PRECIPITATION BEHAVIOR IN Al-Mg-Si ALLOYS

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Abstract/ The precipitation behavior in Al-Mg-Si has been investigated by DSC measurement combined with TEM and Vickers hardness tests. The results obtained in this work suggest that two types of clusters appearing at low temperature are succeeded to β" and β' phases. A new interpretation of the decomposition has been proposed in this work.

Keywords) Al-Mg-Si alloy, precipitation behavior, DSC, TEM, Vickers hardness

Introduction

Al-Mg-Si alloy is one of the most promising light-weight materials for the practical uses. The study of the precipitation behavior in Al-Mg-Si alloy is of great importance not only to understand the fundamental process of the decomposition but also to consider the improvement of the physical and chemical properties for applications. The sequence of the precipitation in Al-Mg-Si alloy with a balanced or nearly balanced composition (Mg/Si~2/1 in atomic ratio) has been interpreted based on a decomposition along a pseudo-binary Al-Mg_2Si phase line : supersaturated solid solution → solute-rich cluster → metastable β"(G.P. zone) phase →metastable β' phase→stable β phase so far [1-3]. The relations of the metastable/stable phases and the structures of the precipitates have, however, not been fully determined by experiments yet, because of the difficulty of experimental identification of the phases.

In these few years, several works have claimed some modifications to the framework of the understanding, with respect to structure and composition [4-6]. Moreover, the origin of the exothermic peak with a shoulder which appears at low temperature (~310-370K) in the DSC curves has emerged as an important point to be investigated with the respect of step-aging.

The present authors have investigated the aging process of the Al-Mg-Si alloy by Differential Scanning Calorimetry (DSC), combining with Transmission Electron Microscopy (TEM), Vickers microhardness (Hv) tests, and reported that the thermal stability of the metastable phases β"and β' showed the behavior rather independent with each other[7,8]. The present work aimed at identifying the origins of the two sub-peaks which appear in the first exothermic peaks and considering the relation of the metastable/stable phases.

Experimental Procedure

The compositions of alloy specimens used in this work are listed in Table 1. All specimens were taken out from the slabs, which were also used for base materials in the previous work [7,8]. The solution treatment was carried out at 828K (555℃)

Table.1 Chemical Composition of the Al-Mg-Si alloys (At%)

	Mg	Si	Mg_2Si	excess Si	excess Mg
B1	1.28	0.56	1.68	- - -	0.16
B2	0.83	0.40	1.19	- - -	0.04
B3	0.54	0.26	0.77	- - -	0.02
S1	0.83	0.58	1.24	0.17	- - -
S2	0.83	0.71	1.25	0.29	- - -
S3	0.83	0.95	1.25	0.54	- - -
S4	0.82	1.03	1.23	0.62	- - -
M1	1.01	0.40	1.21	- - -	0.20
M2	1.18	0.37	1.12	- - -	0.44
M3	1.41	0.40	1.19	- - -	0.61
M4	1.56	0.39	1.16	- - -	0.78

for 3600s (1hour). After the heat treatment, the specimens were quenched into iced water. For step-aging, the specimens were annealed at 483K after pre-aging at 313K. Rigaku 8230D-type DSC apparatus was used for the DSC measurements. JEOL-100CX TEM was operated at 100kV for the bright field image observation. The Vickers hardness was examined by Shimazu HMV-2000 type Vickers microhardness tester.

Results and Discussion

I. Precipitation behavior of Al-Mg-Si alloys in step-aging

1.Vickers hardness tests

In order to know the influence of step-aging on the mechanical property, Vickers hardness tests were carried with the Al-Mg-Si specimens aged at 483K together

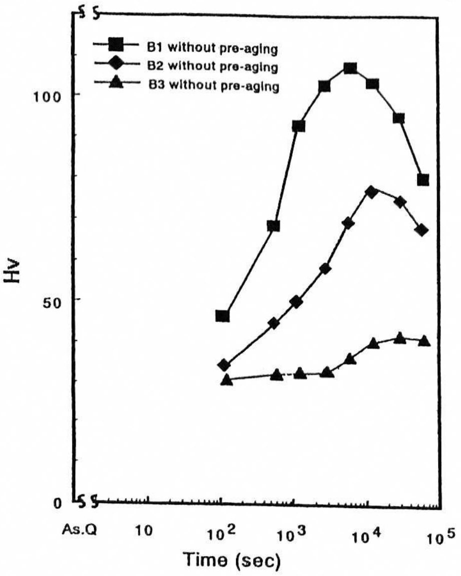

Fig.1 Vickers hardness of Al-Mg-Si specimen (B2) aged at 483K

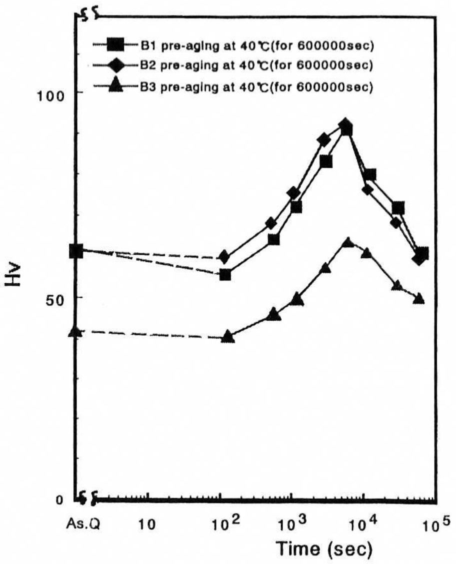

Fig.2 Vickers hardness of Al-Mg-Si specimen (B2) aged at 483K after pre-aging at313K for 600000s

with the specimens aged at 483K after pre-aging at 313K. Fig.1 shows the Vickers hardness obtained for the specimens simply aged at 483K. The Vickers hardness (Hv) curves are arranged with the order of solute concentration. The Hv curve for the specimen B3 is much less hardened by the aging at 433K. The Hv curve for the specimen B2 reaches peak position at slightly longer time for the specimen B1.This suggests that the precipitates formed in the specimens B1 and B2 evolve in a similar manner and the difference of the curves in height is due to the quantity of the precipitates. Fig.2 shows the Hv curves obtained for the specimens which are processed by the step-aging. Compared with the hardness shown in Fig.1, the Hv curve for the specimen B1 reduces the height at peak position, whereas the specimens B2 and B3 increase the hardness, by the step-aging, as shown in Fig.2. The feature seems to be related with the "positive" and "negative" effect of pre-aging, which were reported in previous investigations [9,10].

2.DSC measurements

DSC thermogram shows the phase transition due to the formation or dissolution of phases in Al-Mg-Si alloys, as shown in the previous work done by the present authors[7,8]. In this work, similar DSC measurements were also carried out to investigate the precipitation behavior in step-aging.

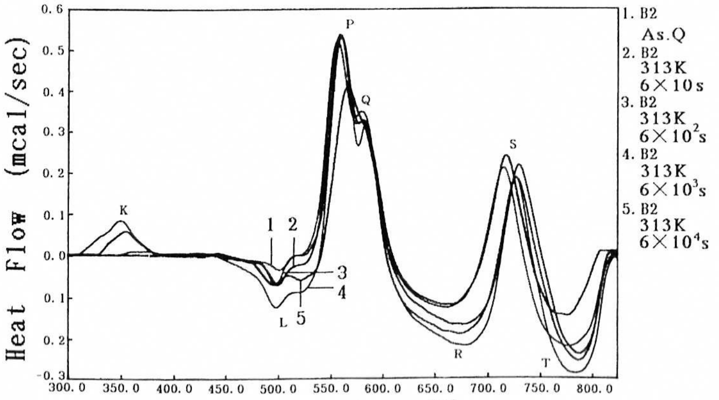

Fig.3 DSC curves for Al-Mg-Si specimen (B2) aged at 313K

Fig.3 shows the DSC curves which were obtained for the specimens B2 aged at 313K for 0s to 6.0x10^5s. The differences in shape of the curves are observed at the first and the second exothermic peaks K, P and the endothermic peak L. The results are compatible with the Vickers hardness shown in Fig.2, based on the fact that the most important precipitate for strengthening is the phase corresponding to the peak P , which was revealed the previous study by the present authors.

3.TEM observation

The microstructures of the Al-Mg-Si specimens processed by the step-aging were examined by TEM.Fig.4 shows bright field TEM images obtained for the specimen B1 which was aged at 483K for 6000s (near peak position) and for the specimen B1 which was aged at 483K for 6000s after pre-aging at 313K for 600000s. The TEM images show that both needle-like and granular precipitates are less distributed in the specimen processed by the step-aging than in the specimen simply aged at 483K for 6000s.

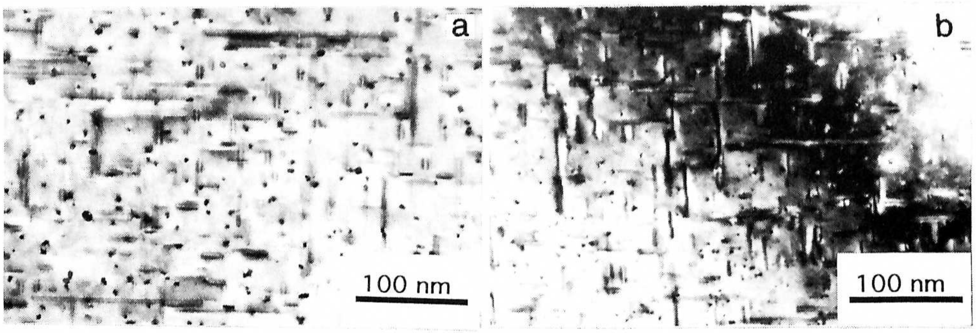

Fig.4 Bright field TEM images for Al-Mg-Si alloy specimen (B1) aged at 483K for 6000s and the specimen aged at 483K for 6000s after preaging at 313K for 600000s

II. Clustering at low temperature (~310-370K)

DSC measurements show that an exothermic peak is formed at ~340K. Careful examination shows that the peak consists of two sub-peaks. This feature was also confirmed by DSC measurements for the Al-Mg-Si alloys containing excess Si or excess Mg. Since the temperature at which the exothermic peak is close to room temperature and accordingly the clustering may give an influence to the aging sequence, the origin of the peaks and the influence to the subsequent precipitation behavior are important points to be investigated. Although the microstructures were also examined by TEM in this work, the detail of the distribution or structure of small solute clusters could not be obtained, due to the sizes. Therefore, DSC measurements were applied to several Al-Mg-Si specimens which have different solute compositions. Fig.5 shows the DSC curves for the specimens containing

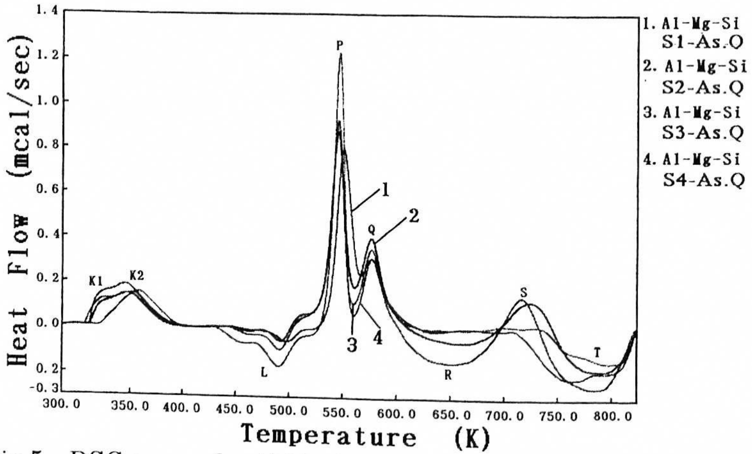

Fig.5 DSC curves for Al-Mg-Si specimens with different Si content

different excess Si. The exothermic peak K changes the shape, depending on the Si concentration. Namely, the sub-peak K1 increases the height with Si concentration except for S4, whereas the second sub-peak K2 is rather stable. This suggests that the formation of the first sub-peak K1 is related to the Si atoms but the sub-peak K2 to Mg content. The reason that the specimen S4 has a different shape is not clear. Si-rich clusters might be formed in Al-matrix if the Si concentration is high. Combining with the fact that the exothermic peaks P and Q are changed with the heat treatment or solute concentration, the present result suggests that the peaks P and Q take over the subpeak K1 and K2, respectively. That is, the subpeak K1 is related to the metastale β'' phase (as a precursor of the β'' phase) , both of which have a Si-rich composition. On the other hand, the subpeak K2 is related to the metastable β' phase (as a precursor of the β' phase). The cluster corresponding to the subpeak K2 and the β' phase are probably comprised with Mg-rich composition. Two paths through different compositions meet each other again at the stable β phase. This is an interpretation which is newly established based on the results obtained in this work and needs to be examined by further experiments. But a similar aging process was reported in Al-Cu-Mg alloy[11].

III. Cooling sensitivity of Al-Mg-Si alloys

In practical purpose, the cooling sensitivity of the Al-Mg-Si alloy is also important to be investigated. Fig.6 shows the DSC curves obtained for the Al-Mg-Si (B2) specimens which were cooling down from the temperature for solution treatment (828K) to room temperature with different rates. The size of specimens used in this experiment is 7mm in length, 7mm in width and 1mm in thickness. According to the previous work by the present authors, the precipitate corresponding to the exothermic peak P in the DSC thermogram has a most important role to strengthening of the alloys.

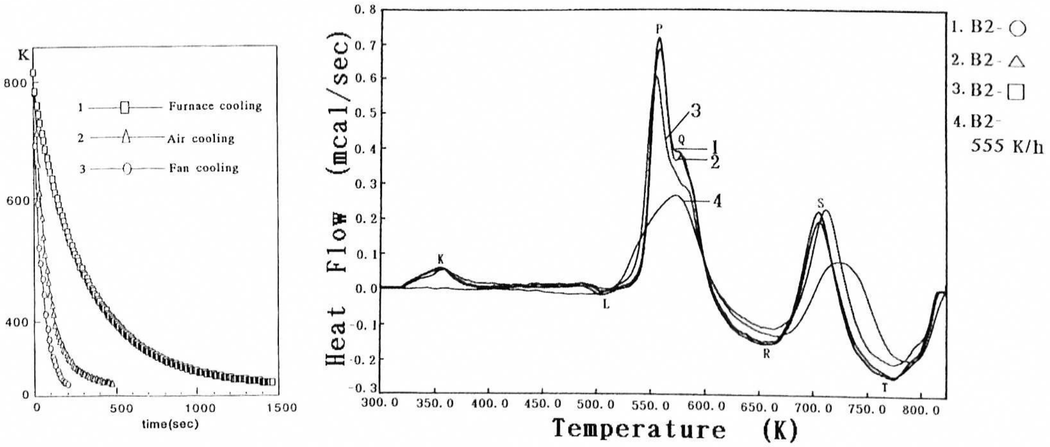

Fig.6 DSC curves for Al-Mg-Si specimen (B2) cooled at different rates

The cooling rates 1 and 2 do not much influence the formation of the precipitates appearing as the peak P. The depth of the valley between the peaks P and Q seems to be influenced by the difference of the cooling rate(cf.Fig.3). At the cooling rate 3, the peaks P and Q are both somewhat reduced but not the peak R. In the DSC curve at the cooling rate 4, the shapes of the peaks modifies significantly. That is, the furnace cooling reduces the peaks P, Q and R probably due to the formation of the phases.

Summary

The present investigation has shown the pre-aging at 313K particularly reduced the exothermic peak P, which has the most important role to strengthen the material. The magnitudes of the subpeak K1 and K2 are both dependent with the solute composition. The former peak seems to be a Si-rich cluster and the latter to be a Mg-rich cluster, respectively. The DSC measurements suggest that the aging process through two paths may simultaneously occur in Al-Mg-Si alloy.

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