HRTEM Study of Precipitates at the Early Stage during Aging in Al-Mg-Si-Cu Alloy

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Abstract

The effect of copper on precipitation sequence of Al-Mg-SI alloys has been studied and discussed by many researchers. However, there is no suitable explanation yet. We are studying precipitation sequence of Al-Mg-Si alloys by using high-resolution transmission electron microscopic (HRTEM) technique. Several metastable phases and their crystal structures as well as the lattice parameter have been determined by our previous research. We have found the segregation of copper happening at the interface between the Q'-phase in the Al-Mg-Si-Cu alloy by energy-filtering TEM (EFTEM). Also, strange shape precipitate was found at that time and we have reported about it at the 8th ICAA [1]. In this work, we observed the precipitate, which has the elongated shape in the sample annealed at the early stage during aging and discussions about its crystal lattice and the differences between this precipitate and the normal ones in this alloy, for example, Q'-,b"-, b'- phases were presented.

1. Introduction

It is well known that the addition of copper to Al-Mg-Si alloys can improve their ductility and aged hardening [2]. It is also well known that Q phase existing in Al-Mg-Si-Cu alloy as its quaternally equilibrium phase. Recently, its metastable phase, Q' phase has been reported by numerous researchers [3,4]. We found that the segregation of copper happening at the interface between the Q'-phase in the Al-Mg-Si-Cu alloy by energy-filtering TEM (EFTEM). We also found that not only random-type or parallelogram-type precipitates, which were generally observed in the quasi-binary Al-Mg₂Si alloys, but also the strange shape precipitates were formed in this Al-Mg-Si-Cu alloy annealed at early stage during aging. These strange shape precipitates in Cu-free Al-Mg-Si alloy. As a result, we considered that these strange shaped precipitates were formed only when Cu was added.

The current studies will focus on characterizing the strange shape precipitates in Al-Mg-Si alloy containing Cu aged at 473K by using high-resolution transmission electron microscopy (HRTEM).

Al-1.0mass%Mg₂Si-0.5mass%Cu (0.5Cu alloy) and Al-1.0mass%Mg₂Si (Cu-free alloy) alloys were prepared by 99.99% Al, 99.99%Mg, 99.99%Si, 99.99%Cu ingots. The obtained ingot was homogenized at 723K for 4 days. Ingots were hot- and cold-rolled to 0.2mm thickness. Sheets were solution heat-treated at 848K for 3.6ks and quenched in to chilled water at 277K. Quenched sheets were aged at 473K in oil bath. TEM sample ware prepared by an electrolytic polishing. TEM was operated at 120kV (Topcon, EM-002B).



Figure 1: TEM images of the 0.5Cu alloy aged at 473K for 60ks. (a) bright field image and (b) dark field image. (c) and (d) are HRTEM images of needle-shaped and strange precipitate respectively.

3. Results and Discussion

Figure 1 shows TEM images of the 0.5Cu alloy aged at 473K for peak hardness (60ks). Figure 1(a) and (b) show the bright field image and the dark field image respectively. Needle-shaped precipitates were observed as arrowed by A. Cross section of needleshaped precipitates can be seen as bright dots in a dark field image of (b). The corresponding HRTEM image was shown in Figure 1(c). Needle-shaped precipitates in (a) were either random-type or parallelogram-type precipitates which can be seen in Cu-free Al-Mg-Si Alloy. The precipitates, which have elongated cross section, were also observed in Figure 1 (a) and (b) and was arrowed by B. The HRTEM image was shown in Figure 1(d). In Cu-free alloy, no precipitates with elongated cross section were observed. Consequently, we concluded that the elongated precipitates were formed with the addition of Cu. Figure 2 shows TEM images taken from different zone axis, <100>, <110>, and <112> of the matrix. It was seen that only the precipitates along the <100> direction could be observed from the images. As a result, we suggested that the precipitates have elongated cross section along <100> direction of the matrix.



Figure 2: TEM images observed from difference direction. (a) <100>, (b) <110>, (c) <112> direction of the matrix.

FFT and Inverse FFT were done on the strange shape precipitates for better image for lattice parameter determination. Figure 3(b) shows the result of FFT which was selected in HRTEM image of Figure 3(a). There are some Spot from strange precipitate were shown by arrows. We could get the image of Inverse FFT image shown in Figure 3(c), we suggested that the precipitates should have hexagonal network with a-spacing as 0.405nm. The crystal lattice of simple hexagonal has been assumed for this precipitate and its simulated SADP was in good agreement with actual SADP obtained from this precipitate.

At the over aged condition (473K for 600ks), Q'-phase was observed. The image was shown in Figure 4. Beside the Q' phase, another precipitates were seen also. These precipitates have one end with elongated cross section as strange shape precipitates, while the other side of the precipitates have the structure as hexagonal network with spacing of 1.04nm at an angle of 10 degree to the [010] direction of the matrix, which is similar to that of Q'-phase. As a result, we proposed that the strange shape precipitates grow and transform to Q' phases as the alloy was further aged and a close relationship of structure between these two phases should exist.



Figure 3: Result of FFT and Inverse FFT. (a) HRTEM image of strange shape precipitate, (b) FFT image, (c) Inverse FFT image.



Figure 4: HRTEM image of 0.5Cu alloy aged at 473K for 600ks. This precipitate is conformed to Q'-phase.

4. Conclusions

The strange shaped precipitates were observed in Al-1.0mass%Mg₂Si-0.5mass%Cu alloy. This precipitates has been suggested of having a simple hexagonal network with spacing 0.405nm from result of FFT and SADP simulation.

References

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