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EFTEM Observation of Cube-Shaped Precipitates in Al-Mg-Si Alloy

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Abstract

It is well known that the equilibrium phase in an Al-Mg-Si alloy is the β-phase (Mg_2Si). The cube-shaped phase has been also reported by a few reports and discussions were made about whether it is the precursor of the β-phase or not. Also, this phase was often found in the Al-Mg-Si alloy with the content of excess Mg. We are studying the application of energy filtering TEM (EFTEM) for quantifying chemical composition of precipitates in aluminium alloys, and some results about β-phase as well as other small precipitates in Al-Mg-Si alloys have been reported in our earlier studies. In this work, the cube-shaped phase in Al-Mg-Si with excess Mg type has been investigated by EFTEM in order to know the crystal structure and its chemical composition.

1. Introduction

A lot of studies have been done on the precipitations in Al-Mg-Si alloys about the crystal structure and chemical composition. The cube-shaped phase has been observed in the Al-Mg-Si alloy with excess Mg type and quasi-binary alloys [1-3]. It has proposed that similar crystal structure [2], however, the chemical composition has not been well investigated yet. The current study was done on analysing the composition of cube-shaped phase by performing the energy-filtered transmission microscopy (EFTEM) as well as the electron energy-loss spectroscopy (EELS) and energy dispersive spectroscopy (EDS). Also, the quantification of chemical composition will be carried out from elemental mapping intensity.

2. Experimental

The Al-1.0mass%Mg2Si-0.4mass%Mg Alloy (excess Mg alloy) was used for the current studies. The ingot was hot-and cold-rolled to 0.2mm thickness, and solution heat-treatment at 848K for 3.6ks and quenched in chilled water at 277K. The aging condition was performed at 623K. For TEM, specimen was prepared by electrolytic polishing using 10 percent perchloric acid and 90 percent ethanol. EFTEM (JEOL-4010T, JEOL, Co.Ltd., Tokyo, Japan) equipped with a post-column energy filter spectrometer (Gatan GIF 200) was operated at 400kV. Elemental maps was calculated by using three-window method with higher loss electron energies, to reduce the effect from the strain contrast. The equation used for quantifying the chemical composition from elemental mapping intensity is shown below [4],

$$n_A = \frac{I_A}{I_T} \cdot \frac{1}{t \cdot \sigma_A(\beta, \Delta E, E_0)} \tag{1}$$

n_A is the area density of the element [atom/nm^3]. I_A is intensity of elemental mapping per unit time [intensity/sec]. I_T is zero-loss intensity per unit time [intensity/sec]. t is thickness [nm]. σ_A is a partial cross section for energy losses within a range ΔE of the ionization threshold and for scattering angles up to β.

3. Results and Discussion

Figure 1 shows TEM image of the excess Mg alloy aged at 623K for 6ks. The square- and rectangle-shaped precipitates were observed from the image. The sides of these precipitates were found to have the directions aligning along [100] and [010] directions of the matrix as marked by the arrows. The corresponding selected area diffraction pattern from this precipitate was shown on Figure 2. It can be calculated from the diffraction pattern that this precipitate is cube-shaped phase as comparing to the previous report [1]

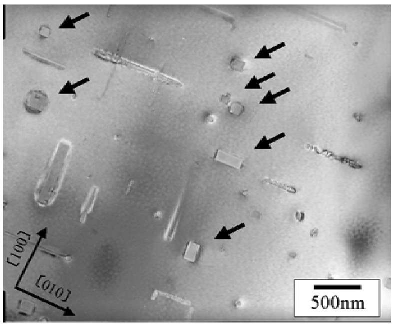

Figure 1: TEM image for A1.0mass%Mg_2Si-0.4mass%Mg aged at 623K for 6ks.

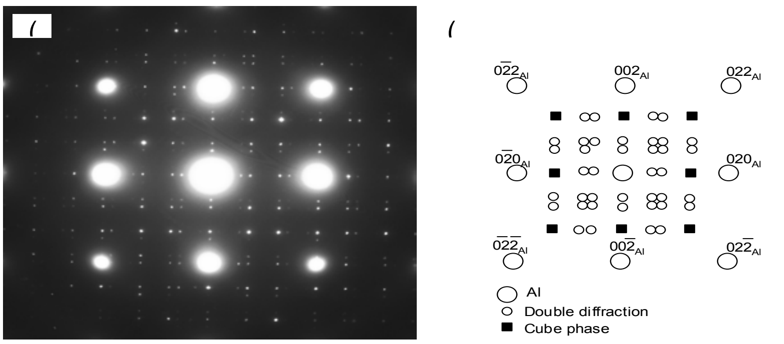

Figure 2: (a) SADP taken from the cube-shaped phase and (b) its schematic diagram.

Figure 3 shows the extrapolation plot of EDS analysis. By extrapolating the curve to the intersection of Y-axis, the effect of Al-matrix can be removed assumedly. This result shows that the ratio of Mg:Si is near 3:1.

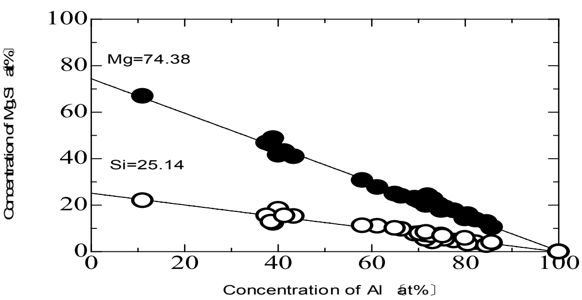

Figure 3: Extrapolation plot for EDS analysis.

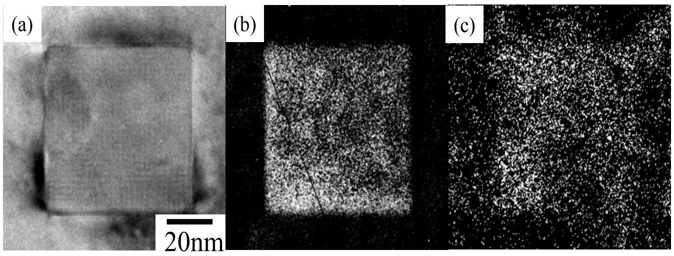

Figure 4: EFTEM images of cube-shaped phase (a)zero-loss image and (b) Mg-K map (c) Si-K map.

The zero-loss image in Figure 4 (a) is a typical cube-shaped phase with around 60nm in diameter. Elemental mapping was done on this precipitate. Figure 4 (b) is Mg-map (K-edge, 1305eV) and Figure 4(c) is Si-map (K-edge, 1839eV). These images showed bright contrast corresponding to the precipitates area in Figure 4 (a). In this case, Mg-map was clear bright contrast, but Si-map was unclear. The reason why Si K-edge is near Al K-edge as shown Figure 5, and cube-shaped phase is in the Al matrix, so background of Al K-edge affects Si K-edge. By using equation (1), the obtained mapping intensity from Figure 4 (b) and (c) can be transformed into the quantification of chemical composition for the precipitate. The quantification results with the unit as atom per unit volume were presented. The calculated Mg/Si ratio was found near 3, which is almost the same as the EDS analysis. From EDS analysis and elemental mapping, it can be considered that cube-shaped phase were near Mg/Si=3.

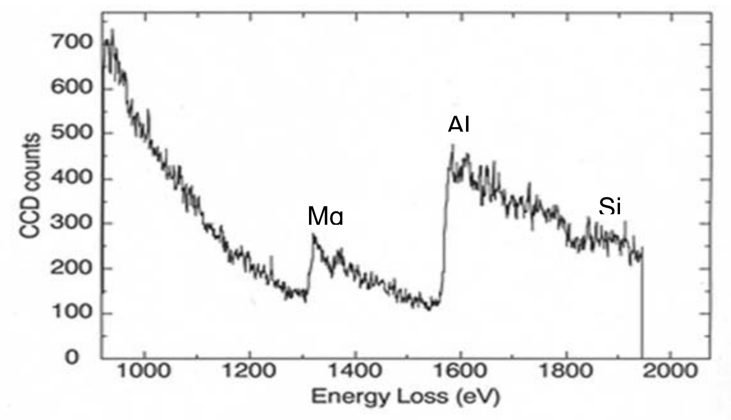

Figure 5: EELS spectrum taken from cube shaped phase.

4. Conclusion

Composition of cube-shaped phase precipitated in Al-1.0mass%Mg_2Si-0.4mass%Mg alloy as aged at 623K for 6ks was analysed and the result of Mg/Si ratio as 3:1 was obtained by using EDS and EELS. Furthermore, the quantification of chemical composition was done also from the intensity on Mg and Si elemental maps. Same results were obtained between these two methods.

References

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