Al(Mn,Fe)Si dispersoids precipitated in an AA3003 alloy at 500 °C during heating. Most of the dispersoids have a specific OR with the aluminium matrix, which can be described as $[1\text{-}11]_p$ //$[1\text{-}11]_m$ and $(572)_p$ //$(110)_m$. With this OR, the dispersoids have many pairs of coinciding planes with the Al matrix. The habit plane of one of the dispersoids is {-12-1} and the corresponding habit plane of the Al matrix is {2-54}. A HRTEM study confirmed that a one-dimensional coherence exists between the dispersoid and the matrix across the dispersoid/matrix interface plane. It is supposed that the dispersoids are partially coherent with the aluminium matrix at the early stage of precipitation.

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