

STRUCTURE OF G. P. ZONE IN Al-Mg₂Si ALLOYS

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ABSTRACT The existence of G.P. zone in Al-Mg-Si alloys have been reported by previous workers using X-ray diffraction method or DSC method. The structure of the G.P. zone in an Al-1.6mass%Mg₂Si alloy aged at 343 and 423K was investigated by high resolution transmission electron microscopy in this work. The morphology of the G.P. zone is fine-plate having dimensions of a mono-layer in thickness, 2.5nm in width and less than 30nm in length. Its elongated direction is parallel to the <100>_m direction. The chemical composition of the G.P. zone in an Al-Mg₂Si alloy was determined to be Mg/Si=1.0.

Keywords : *aluminum-magnesium-silicon alloy, aging, G.P.zones, high resolution transmission electron microscopy*

1. INTRODUCTION

It has been well known that the nuclei of metastable phases and composition fluctuation are found in an Al-Mg₂Si alloy during the early stage of aging[1-3]. The specific heat and the electrical resistivity measurements have been performed over the entire range of aging temperatures, and the existence of clusters and the G.P. zones have been proposed[1-5]. However, the structure of the zones formed during aging were identified only in a limited range of aging temperatures higher than 423K using X-ray and electron diffraction techniques[2,6-11].

In our recent systematic work, new types of metastable phases were found in this alloy system. The morphologies, crystal structures and crystallographic orientation relationship with the matrix of the metastable phases in this alloy system have been clarified using high resolution transmission electron microscopy (HRTEM), a micro-beam electron diffraction technique and energy dispersive X-ray spectroscopy[12-14]. The changes in the number of each metastable phase have also been reported[15]. However, we have not yet clarified the structure and chemistry of clusters or G.P. zones during the early stage of aging.

The aim of this study is to clarify the structure and chemical composition of the G.P. zones in an

Al-1.6mass%Mg₂Si alloy aged at temperatures lower than 423K using HRTEM.

2. EXPERIMENTAL

An Al-1.6mass% Mg₂Si alloy was prepared using 99.99% pure aluminum, 99.9% pure magnesium and 99.9% pure silicon. The ingot obtained was formed into 0.2 mm thick sheets by hot- and cold-rolling. A solution heat treatment was performed at 848K for 3.6ks and then quenched in chilled water (273K). The aging treatment was performed at 343 and 423K. Thin specimens for HRTEM were prepared by ordinary electrolytic polishing and observed using the EM-002B (TOPCON Co., Ltd.) operated at 100kV. Electrical resistivities of the specimens were measured using the direct current, four-terminal method in liquid nitrogen at 77K. HRTEM images were calculated using the multi-slice method to identify the experimentally observed images.

3. RESULTS

Fig. 1 shows changes in the electrical resistivity(ρ) with aging time at 343 and 473K. The values of ρ during the early stage at each aging temperature are higher than that of the quenched ones. The electrical resistivity, ρ in the specimen aged at 343K slightly increases with increasing aging time in a range examined in this work. However, the values of ρ at 423K decreases with increasing aging time after showing a constant value slightly higher than the as-quenched value during the early stage of aging. These are the same tendency as the result of electrical resistivity reported by Panseri et al.[16].

Fig. 2 shows the bright field image of the specimen aged at 343K for 1,200 ks. Extremely fine dark contrasts are observed. However, no clear precipitates with a needle shape are observed at a magnification of 200,000 times.

Fig. 3(a) shows an HRTEM image of the specimen aged at 343K for 1,200ks. Figures 3(b) and (c) are enlarged photographs of the circled regions A and B in Fig.3(a), respectively. Bright lattice points are arrayed in a row having a spacing of 0.405nm only along the (200) lattice plane of the matrix as shown by the arrows in region A of Fig.3(a). Two rows of bright lattice points indicated by the arrows are arrayed parallel to each other in Fig.3(c). Fig.4 (a) shows a SADP corresponding to Fig.3(a). Streaks were observed towards the [100] and [010] matrix directions on the center points of the 000 and 110 reciprocal lattice points. Fig. 4(b) shows a schematic illustration of Fig.4(a). The morphology of the zones in this alloy is assumed to be a fine plate like the G.P. zone in Al-Cu alloys along the $\langle 100 \rangle$ m.

There are several structure models of the G.P. zones in this alloy[6,7,10]. The classical models are proposed by Geisler[6] and Guinier[7]. Geisler[6] proposed that the shape of the zone was a fine-plate having a dimension of 2nm in width and 1nm in thickness. However, he did not describe the structure and the chemical composition of the zone. Guinier[7] also proposed that the zone in this alloy had a chemical composition nearly equal to Mg₂Si stoichiometry and a part of the crystal structure of the anti-CaF₂ type, and its shape was a needle. On the other hand, Thomas[10] proposed that zones were made by alternately stacking two Mg-layers and one Si-layer on the basis of the

(011) lattice plane of the matrix, and the zones grew along the [100] direction of the matrix. Recently, Huppert and Hornbogen[17] proposed the formation mechanism of the G.P. zone in this alloy as follows: First, the clusters composed of Al, Mg, Si and vacancies were produced during the early stage of aging. Second, Al atoms diffused from the clusters to the matrix, and then G.P. zones comprised of Mg and Si atoms were finally formed. However, they had no direct evidence and did not describe the structure of the G.P. zones.

Based on our results, we propose that the G.P. zone is a fine-plate that is one atomic layer in thickness, 2.5nm in width and less than 30nm in length. It is considered that there is a periodic arrangement of Mg atoms or Si atoms having a spacing of 0.405nm along the [100]m or [010]m directions, because strong bright dots are aligned having a spacing of 0.405nm along the [100]m or [010]m directions. Simulated images of the HRTEM about the G.P. zone were suggested for one idea of its chemical composition, although the chemical composition of this zone has not been obtained by EDX analysis in the present work because this zone is very fine. Fig.5 shows a schematic illustration of the G.P. zone we propose. When a column of Mg atoms and a column of Si atoms along [001]m are alternately arranged on the (200)m, the spacing of 0.405nm of bright dots was obtained as shown in Fig. 5. Namely, the G.P. zone in Al-Mg₂Si alloys is made by the alternating arrangement of the atom columns of Mg and Si atoms along <100> having the spacing of 0.405nm. The chemical composition of this G.P. zone is Mg:Si=1.0. This model is similar to that proposed by Guinier, inadvertently, except for its chemical composition. We do not consider the existence of vacancies in this zone in our present work.

4. CONCLUSIONS

The structure of the G.P. zone in an Al-1.6mass% Mg₂Si alloy aged at 343 and 423K was investigated by HRTEM. The obtained results are as follows:

- (1) The morphology of the G.P. zone is a fine-plate having dimensions of a mono layer in thickness, 2.5nm in width and less than 30nm in length. Its elongated direction is parallel to the <100>m direction.
- (2) The assumption that the chemical composition of the G.P. zone is Mg:Si=1.0 was in good agreement with the simulated HRTEM images and those actually obtained. The structure of the G.P. zone proposed in our present study was as follows: a column of Mg atoms and a column of Si atoms having a spacing of 0.405nm along the [001]m direction alternately arranged on the (200)m lattice plane.

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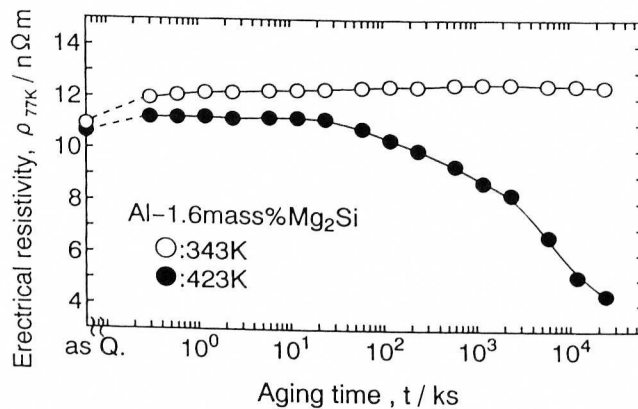


Fig.1 - Changes in electrical resistivity of the specimens aged at 343K(○) and 423K(●) versus aging time.

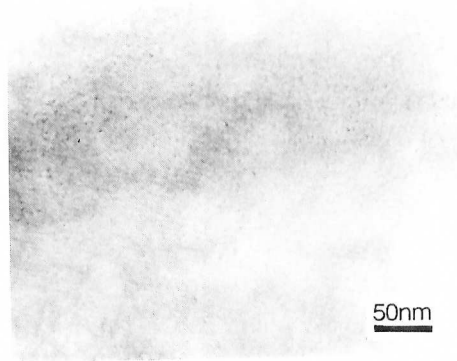


Fig.2 - Transmission electron micrographs of the specimens aged at 343K for 1,200ks.

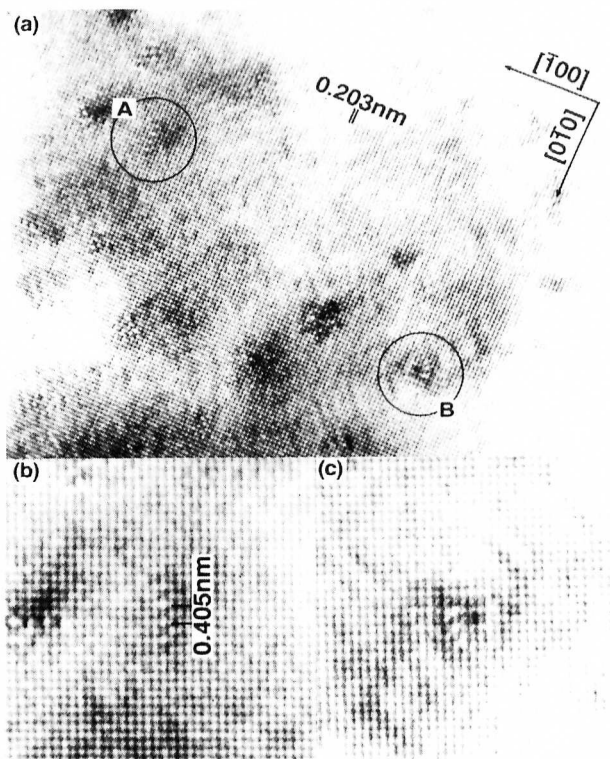


Fig.3 - Transmission electron micrographs of the specimens aged at 343K for 1200ks.
 (a) An HRTEM image, (b) and (c) are enlarged photographs of regions marked by circles A and B in (a), respectively.

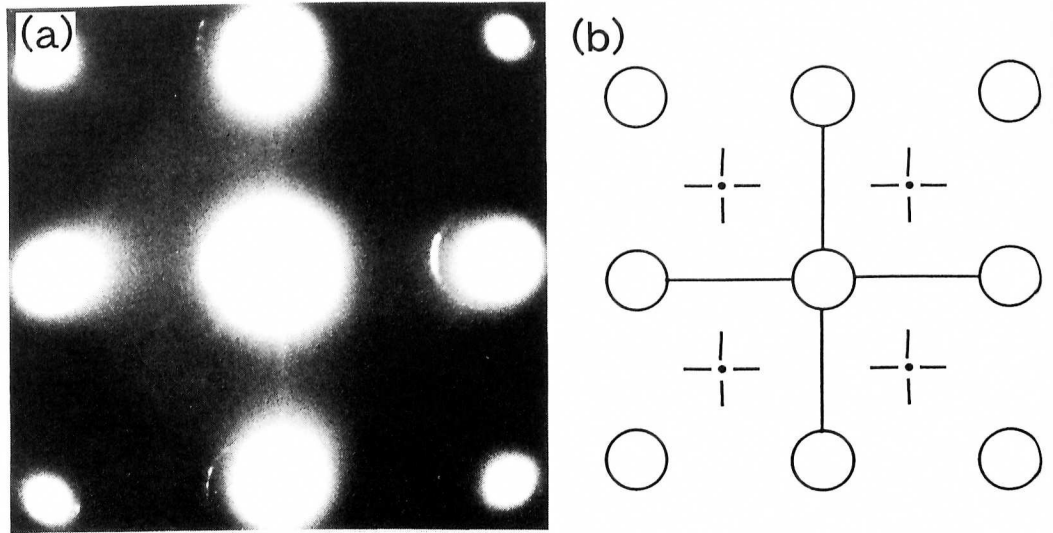


Fig.4 - (a) Selected area diffraction pattern taken from specimen aged at 343K for 1200ks and (b) schematic illustration of (a).

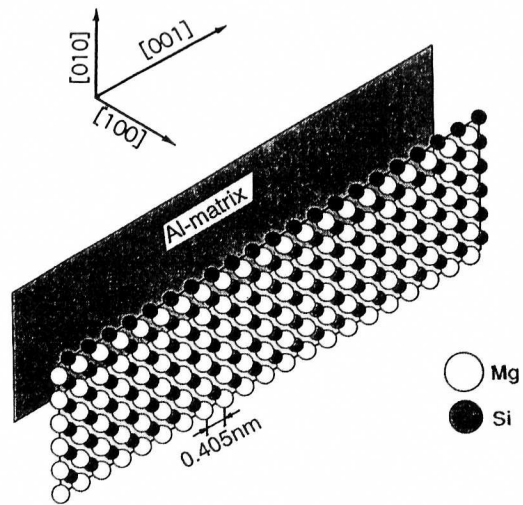


Fig.5 - The structure of the fine-plate G.P. zone in an Al-Mg₂Si alloy proposed in this work.