



THE CRYSTAL STRUCTURE OF β'' -PHASE IN Al-1.0mass%Mg-0.4mass%Si ALLOY

Tsutomu NAOI*, Kenji MATSUDA**, Tatsuo SATO***

Akihiko KAMIO***, Susumu IKENO**

* Graduate student, Toyama University, 3190, Gofuku, Toyama, 930-8555, JAPAN

** Faculty of Engineering, Toyama University, 3190, Gofuku, Toyama, 930-8555, JAPAN

*** Faculty of Engineering, Tokyo Institute of Technology, 2-12-1 O-okayama, Meguro-ku

Tokyo, 152, JAPAN

ABSTRACT The crystal structure of the β'' phase has been investigated by high resolution transmission electron microscopy(HRTEM), electron diffraction pattern(EDP), micro beam diffraction(MBD) technique and energy dispersive X-ray(EDX) analysis. The β'' phase is a monoclinic lattice and its lattice parameters are $a=0.77 \pm 0.02\text{nm}$, $b=0.67 \pm 0.01\text{nm}$, $c=0.203\text{nm}$ and $\gamma=75^\circ \pm 0.5^\circ$. Its space group is B2/m. The crystallographic orientation relationship is as follows:

$$[001]_{\beta''} // [001]_{\text{M}} \text{ and } [100]_{\beta''} // [130]_{\text{M}}.$$

The β'' phase is a needle shape and its longitudinal direction parallels to the direction of $[001]_{\beta''}$. The chemical composition of this phase is Si:Al:Mg=6:3:1.

Keywords : Al-Mg-Si alloy, β'' phase, HRTEM, Aging precipitate

1. INTRODUCTION

The aging sequence of Al-Mg-Si alloy had been suggested as follows[1];

G.P. zones \rightarrow *metastable β' phase* \rightarrow *equilibrium β phase*

The existence of β'' phase as a precursor of the β' phase in Al-Mg-Si alloys has been pointed out from several reports by thermal analysis method[2,3]. The structure of the β'' phase has been not perfectly identified by X-ray diffraction(XRD) and electron diffraction pattern(EDP) techniques[4-8].

This β'' phase is very important because it may be a major strengthening precipitate of Al-Mg-Si alloys[9]. Ordinarily the volume fraction of the β'' phase is changed with both the chemical composition of alloys and the aging temperature. If the suitable conditions of time, temperature and composition are selected, there is an aging condition that only the β'' phase is existed in the whole specimen.

The aim of this work is to clarify the crystal structure of the β'' phase in Al-1.0mass%Mg-0.4mass%Si alloy aged at the condition existing only β'' phase by high resolution electron microscopy(HRTEM), EDP, micro beam diffraction (MBD) technique and energy dispersive X-ray (EDX) analysis.

2. EXPERIMENTAL DETAILS

The alloy used in this work is an Al-1.0mass%Mg₂Si alloy containing 0.4mass%Si in excess (the excess Si alloy, hereafter). The excess Si alloy was prepared using 99.99%Al, 99.9%Mg and 99.9%Si ingots by melting. The iron content as an impurity was less than 0.01mass%. Obtained ingot was hot- and cold-rolled to 0.2mm thick sheets. These sheets were solution heat treated at 848K for 3.6ks and then quenched into chilled water (approximately 273K). Aging treatment was performed at 423K for various time in the silicone oil bath. Thin foils for HRTEM was prepared by electrolytic polishing technique. An HRTEM is the EM-002B type (Topcon co.ltd., point resolution=0.18nm, line resolution=0.14nm, Cs=0.4mm) equipped with EDX (EDAX inc.) and is operated at 200kV.

3. RESULTS AND DISCUSSION

Figure 1 shows a bright field image of the specimen aged at 423K for 2400ks. The needle-shaped precipitates are observed along the [100] and [010] direction of the matrix in Fig. 1(a). Fig. 1(b) shows a bright field image of the specimen observed along the [110] direction of the matrix. There is no precipitate having different shape from the needle. Its mean length is 23nm. The needle-shaped precipitates also indicate a particle shape in Fig. 1 because they parallel [001] direction of the matrix. We call this the end-on that is a target of this work.

Figure 2 shows a typical HRTEM image of the end-on that exists predominantly in this aging condition. The diameter of this end-on is 6.0nm and its out line is a parallelogram. The arrangement of bright dots in it appears a parallelogram network having spacings of 0.77nm, 0.67nm and an interior angle of 75 degrees as shown in Fig. 2. The arrangement of bright dots in this end-on is different from those of four types of metastable phases we reported, i.e. the β' phase, TYPE-A, TYPE-B and TYPE-C precipitates[10-13]. An angle the [100] direction of the matrix is 20 degrees. We call this end-on as the β'' phase hereafter.

Figure 3 shows the electron diffraction pattern taken along both the [310] direction of the matrix parallel to a side of the parallelogram network and the normal direction to the longitudinal direction of the β'' phase, because the β'' phase has a specific orientation relationship with the matrix as shown in Fig. 2. There are diffraction spots with separated streaks at the center of between the 000 and 002 diffracted spots of the matrix.

Figure 4 shows the electron diffraction pattern taken from the β'' phase extracted from the matrix by the thermal phenol method. There is no ring corresponding to the spacing of 0.405nm. According to the result of this work, the lattice parameter of the β'' phase will be estimated as follows: monoclinic, $a=0.77\pm 0.02\text{nm}$, $b=0.67\pm 0.01\text{nm}$, $c=0.203\text{nm}$, $\gamma=75^\circ \pm 0.5^\circ$.

The ratio of Si/Mg of β'' phase is about 6 from the result of EDX analysis for the β'' phase extracted from the matrix. The spectrum of Al was also detected in the β'' phase and the ratio of

Si:Al:Mg is 6:3:1.

We propose the crystal structure of the β " phase on this work. It is the base centered monoclinic lattice and one base centered atom is Mg and the others are Si (+Al) atoms. The lattice parameters are $a=0.77 \pm 0.02\text{nm}$, $b=0.67 \pm 0.01\text{nm}$, $c=0.203\text{nm}$, $\gamma=75^\circ \pm 0.5^\circ$. The space group is B2/m. The chemical composition is Si:Al:Mg=6:3:1. Simulated HRTEM images using the structure of the β " phase are in good agreement with actual HRTEM images for the verification of the amount of the defocusing.

4. CONCLUSION

The crystal structure of the β " phase has been investigated by HRTEM, EDP, MBD technique and EDX analysis. The β " phase is a monoclinic lattice and its lattice parameters are $a=0.77 \pm 0.02\text{nm}$, $b=0.67 \pm 0.01\text{nm}$, $c=0.203\text{nm}$ and $\gamma=75^\circ \pm 0.5^\circ$. Its space group is B2/m.

The crystallographic orientation relationship is as follows:

$[001]_{\beta} // [001]_M$ and $[100]_{\beta} // [130]_M$. The β " phase is a needle shape and its longitudinal direction parallels to the direction of $[001]_{\beta}$.

The chemical composition of this phase is Si:Al:Mg=6:3:1.

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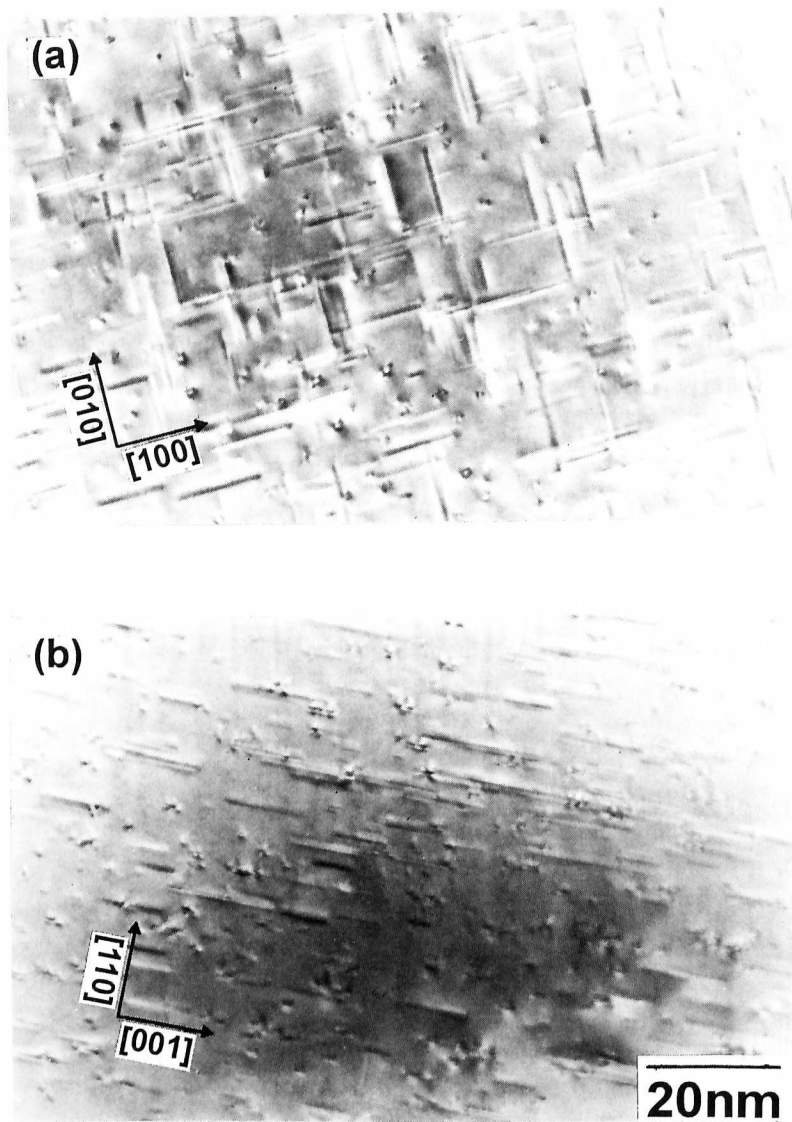


Fig.1 TEM bright field image of the Al-1.0mass%Mg-0.4mass%Si alloy aged at 423K for 2400ks.

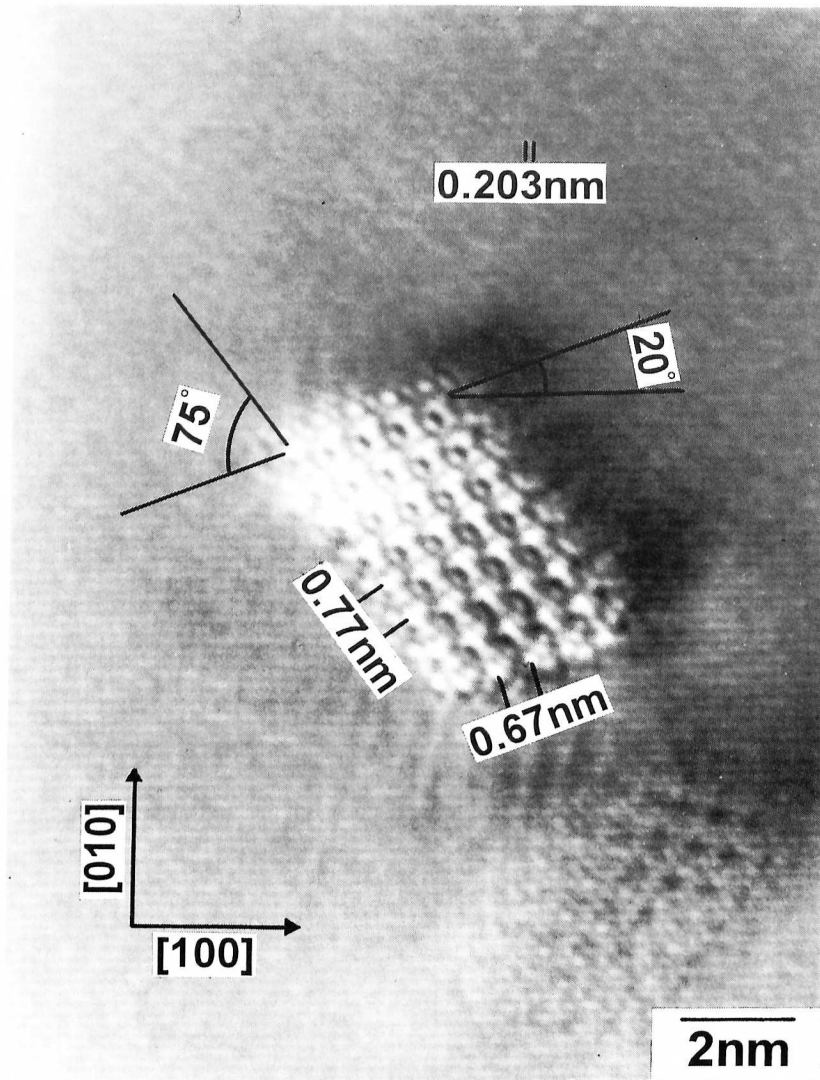


Fig.2 a high resolution image of the cross section of a precipitate.

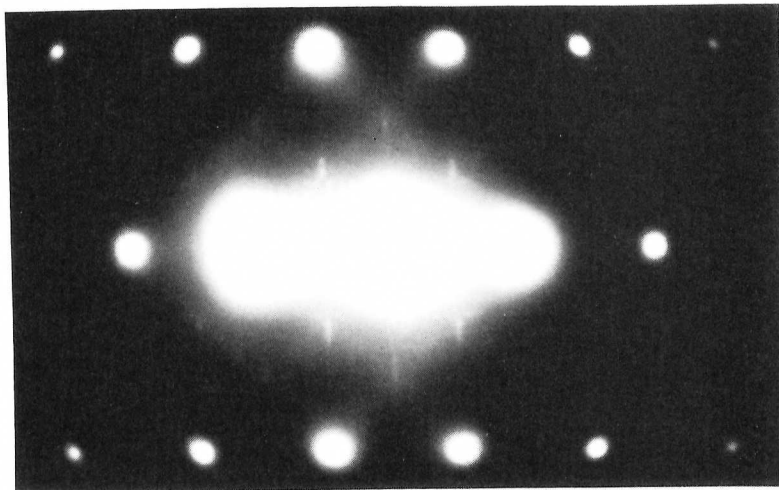


Fig.3 TEM observation along the normal direction to the longitudinal direction of the precipitate.

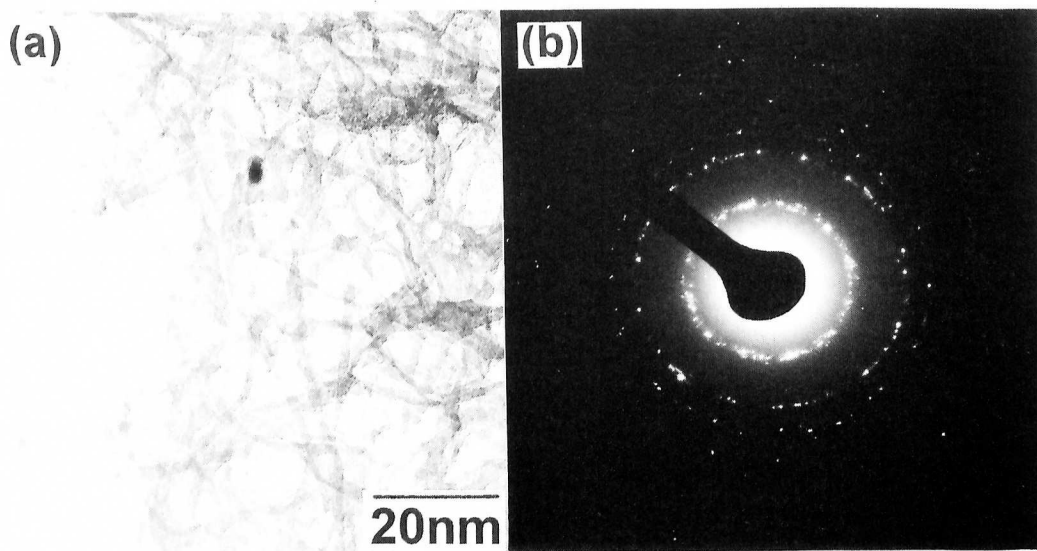


Fig 4 (a) a bright field image of the precipitate extracted by the thermal phenol method
(b) its selected area diffraction pattern