

## Effects of Natural Aging Conditions on the Bake Hardenability of Al-Mg-Si Alloys

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### Abstract

The effects of natural aging on the bake hardenability of three Al-Mg-Si alloys have been studied. The bake hardenability of an excess Si alloy during baking without natural aging is higher than that of the Al-Mg-Si alloys without excess Si. However, the hardness of the baked Al-Mg-Si alloy with a high Si content clearly decreased with the natural aging time before baking. The  $\beta''$  phase peak of the high bake hardened sample upon DSC analysis appeared at a lower temperature compared to that of the low bake hardening sample upon DSC analysis. The  $\beta''$  phase, which is located at a lower temperature upon DSC analysis, quickly precipitates during baking at 170°C, and the hardness is increased during the baking for 1.2ks at 170°C.

### 1. Introduction

Heat treatable Al-Mg-Si alloys have been the material of choice for automotive bodies. The aluminum sheets are formed in T4 and subjected to a heat treatment during the paint bake cycle at elevated temperature in an automotive company. The higher strength of the sheet after the paint bake cycle is better because of its high dent resistance. Therefore, the improvement of bake hardenability has been a concern by many investigators [1-5]. On the other hand, it is well known that the formation of clusters and zones in the alloys during aging is complicated, especially during natural aging and short artificial aging like the bake cycle. Although it is difficult to observe the clusters during natural aging by TEM, these affect the following precipitation. In this study, the effect of natural aging on the bake hardenability at 170°C of three Al-Mg-Si alloys has been studied using differential scanning calorimetry and electrical resistance measurement.

### 2. Experimental Procedures

The chemical compositions of the alloys for this study are shown in Table 1. The alloy sheets which were hot and cold rolled to a 1.0mm thickness were prepared. A schematic diagram of the heat treatment is shown in Figure 1. Solution treatment was carried out in a salt bath for 60s at 550°C followed by quenching into water at 0°C. The quenched samples were held in the water at 0°C for only 10s. The samples were naturally aged at 20°C for various times in a water bath. The bake hardening treatment at 170°C for 1.2ks or 86.4ks was then applied using an oil bath.

Table 1: Chemical compositions of the alloys used in this study (mass%).

alloy	Mg	Si	Fe	Al	Mg <sub>2</sub> Si	excess Si
Al-1Mg <sub>2</sub> Si	0.62	0.39	0.03	Bal.	0.98	0.03
Al-1.5Mg <sub>2</sub> Si	0.99	0.56	0.03	Bal.	1.53	-
Al-1Mg <sub>2</sub> Si-0.6Si	0.62	0.96	0.03	Bal.	0.98	0.60

The bake hardening was based on the Vickers hardness. The samples before and after the bake hardening treatment were analyzed by differential scanning calorimetry (DSC, Perkin-Elmer Pyris 1) using a heating rate of 40°C/min. The changes in the electrical resistivity during the natural aging and bake hardening treatments were measured by the DC four-terminal method.

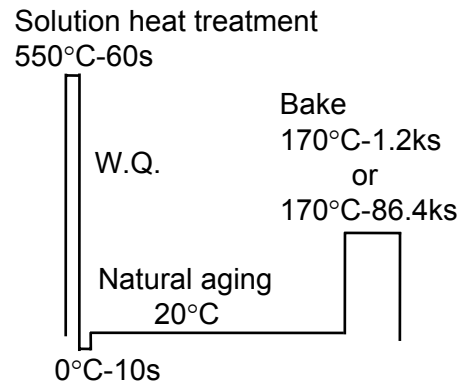


Figure 1: Schematic diagram of heat treatment.

### 3. Results and Discussion

#### 3.1 Bake Hardenability

The hardness change during the natural aging at 20°C after quenching and the hardness change in the naturally aged sample followed by baking at 170°C for 1.2ks or 86.4ks are shown in Figure 2. For the Al-1Mg<sub>2</sub>Si alloy, the hardness before the baking slowly increases with the natural aging time at 20°C and the hardness after the baking at 170°C

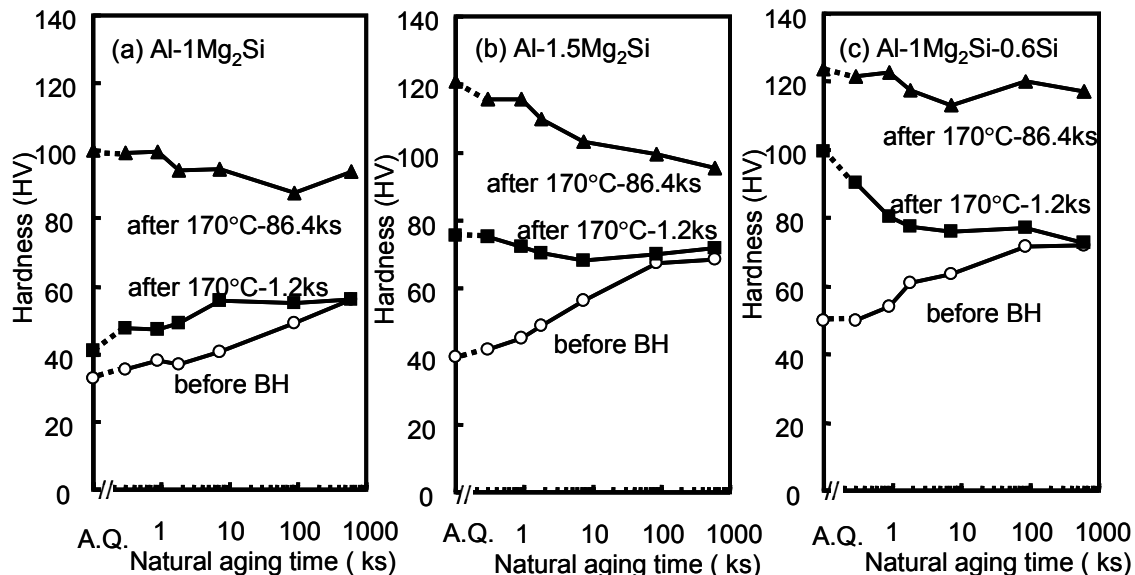


Figure 2 Changes in the hardness with natural aging at 20°C before and after bake hardening at 170°C for 1.2ks and 86.4ks of the alloys (a) Al-1Mg<sub>2</sub>Si, (b) Al-1.5Mg<sub>2</sub>Si and (c) Al-1Mg<sub>2</sub>Si-0.6Si.

for 1.2ks is also slowly increasing. The bake hardenability, which is expressed as the difference in the hardness before and after the baking, is small. The hardness of the sample baked at 170°C for 86.4ks slightly decreases with the natural aging time. On the

other hand, for the Al-1.5Mg<sub>2</sub>Si alloy, which has a higher Mg<sub>2</sub>Si content than the other alloys, the hardness after the baking at 170°C for 1.2ks decreases with the natural aging time. The decline in the hardness for the baking at 170°C for 86.4ks becomes greater than that for the baking at 170°C for 1.2ks. For the Al-1Mg<sub>2</sub>Si-0.6Si alloy which has a higher Si content than the other alloys but a low Mg<sub>2</sub>Si content, the hardness after the baking at 170°C for 1.2ks dramatically decreases with the natural aging time. However, the hardness of the sample baked at 170°C for 86.4ks slightly decreases with the natural aging like the Al-1Mg<sub>2</sub>Si alloy.

It is well known that the decline in the hardness with the natural aging time for the T6 tempered sample is mainly related to the Mg<sub>2</sub>Si content [6]. This agrees with these results for the baking at 170°C for 86.4ks. However, the decline in the hardness with the natural aging time for the sample baked at 170°C for 1.2ks increases with the Si content even if the Mg<sub>2</sub>Si content is low. These results show that the effect of the natural aging time for the baking at 170°C of 1.2ks is different from that for the baking at 170°C for 86.4ks.

### 3.2 Changes in the Electrical Resistivity

The changes in the electrical resistivity during natural aging for the quenched sample are shown in Figure 3. The electrical resistivity increases with the natural aging time for all the samples. The change in the electrical resistivity of the Al-1Mg<sub>2</sub>Si-0.6Si alloy is greater than that of the Al-1.5Mg<sub>2</sub>Si and Al-1Mg<sub>2</sub>Si alloys. This means the changes in the electrical resistivity are dominated by rather the total Si content than the Mg<sub>2</sub>Si content. These results suggest that the changes in the electrical resistivity during natural aging are mostly related to the clustering of the Si atoms.

For the Mg<sub>2</sub>Si balanced alloys, the change in the electrical resistivity of the Al-1.5Mg<sub>2</sub>Si alloy is greater than that of Al-1Mg<sub>2</sub>Si. Based on the assumption that the Si atoms might preferentially form the Si rich clusters, so insufficient number of Si atoms causes decreasing amount of  $\beta''$  precipitates during baking. As a result, a large decline in the hardness after the bake at 170°C of 86.4ks for the high Mg<sub>2</sub>Si alloy occurs.

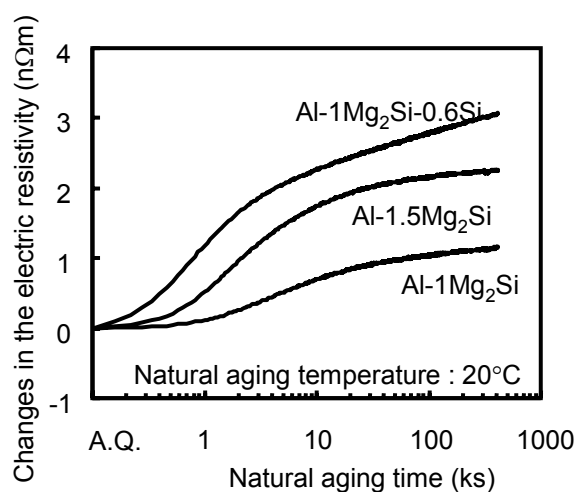


Figure 3 Changes in the electric resistivity during natural aging at 20°C for quenched sample

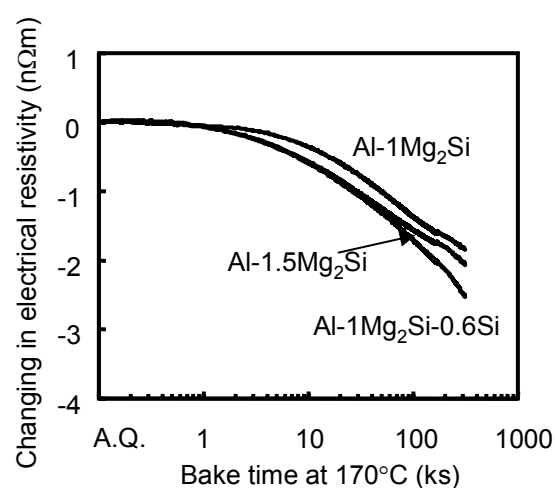


Figure 4 Changes in the electric resistivity during bake at 170°C after quenching without natural aging.

Changes in the electrical resistivity during baking at 170°C after quenching without natural aging are shown in Figure 4. The electrical resistivities for all the samples decrease with the baking time at 170°C. Although there is no difference in the changes for the electrical resistivity between these alloys up to 1.2ks, the bake hardening occurs up to 1.2ks as

shown in Figure 1. These results suggest that the electrical resistivity does not produce changes in the precipitation concerning the bake hardening.

### 3.3 DSC Analysis

DSC curves of the quenched sample and the baked sample at 170°C for 1.2ks without natural aging for the Al-Mg-Si alloys are shown in Figure 5. There are four peaks in the DSC curve of the Al-1Mg<sub>2</sub>Si-0.6Si alloy. Some reports suggest that peaks A and B are due to clusters [7], and peaks C and D are the precipitation of the  $\beta''$  and  $\beta'$  phases, respectively. The temperature of the  $\beta''$  phase for Al-1.5Mg<sub>2</sub>Si is higher than that for Al-1Mg<sub>2</sub>Si-0.6Si. Moreover, the temperature of the  $\beta''$  phase for Al-1Mg<sub>2</sub>Si is the highest and almost becomes the same temperature as the  $\beta'$  phase. After the baking at 170°C for 1.2ks, although the peak of  $\beta''$  phase for Al-Mg<sub>2</sub>Si-0.6Si completely disappears, the peak for the other alloys slightly remains. This suggests that the  $\beta''$  phase quickly precipitates during baking at 170°C for 1.2ks for the high Si content alloy such as the Al-1Mg<sub>2</sub>Si-0.6Si alloy.

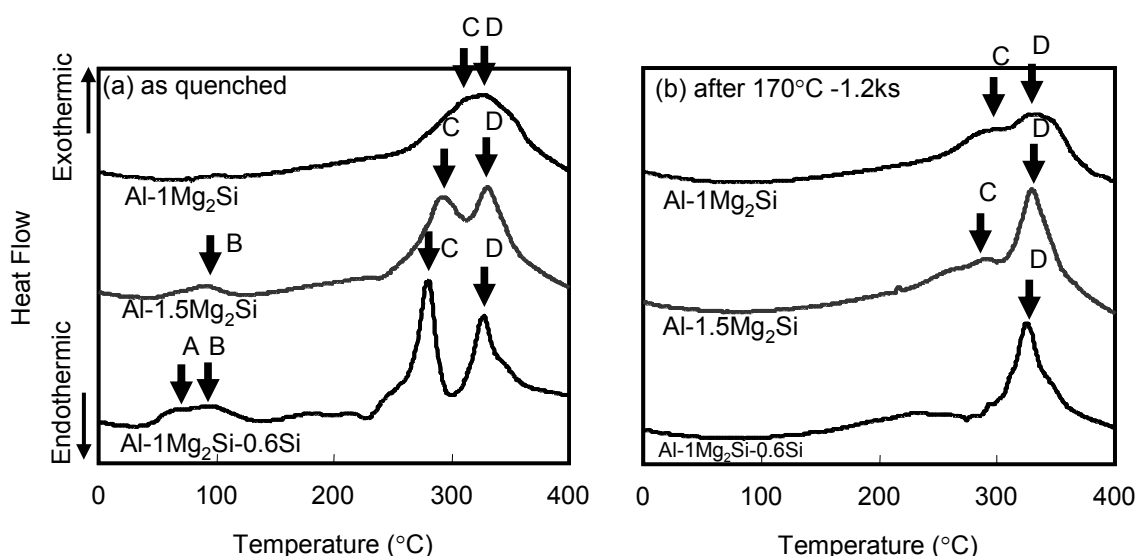


Figure 5 DSC curves for Al-Mg-Si alloys (a) as quenched and (b) after 170°C - 1.2ks bake.

DSC curves for the naturally aged sample at 20°C for various times after quenching of the Al-Mg<sub>2</sub>Si-0.6Si alloy are shown in Figure 6. The temperature of the  $\beta''$  peak shifts to a high temperature with the natural aging time, and peak A disappeared with the natural aging time. These changes happen after a very short natural aging time at 20°C. These results mean that the changes are related to vacancies.

DSC curves of the sample before or after baking at 170°C for 1.2ks for the quenched sample without natural aging and the naturally aged sample at 20°C for 1.8ks are shown in Figure 7. There are four peaks for the quenched sample before baking, but peaks A, B and C disappeared after the baking. This means that the formation of the cluster and the precipitation of the  $\beta''$  phase occur during the baking. However, peak C remains after the bake for the naturally aged sample at 20°C for 1.8ks. This suggests that the  $\beta''$  phase of the naturally aged sample slowly precipitated during the baking at 170°C for 1.2ks.

Changes in the temperature of the  $\beta''$  peak upon DSC and the bake hardening with natural aging at 20°C for the Al-1Mg<sub>2</sub>Si-0.6Si alloy are shown in Figure 8. The temperature of the  $\beta''$  peak is shifted to a higher temperature with the natural aging time of 1.8ks. The temperature still remains high after 1.8ks. On the other hand, the bake hardening is decreases with the natural aging time for the Al-1Mg<sub>2</sub>Si-0.6Si alloy. These suggest that the  $\beta''$  phase, which is located at a lower temperature on the DSC analysis, quickly precipitates during the bake at 170°C, and the hardness is increased by the  $\beta''$  phase during baking for 1.2ks at 170°C.

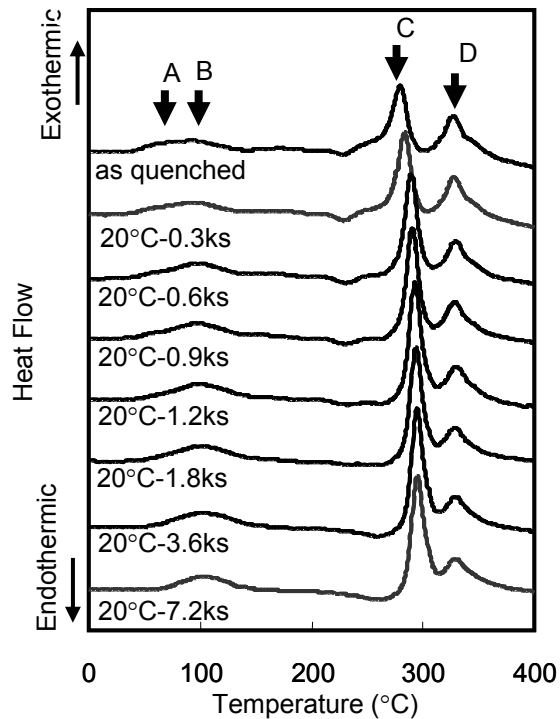


Figure 6 DSC curves for the Al-1Mg<sub>2</sub>Si-0.6Si alloy naturally aged at 20°C for various times after quenching.

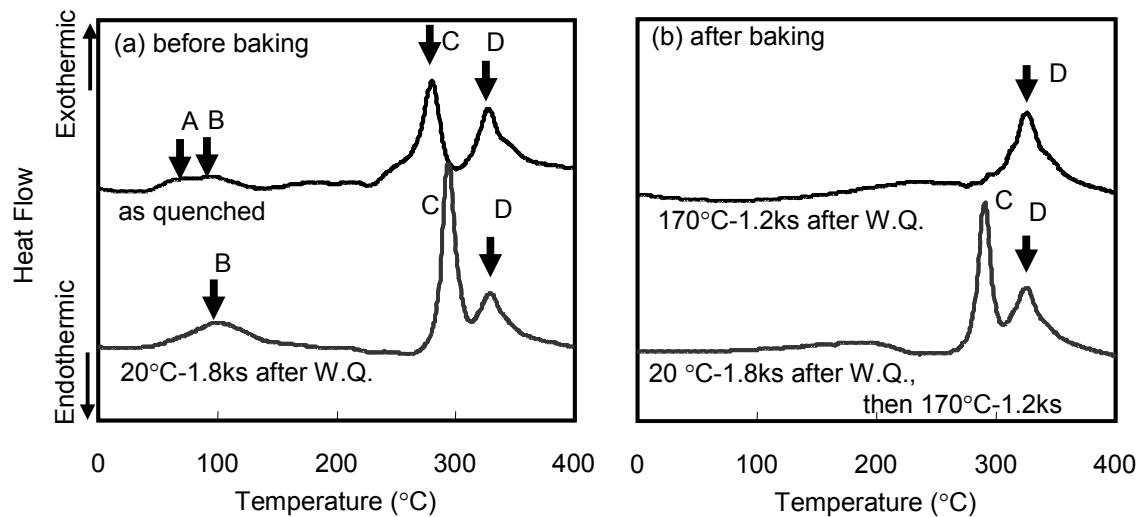


Figure 7 DSC curves for Al-1Mg<sub>2</sub>Si-0.6Si alloy without natural aging or with natural aging at 20°C for 1.8ks (a) before bake, and (b) after bake at 170°C for 1.2ks.

#### 4. Conclusions

- (1) The bake hardenability of excess Si alloy during baking without natural aging is higher than that of the Al-Mg-Si alloys without excess Si. However, the hardness of the Al-Mg-Si alloy with a high Si content after baking clearly decreased with the natural aging time before baking.

- (2) The  $\beta''$  phase peak of the highly bake hardening sample on the DSC analysis appeared at a lower temperature compared with the low bake hardening sample. The  $\beta''$  phase, which is located at a lower temperature on the DSC analysis, quickly precipitates during baking at 170°C, and the hardness is increased during baking for 1.2ks at 170°C.

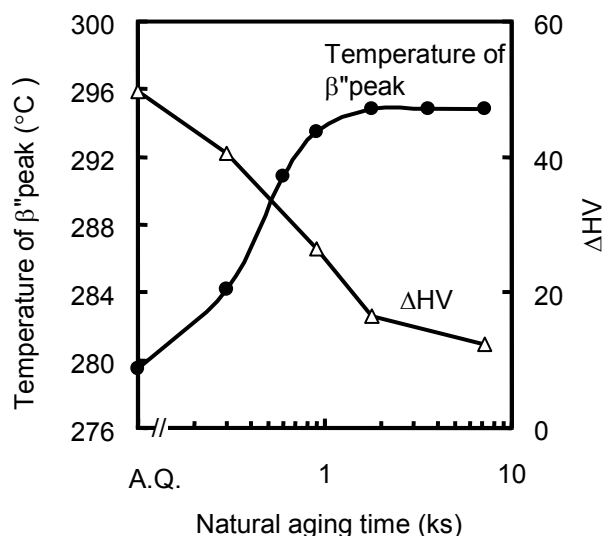


Figure 8 Changes in the temperature of  $\beta''$  peak and the  $\Delta$ HV with natural aging at 20°C for the Al-1Mg<sub>2</sub>Si-0.6Si alloy.

$\Delta$ HV: Changes in Vickers hardness from T4 to after bake hardening

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