

# Precipitation kinetics of Al–1.12 Mg<sub>2</sub>Si–0.35 Si and Al–1.07 Mg<sub>2</sub>Si–0.33 Cu alloys

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## Abstract

The kinetics of hardening precipitates of Al–1.12 wt.% Mg<sub>2</sub>Si–0.35 wt.% Si (excess Si) and Al–1.07 wt.% Mg<sub>2</sub>Si–0.33 wt.% Cu (balanced + Cu) alloys have been investigated by means of differential scanning calorimetry and hardness measurements. The excess Si enhances the precipitation kinetics and improves the strength of the material. On the other hand, however addition of Cu assist formation of the Q' phase which positively changed the alloy strength. The high binding energy between vacancies and solute atoms (Si and Mg) enhances the combination of Si, Mg and vacancies to form Si–Mg-vacancy clusters. These clusters act as nucleation sites for GP-zones. The coexistence of the β'- and Q'-precipitates in the balanced + Cu alloy results in a higher peak age hardening compared to the alloy with Si in excess.

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**Keywords:** Precipitation kinetics; Al–Mg–Si alloys; GP-zones; β''-Precipitates; β'; β-(Mg<sub>2</sub>Si) phase; Q' phase

## 1. Introduction

In recent years aluminum alloys are widely used in automotive industries. This is particularly due to the real need to weight saving for more reduction of fuel consumption and exhaust emissions [1]. Aluminum alloys of the 6xxx series, containing major elemental additives of Mg and Si, are now being used to replace steel skin or closure panels of various car models. For these reasons such alloys were subjected to several studies in the past two decades [2–8]. The strengthening of Al–Mg–Si alloys is based on a precipitation hardening process. Precipitation in Al–Mg–Si alloys is generally thought to be in the sequence [9–11]: α (SSS) → GP-zones → β''-precipitates → β' phase → β phase. In this sequence α (SSS) is the supersaturated solid solution. The Guinier Preston (GP) zones are generally considered either spherical- or lath-like clusters with unknown structure. The β'' phase is fine needle-shaped zones extending along the  $\langle 100 \rangle_{\text{Al}}$  direction, with a monoclinic structure [10,11]. The β' phase is rod-shaped precipitates with circular cross sections along the  $\langle 100 \rangle_{\text{Al}}$  direction, having a hexagonal structure with  $a = 0.705$  nm and  $c = 0.405$  nm [9,11], however,

other lattice parameters for β' have been also reported [12]. The β phase is usually Mg<sub>2</sub>Si platelets precipitating on the {100} plane of Al having the FCC CaF<sub>2</sub> structure with  $a = 0.639$  nm [11]. Although the precipitation process in Al–Mg–Si alloys has been extensively studied, the understanding of the hardening process is still incomplete. Any change in composition, processing and aging practices, etc. can affect the precipitation hardening behavior [11]. Cu-free Al alloys like AA6016 and AA6022 are currently used on a limited basis, although development of more formable versions of such alloys continues. The automotive alloys generally contain an excess of Si above that ratio required to form stoichiometric Mg<sub>2</sub>Si in order to improve the age hardening response during the automotive paint cure treatment [13–17]. The hardening in such alloys occurs mostly due to the precipitation of the metastable β''-precipitates. It has been suggested that the excess Si does not alter the precipitation sequence, structure and lattice parameters of different metastable precursors, but rather resulted in a modification of the composition and density of precipitates [5,6,8]. However, excess Si seems to promote the formation of additional phases during the later stages of aging [4,6,7], which does not contribute significantly to hardening [17]. In the balanced alloy, the ratio Mg/Si is close to 1.74. After forming the β'' phase in excess Si alloys, this ratio decreases to about 1:1 [13] or even less [14,15] however, a systematic

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study of the Mg/Si ratio with alloy composition is lacking [18].

The effect of Cu on the precipitation sequence of Al–Mg–Si alloy has been also previously investigated, albeit less extensively. It has been found that addition of Cu enhances the precipitation hardening kinetics [19–24]. While some of the investigators attribute this result to the refinement of the microstructure [19–21], others argue that the presence of Cu changes the precipitation sequences [22–24]. Miao and Laughlin [25] reported that Cu induces the Q phase and its precursor metastable phases and has a beneficial effect on the kinetics of artificial aging. For an alloy with 0.07 wt.% Cu, the precipitation sequences is GP-zones → needle-like  $\beta''$  phase → rod-like  $\beta'$  phase + lath-like Q' phase →  $\beta$  phase + Si [25]. On the other side, the precipitation sequence of an alloy with 0.91 wt.% Cu is, GP-zones → needle like  $\beta''$  phase → lath-like Q' phase → Q phase + Si [25]. Ravi and Wolverson [26] reported also that, the generic precipitation sequence in Al–Mg–Si–(Cu) alloys is: SS → GP →  $\beta''$  →  $\beta'$  →  $\beta$  (+Q). Here SS represents the supersaturated solid solution and the GP-zones are aggregates of solute atoms in the aluminum matrix that can be developed during the initial stages of precipitation. The  $\beta''$ -precipitate is generally presents in alloys aged to the maximum hardness and hence is considered important strengthening phase. Rods of  $\beta'$ -precipitates are formed in the over aged specimens. The  $\beta$ -(Mg<sub>2</sub>Si) phase is equilibrium one in the precipitation sequence. The equilibrium quaternary Q phase is observed in copper containing alloys, along with the  $\beta$  phase.

The precipitation sequence of Al–1 wt.% Mg<sub>2</sub>Si (balanced alloy) and in the balanced alloy with Si in excess has been investigated earlier [27]. On the other hand, a comparison between the balanced and balanced+Cu alloys has been also studied before [1]. As a result this work is aimed to clarify the kinetics of the evolved precipitates in both Al–1.12 wt.% Mg<sub>2</sub>Si alloy with excess Si and Al–1.07 wt.% Mg<sub>2</sub>Si alloy with Cu addition. Parameters sharing the mechanism of precipitate formation are also a subject of discussion in this study both qualitatively and quantitatively.

## 2. Experimental

Materials participating in the two alloys subjected to the present study and their percentage sharing (in wt.%) are given in Table 1. The differential scanning calorimetry (DSC) technique was used to follow the precipitation processes which take place in Al–1.12 wt.% Mg<sub>2</sub>Si+0.35 wt.% Si and Al–1.07 wt.% Mg<sub>2</sub>Si+0.33 wt.% Cu alloys during continuous heating with constant rates of quenched specimens from the solid solution state. Disc-shaped samples of 5 mm diameter and 0.5 mm thickness of average weight of ~24 mg were machined from the alloy ingot. The specimens were solution heat treated for 1 h at 803 K in a standard convection furnace and then quenched into a mixture of ice and water at equilibrium (~273 K). An annealed pure aluminum disc of similar shape and mass was used as a reference. Non-isothermal scans for the as-quenched

specimens using a DSC thermal analyzer (DSC-DO 8T-12TG01 type Shemadzu) at heating rates of 5, 7.5, 10, 15, 20, 30, 40 and 50 K min<sup>-1</sup> were carried out. Scans were performed between room temperature and 773 K in purified nitrogen flow at a rate of 30 ml min<sup>-1</sup>. The output signal was in mW and the net heat flow to the reference material was recorded with temperature. The peak temperature of the reaction processes was identified with an uncertainty of (±0.1 K) using the microprocessor of the thermal analyzer.

For isochronal and isothermal microhardness (HV) measurements, disc-shaped specimens of about 15 mm diameter and 2 mm thickness were used. The surfaces of the specimens were mechanically polished. The final polishing was achieved using (Struers) diamond paste of ~0.25  $\mu$ m grain size and a piece of cloth. Prior to every HV measurement, the specimen surfaces was gently polished by the 0.25  $\mu$ m diamond paste to remove the surface reactions that may occur during the heat treatment. The microhardness measurements were performed using Vickers method. Each given HV value is the average of at least ten readings distributed over the whole surface of the specimen without paying attention to the positions. The probable error in HV is due to the diagonal of impression measurement, which leads to an overall error in HV of about 3%.

In order to confirm the results obtained by DSC and HV measurements, transmission electron microscopy (TEM) examinations were performed at Toyama University, Japan. For microstructural examinations, a 200 kV-TEM (TOPCON-002B) was used. It was operated at 120 kV to avoid sample damage by the electron beam. Thin foils were prepared from the quenched and aged specimens. Thin discs of 3 mm diameter were punched from the foils and then electropolished by the twin-jet technique using a solution of 25% nitric acid and 75% methanol cooled to 243 K. The electropolishing was performed using a DC current source operating at ~20 V.

## 3. Results and discussion

### 3.1. Precipitation sequence in the Al–1.12 wt.% Mg<sub>2</sub>Si–0.35 wt.% Si alloy (Si in excess)

Fig. 1, presents the DSC scan obtained at a heating rate of 20 K min<sup>-1</sup> (as a representative case) for Al–1.12 wt.% Mg<sub>2</sub>Si–0.35 wt.% Si alloy immediately after solutionizing and quenching. The DSC curve shows five exothermic and two endothermic peaks. The five exothermic peaks are centered near 435 K (peak I), 560 K (peak III), 590 K (peak IV), 645 K (peak V), and 750 K (peak VII), successively. It is obvious from the insert of the figure that the exothermic reaction peaks are shifted

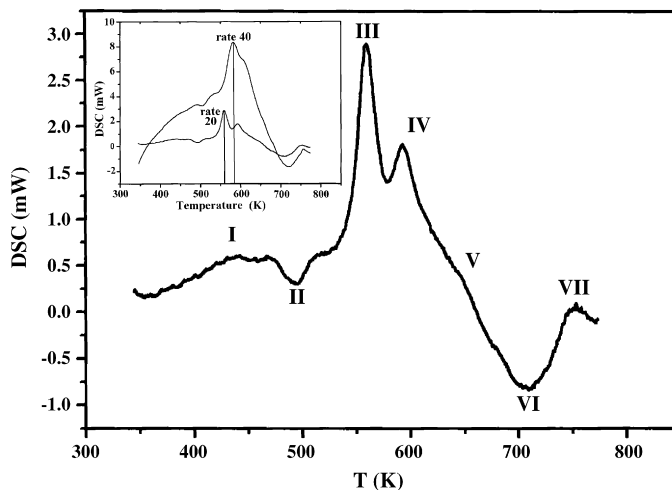


Fig. 1. Typical DSC scan at a heating rate of 20 K min<sup>-1</sup> for Al–1.12 wt.% Mg<sub>2</sub>Si–0.35 wt.% Si alloy with excess Si. The inset to the figure shows a change in a peak temperature with heating rate (vertical lines are just guide to the eye).

Table 1  
Materials participating in the composition (wt.%) of the two alloys

Sample type	Si	Fe	Cu	Mg	Mn	Zn	Mg <sub>2</sub> Si	Excess Si
Si-in excess	0.76	0.03	0.01	0.71	0.01	0.01	1.12	0.35
Balanced + Cu	0.45	0.03	0.33	0.68	0.01	0.01	1.07	0.06

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