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Morphology and thermal stability of metastable precipitates formed in an Al–Mg–Si ternary alloy aged at 403 K to 483 K

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Abstract

Morphology and thermal stability of metastable phase precipitates formed in a balanced (Mg/Si=2) Al–Mg–Si alloy were studied by means of high-resolution electron microscopy (HRTEM) observations and differential scanning calorimetry (DSC) measurements. The present study revealed that the four types of precipitate morphology were found in the HRTEM images, that the heat change of the exothermic peak P in DSC measurements was mainly caused by the formation of type-2 and 3 precipitates in morphlogy and that the heat change of the exothermic peak Q which was yielded after the peak P probably corresponded to the formation of type-4 precipitates.

Keywords: Heat treatment; Electron microscopy; Microstructure; Al–Mg–Si alloy; Differential scanning calorimetry; Morphology of precipitates

1. Introduction

Aluminium-based Al–Mg–Si ternary alloys are widely used for practical applications, such as structural materials for transportation vehicles, vessels and construction. The precipitation behaviour in an Al–Mg–Si alloy has been generally considered to follow the sequence: supersaturated solid solution →clusters→metastable β″→metastable β′→stable β [1–4]. Some papers have claimed that other metastable phases are additionally formed during isothermal ageing [5–9]. There are, however, a number of ambiguities in the structure, alloy composition, and properties of metastable phases.

Differential scanning calorimetry (DSC) measurements and high-resolution transmission electron microscopy (HRTEM) observations are very helpful tools to investigate the precipitation behaviour in an Al–Mg–Si alloy, but there are few works which aim at quantitatively examining the relation between the thermal stabilities of phases detected by DSC measurements and the microstructures observed by HRTEM. This work was intended to clarify the precipitation behaviour and the thermal stabilities of the phases formed during isothermal ageing in an Al–Mg–Si alloy, using HRTEM observations and DSC measurements.

2. Experimental

Two Al–Mg–Si alloy specimens with balance compositions (Mg/Si=2 in atomic ratio) were used in this study. The chemical compositions of the alloy are listed in Table 1. The 75 mm-diameter billets were extruded at 773 K and the sheets were formed by cold rolling. The final test specimens

Table 1
Composition (at%) of the specimen studied

Alloy	Mg	Si	Al	Mg_2Si
B1	1.15	0.58	bal.	1.59
B2	0.75	0.41	bal.	1.12

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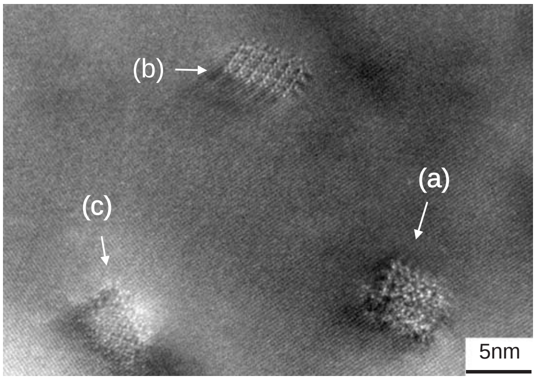

Fig. 1. HRTEM images of the precipitates for the B2 specimen aged at 433 K for 60 ks.

were solution-treated at 828 K for 3.6 ks and subsequently quenched in ice water. Then they were isothermally aged in an oil-bath at 403 K from 0.06 ks to 4800 ks, at 433 K from 0.06 ks to 900 ks, at 463 K from 0.06 ks to 600 ks, and at 483 K from 0.06 ks to 60 ks. The ageing time was chosen from an early stage to overage, to consider the wide range of precipitation process at these temperatures. A Topcon EM-002B microscope was used at 180 kV accelerating voltage. HRTEM images were taken along the crystallographic $\langle 001 \rangle$ orientation in the Al matrices. Nine reflections–the transmitted beam, four $\langle 200 \rangle$ and four $\langle 220 \rangle$ reflections symmetrically excited–were used to obtain the HRTEM images. The DSC measurements were conducted using a Rigaku TAS300-8230D instrument with a heating rate of 0.17 K/s.

3. Results and discussion

3.1. Morphology and the density of the precipitates

Fig. 1 shows a typical HRTEM image of the precipitates for the B2 specimen aged at 433 K for 60 ks. The precipitate indicated by arrow (a) shows an irregular arrangement of black–white contrast inside the precipitate. However, two other precipitates are simultaneously observed with parallelogram lattice fringes, as indicated by arrows (b) and (c). Careful HRTEM observations of the B1 and B2 specimens, which were aged for up to 7200 ks at four different ageing temperatures from 403 K to 483 K, revealed that the morphology of precipitates could be classified as four types, as shown in Fig. 2. Based on the HRTEM observations, we specified the following four types of precipitates. The type-1 precipitate is approximately 1 to 2 nm in size and shows a very weak contrast inside the precipitate. Type-2 is slightly larger, 3 to 8 nm in size, and ellipsoidal in shape with an irregular arrangement of black–white contrast internally. The type-3 precipitate shows a parallelogram-shaped image that is 3–8 nm in size, and has lattice fringes mutually intersecting at an angle of 65° to 80°. Type-4 is an ellipsoidal precipitate 6–11 nm in size, which is approximately twice as large as the type-3 precipitate. This type of precipitate has apparently similar contrast to the type-3 precipitate, but the lattice fringes intersect at an angle of 60°.

We first considered the change in density of each type of precipitate versus ageing time. Fig. 3(a), (b), (c) and (d) show the changes in the density of the precipitates of type-1, type-2, type-3 and type-4, respectively. The foil thickness was not precisely measured, but was estimated to be approximately 40 nm, referring to the data obtained by electron energy loss spectroscopy (EELS). In all cases, the peak position moved toward shorter times when the ageing temperatures increased. The type-1 precipitate density decreased as the ageing temperature decreased. The density curve for type-2 increased at lower temperatures and took a very high value at 403 K. The curve for type-3 is comparable to type-1 in height at 463 K and appeared later than type-1. The density of type-3 did not increase continuously, but saturated at 463 K. Although type-4 precipitates were observed at 483 K and 433 K, the density of these precipitates was relatively small.

3.2. Thermal stability

Fig. 4 shows the DSC curves for B2 specimens that were as-quenched and aged at 403 K. The curve for the as-quenched specimen has four large exothermic peaks at approximately 350 K (peak K), 550 K (peak P), 580 K (peak Q) and 730 K (peak S), respectively. We attributed peak K

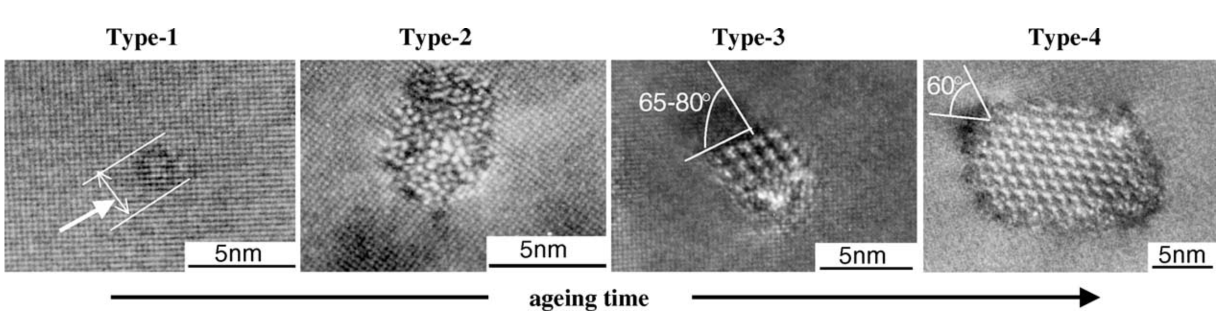

Fig. 2. Four types of precipitate morphology on the HRTEM images.