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A CALORIMETIRIC STUDY OF PRECIPITATION PROCESS IN A1-Li-Mg ALLOY

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### ABSTRACT

The precipitation process in a supersaturated Al-2.3wt%Li-2.3wt%Mg alloy has been studied by means of differential scanning calorimetry (DSC). It has been shown that the pre-precipitate something like G.P. zones is formed almost simultaneously with the  $\delta'$  phase during and after quenching. The activation energies for dissolution of the pre-precipitate and for precipitation of the  $\delta'$  phase have been determined by applying Ozawa's method to the DSC curve, to be 87±5 kJ/mol and 83±3 kJ/mol, respectively. The precipitation sequence in this alloy is suggested to be: supersaturated solid solution — pre-precipitate — metastable  $\delta'$  phase — equilibrium  $\delta$  and T phases.

#### INTRODUCTION

## EXPERIMENTAL PROCEDURE

An Al-2.3wt%Li-2.3wt%Mg alloy was prepared from high purity materials. They were melted together in an alumina crucible and cast into an iron mould under an argon gas stream. The ingot of 20 mm ø rod was annealed for homogenization at 773 K for 7 days. Specimens for the DSC measurements were made by hot forging and swaging the ingot to discs, 4.5 mm in diameter and 2.3 mm in thickness. All specimens were solution heat treated at 823 K for 2 h and quenched into ice water and aged at room temperature, 323 K, 353 K, Proceedings of ICTA 85, Bratislava 403 K and 433 K. The DSC measurement has been carried out over the temperature range from room temperature to 723 K during heating the specimen at a rate of 5 K/min under 1.01 X  $10^5$  Pa argon gas atmosphere.

# RESULTS AND DÍSCUSSION

## DSC Curves of As-quenched and Slowly Cooled Specimens

Fig. 1 shows the DSC curves of Al-2.3wt%Li-2.3wt%Mg alloy obtained during heating the specimen, immediately after quenching into ice water (a) and after slowly cooled from 653 K to room temperature (b). In as-quenched specimen, two heat absorptions denoted by R and T, and three heat evolutions denoted by Q,  $S_1$  and  $S_2$  are observed. In the slowly cooled specimen, only one heat absorption denoted by T is observed.



Fig.1: DSC curves of Al-2.3wt%Li-2.3wt%Mg alloy (a) as-quenched and (b) slowly cooled.

Comparing with the results by TEM[1], the heat evolution Q in Fig.1 (a) is attributed to precipitation of the  $\delta'$  phase from the supersaturated  $\alpha$  solid solution, the heat absorption R to dissolution of the  $\delta'$  phase. Comparing with the phase diagram of Al-Li-Mg system[4], the heat evolution S<sub>1</sub> and S<sub>2</sub> can be attributed to precipitation of the equilibrium  $\delta$  (AlLi) and T (Al<sub>2</sub>MgLi) phases, the heat absorption T to dissolution of the equilibrium  $\delta$  and T phases, respectively. The DSC curve of as-quenched specimen shows that the amount of heat absorption R is larger than that of heat evolution Q. This suggests that some amount of the  $\delta'$  phase has been formed before the DSC measurement, that is, during and immediately after quenching. DSC Curves of Aged Specimens

DSC curves of the specimens which were quenched from 823 K into ice water and aged at room temperature and 353 K for various ageing periods are shown in Fig.2 and Fig.3, respectively. The DSC curve of as-quenched specimen is also shown in Fig.2 and Fig.3 by dotted curve for comparison. It is noted that DSC curves in the temperature range between 550 K and 700 K does not affected by ageing at room temperature and 353 K. As shown in Fig.2, the heat evolution Q decreases with ageing time, while a new heat absorption P appears in



Fig.2: DSC curves of Al-2.3wt%Li-2.3wt%Mg alloy aged at room temperature.



Fig.3: DSC curves of Al-2.3wt%Li -2.3wt%Mg alloy aged at room temperature.

the temperature range lower than the heat evolution Q due to the precipitation of the  $\delta'$  phase. The heat absorption P may be attributed to dissolution of the pre-precipitate something to like G.P. zones formed during ageing at room temperature. The present results suggest that the pre-precipitate is formed almost simultaneously with the phase during and after quenching. As shown in Fig.3, the DSC curves of the specimens aged at 353 K are different from those aged at room temperature. Here, the heat absorption P appears at higher temperature range than that in Fig.2 and overlaps with the heat absorption R. The heat evolution Q decreases and disappears on ageing for more than 2 h. This suggests that stability of the pre-precipitate formed at 353 K is higher than that formed at room temperature.

# Thermal Stability of Pre-precipitate

 $\Delta H^*$  is defined as the change in enthalpy due to the formation of the pre-precipitate during ageing. T<sub>p</sub> appears to be related to a mean particle size of the pre-precipitate.  $\Delta H^*$  increases in the early stage of ageing and decreases after 240 h of ageing. However, T<sub>p</sub> increases almost linearly with the logarithm of ageing time even after



Fig.4: Change of  $\Delta H^*$  and  $T_p$  as a function of ageing time at 353 K. of ageing temperature.

Fig.5: Change of  $\ensuremath{\mathbb{T}}_p$  as function

620 h of ageing. This tendency shows two significant features: (1) The pre-precipitate may be stable even after prolonged ageing, and (2) Coarsening of the pre-precipitate increases linearly with ageing time. Fig.5 shows the relationship between Tp and ageing temperature in the specimen aged for 2 h, 24 h, and 120 h. T<sub>D</sub> curves seem to cross at one point, when they are extrapolated to higher temperature. The crossing point which appears to be related to the solvus temperature of the pre-precipitate approaches the solvus temperature of the  $\delta$  phase, T<sub>R</sub>, determined by the DSC measurements. Activation Energies for Precipitation Process

Table 1 shows the activation energies for the precipitation process in Al-2.3wt% Li-2.3wt%Mg alloy by Ozawa's method[5] and Table 1 Activation energies for reactions at peak P and Q.

n			ACTIVATION ENERGY(kJ/mol)	
	PEAK		OZAWA'S METHOD	NAGASAKI-MAESONO'S METHOD
	P	DISSOLUTION OF THE PRE-PRECIPITATE	87±5	
3	Q	PRECIPITATION OF THE & PHASE	83±3	80±2

Nagasaki-Maesono's method[6]. The values of the activation energies shown in Table 1 are lower than the activation energies for diffusion of Li(119.2 kJ/mol)[7] or Mg(114.8 kJ/mol)[8] in Al. Thus, in the precipitation process of Al-2.3wt%Li-2.3wt%Mg alloy, role of the quenched-in vacancy may be important.

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