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Natural Ageing of Al-Mg-Si Alloys

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Al-Mg-Si alloy possesses negative strength response after room temperature storage. Although it is already known that this is due to clustering during room temperature storage or natural ageing, the mechanism of clustering is still not yet fully understood. This is because the 'nano-clusters' are small and difficult to be revealed. Therefore, the aim of this research is to investigate the clustering mechanism of Al-Mg-Si alloy by using in-situ resistivity measurements and low temperature Differential Scanning Calorimetry (DSC). Hardness measurements were also performed to study the aging response during artificial ageing. Al-0.6wt%Mg-0.8wt%Si is used and the samples were solution heat treated and then ice water quenched. After quenching they were kept at room temperature for different time. Artificial ageing at 180°C was also performed after natural ageing. In this alloy, three types of clusters can be formed, but only 2 types of clusters, clusters 1 and 2, are dominating. By comparing the resistivity and DSC measurements, it is found that formation of cluster 1 is finished after about 1h of pre-aging at room temperature, while cluster 2 forms slowly during the 1st hour and finished forming after 2 weeks of pre-aging. The negative strength response is already settled after 10-18min of natural ageing and this is within the time range of the formation of cluster 1.

Keywords: *thermo-mechanical treatments, precipitation, age hardening, thermal analysis, Al-Mg-Si alloys.*

1. Introduction

In 6000 series Al-Mg-Si alloys containing typically 0.5–1 wt.% of both Mg and Si, the strength after Artificial Ageing (AA) is much slower and leads to lower peak strengths after natural Pre-Ageing (NPA) [1], expressed by the decrease of the number density and the increase in length of the strengthening phase β'' in the peak ageing condition [2]. This is so-called the negative strength response. Although a lot of research work based on different experimental methods has been performed to identify the processes during NPA and the reasons for negative strength response, the exact mechanisms are not yet known. Therefore, in this paper, low temperature DSC was performed with an emphasis on separating the various clustering reactions occurring isothermally at 'room temperature' especially within the first hour. The purpose of this work is to verify the clustering stages during low temperature ageing and their relationship to negative strength response.

2. Experimental Setup

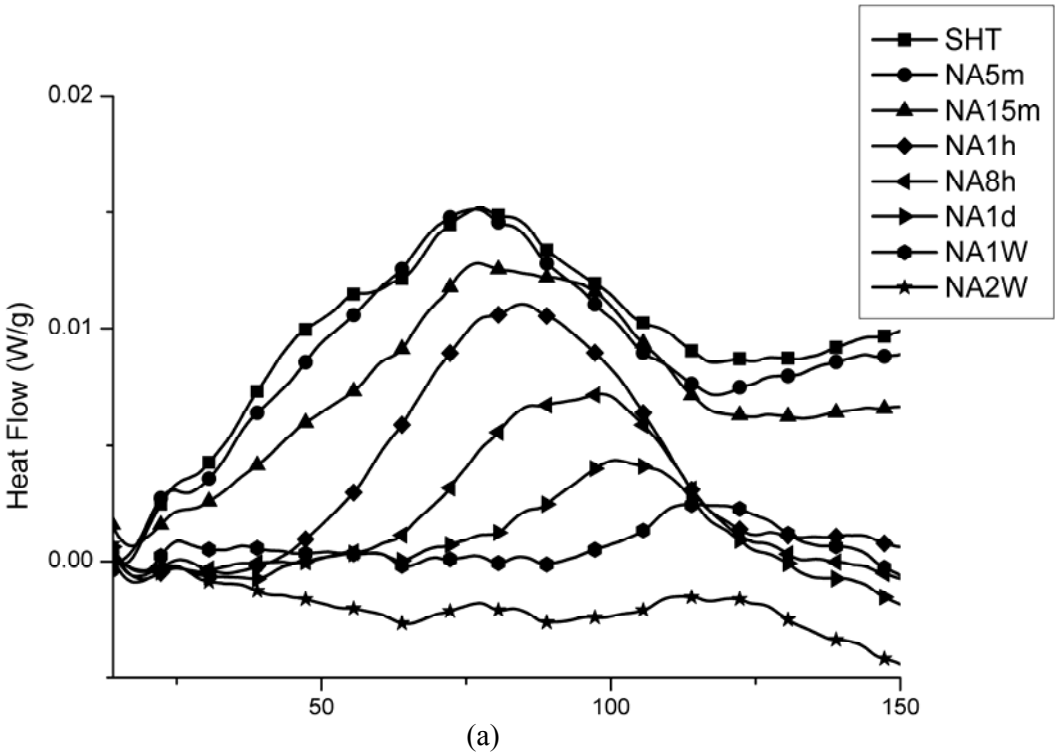
An alloy Al-0.59wt%Mg-0.82wt%Si was provided by Hydro Aluminium, Bonn. The alloy was based on ultra-pure elements, implying that the level of all impurities detected was <10 ppm. Solution heat treatment at 540°C for 1 h in an air circulation furnace was followed by ice water quenching. The samples were loaded into a differential scanning calorimeter pre-cooled to −50°C and were left there to equilibrate for 5 min before starting the measurement. The DSC used is a Perkin Elmer 'Pyris 6' located at the Department of Mechanical Engineering at The University of Hong Kong.

3. Results and Discussions

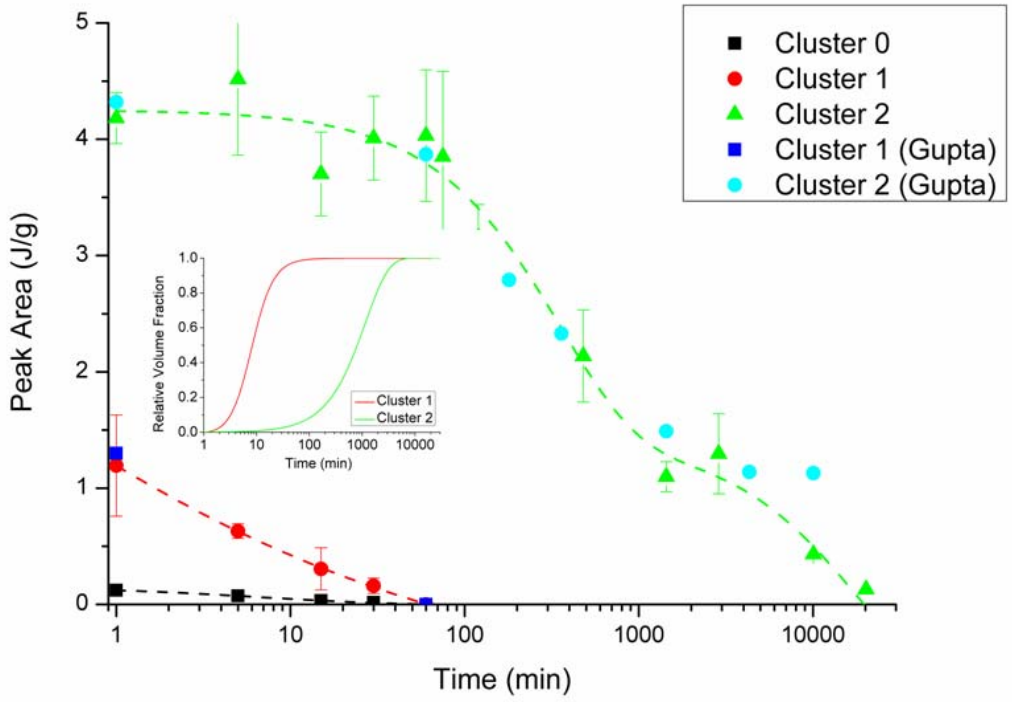
The DSC result of sample directly after solution heat treatment and quenching shows typical result of this type of alloy and shows individual exothermic reactions which corresponds to the

precipitation sequence assigned to these reactions as: Clusters → GP zones → β'' → β' → β [3]. Three overlapping cluster peaks could be observed in the temperature range from 10°C to 130°C. This has not been reported previously in the literature, most likely because unlike there we started DSC at −50°C. We first number the reactions 'Cluster 0 = C0', 'Cluster 1 = C1', 'Cluster2 = C2'. The heat release from C0 is very small compared to the other two cluster peaks and the peak temperature is very low, at around 25°C. The obvious question whether C0 is just a transient stage of C1 cannot be answered without further input from methods other than DSC.

Fig. 1(a) shows additional heat flow curves of samples after different NPA times. Only cluster peaks are shown. Clearly, the cluster peak areas decrease with NPA time. From this figure it is obvious that the peaks corresponding to C2 move to higher temperatures for longer NPA times. The cluster peaks are fitted by four Gaussian functions, after which the peak areas are calculated. Fig. 1(b) shows these various cluster peak areas against NPA time. The peak areas of C0 and C1 decrease simultaneously and have completely disappeared after 1h of NPA, while the peak area of C2 remains fairly constant within the error limits during the first hour of NPA. After 1 h, the peak area of C2 also starts to drop significantly and has disappeared after 2 weeks of NPA. The slight kink observed after about 1000 min of NPA is very close to the confidence limit of the measurement. Fig. 1(b) also displays data taken from the literature [3]. The agreement with the literature values is almost perfect even in terms of absolute values, except for the value after 1 week. This is notable, especially because in the work cited an alloy richer both in Mg and Si, namely 0.8%Mg, 0.9% Si, was used and one could expect higher heat flows. That this is not the case indicates that clustering is saturated above a certain (unknown) level of solute.



(a)



(b)

Fig. 1(a) *Heat flow curves of samples that were naturally pre-aged before the DSC experiments, showing only the clustering stages and* (b) *change of peak area of the clusters with NPA time. The small graph represents the relative volume fractions of C1 and C2 as a function of NPA time. Values taken from Ref. 3 are included.*

The inset in Fig. 1(b) shows the relative volume fraction against NA time and demonstrates that the formation of C1 is finished after 1 h of NA. This actually corresponds very well to the resistivity measurements of a similar alloy Al-0.5%Mg-1%Si described in Ref. 4, which show that a first stage of fast resistivity increase during NA turns into a slower increase after ≈1 h. This suggests that the regime of fast resistivity increase as given by Refs. 4 and 5 corresponds to the formation of C1. The formation of C2 is almost finished after 2 weeks of NA and the rate is much slower as represented by a smaller slope of curve C2 in Fig. 1(b). C2 starts forming after 1 h of NA, which suggests that C2 might form after C1 or that C1 even evolves into C2. Note that if one accepts the kink in Fig. 1(b) at ≈1000 min as real effect, it could be related to a further change in electrical resistivity [4].

It should also be noted that the negative effect on AA is being settled within the first 10 to 18 minutes of NPA [5]. This time period is within the formation process of C1. Therefore, from the inset in Fig. 1(b), we estimate that roughly 60% to 80% of C1 formation is already sufficient to fully establish the negative effect.

References

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