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Effect of the Long Natural Aging on the Precipitation Sequence in Al-Mg-Si alloy

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Abstract. In this study, the effect of the long natural aging on the precipitation sequence of Al-Mg-Si alloy was investigated by differential scanning calorimetry and hardness examinations. This investigation revealed that the natural aging has a negative effect on the artificial aging. The reason behind the influence of natural aging on precipitation behavior of the Al-Mg-Si alloy is assumed to be the formation of clusters and G.P. zone during natural aging. The hardening mechanism during artificial aging was explained.

Introduction

Al-Mg-Si alloys are widely used in industry due to their mechanical and chemical properties. The strengthening mechanisms of Al-Mg-Si alloys by precipitation have attracted attention of many researchers. We notice that the most important research focused on the effect of aging on the precipitation sequence in these alloys. As reported by Li et al [1], the precipitation sequence during aging at constant temperature of quenched Al-Mg-Si alloys can be expressed as: Supersaturated solid solution (SSSS) → atomic clusters → G.P. zones → pre-β″→ β″→ β′/U1/U2/B′→ β (stable).

On the other hand, another simplified precipitation sequence of a solution and quenching that treated Al-Mg-Si alloy during artificial aging has been proposed as follows: Supersaturated solid solution (SSSS) → G.P. zones → metastable β″ precipitates→ metastable β′ precipitates→ stable β phase [2, 3].

However, a considerable confusion still exists concerning the precipitation process and hardening mechanism in these alloys because this precipitation sequence depends on some parameters, the alloy composition as indicated by many authors [4, 5, 6], the aging temperature of the quenched alloys or the previous heat treatment before aging [6]. According to Banhart et al [7], the effect of the natural aging (NA) on the precipitation sequence during artificial aging (AA) has received less attention. As indicated by Pogatsher et al [8] the storage period longer than a few minutes after quenching has proved too strongly retard the precipitation kinetics of the major strengthening phase β″ formed during artificial aging [9,10]. This last phenomenon is called negative effect because it reduces kinetics and achievable strength during artificial aging [10, 11]. In practical usage, it is difficult for the artificial aging to be performed immediately after solution heat treatment; which means that a delay at room temperature prior to artificial aging is unavoidable [12].

The purpose of this investigation is to explore and find out the long natural aging effect on the precipitation sequence in Al-Mg-Si by means of DSC, Vickers hardness measurements and SEM observations.

Experimental techniques

The material investigated in this study is an industrial Al-Mg-Si alloy which is used as an electric wire. The chemical composition of this material is indicated in Table 1.

Table 1 Chemical composition of investigated Al-Mg-Si alloy.

Elements	Al	Mg	Si	Cu	Fe
at.%	98.45	0.653	0.595	0.014	0.215

DSC tests are carried out using an analyzer Q 20 TA instruments (USA). The DSC measurements were conducted out constant heating rate (20°C/min) in a dynamic N_2 atmosphere to minimize oxidation. The samples for the DSC measurements were cut from rods with a diameter of 3 mm and 2-2.5 mm height, giving messes between 30 and 50 mg. Micro-Vickers hardness measurements are taken by Digital Micro-Vickers Hardness Tester type HVS -1000 Z under load of 200 g. Each hardness value is the average value from five individual tests.

Concerning the investigated of this industrial alloy, there were two categories of specimens, the first category was heat treated at 480°C for two hours, quenched in water and then natural aged, i. e. it had been stored at room temperature (R T) for 27 months before aging treatment at 150° C or DSC testing; however the second category was homogenized 6 h at 550°C and quenched in water before artificial aging treatment at 150°C, or DSC analyses. The quenched specimen for the Vickers hardness measurements were also aged at 150°C. To better compare the DSC trace with hardness variation, the specimens for Vickers hardness measurements were also heat treated in DSC instrument during non-isothermal heat treatments of Al-Mg-Si alloy.

Results and discussion

DSC tests

DSC tests after quenched alloy (non-natural aged)

Figure 1 shows the DSC curve of the quenched alloy during constant heating rate (20°C/min). By examining the DSC curve; seven peaks were recognized, four exothermic peaks (A, C, D and F) and three endothermic peaks (B, E and G), which correspond to four exothermic reactions and three endothermic reactions respectively. We attributed peak A to the formation of GP zone and peak B to its dissolution, whereas peaks C and D are associated with the formation of β″ and β′ phase respectively, while peak E is related to the dissolution of β′. However, peak F is associated with the formation of β stable phase. By raising temperature, this last phase dissolves in matrix (peak G), which forms the homogeneous single phase. Accordingly, the precipitation sequence can be summarized as follows:

Al supersaturated solid solution → G.P. zone (peak A) → dissolution of G.P. zone (peak B) → β″ (peak C) → β′ (peak D) → dissolution of β′ (peak E) → β (peak F) → dissolution of β (peak G).
We deduce that this precipitation sequence is consistent with the generally accepted precipitation in Al-Mg-Si alloys. It is in good agreement with the previous results [2, 3, 13 and 14].

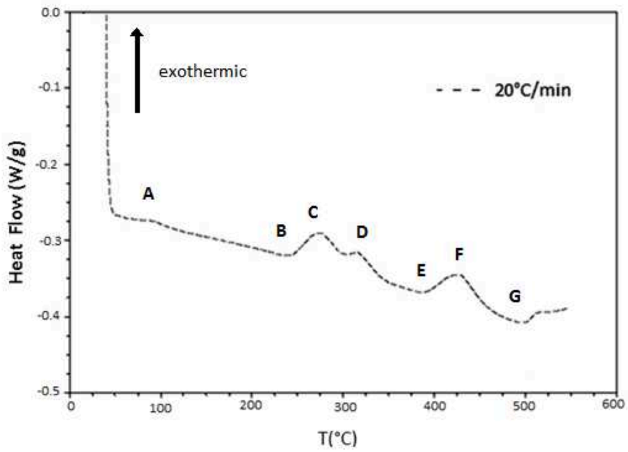

Fig. 1 DSC curves of the Al-Mg-Si alloy homogenized 6 h at 550°C, quenched in water and heated at 20°C/min.

DSC tests after natural ageing

Figure 2 presents the DSC curves obtained after natural aging of alloy which has been naturally aged during 27 months. This result shows some different features compared to the quenched alloy. The main observation is the absence of the first peak (A) which corresponds to the G.P. zone. This G.P. zone was precipitated during a natural aging, as reported by many researches [15, 16].

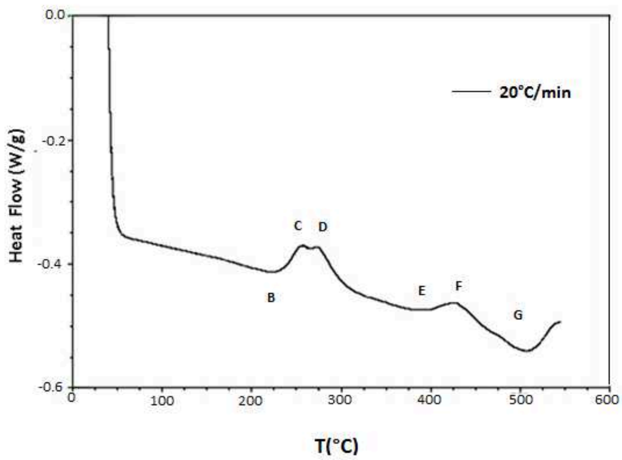

Fig. 2 DSC curve of pre-natural aged Al-Mg-Si alloy during 27 months (heating rate =20°C/min).

According to Hälldahl [17], during the storing of Al-Mg-Si alloy at RT, spherical GP- zones form, but during heating in the DSC instrument, the first thing that happens is the dissolution of these particulates (peak B).

Consequently, the precipitation sequence during AA of natural aged alloy is: Dissolution of G.P. zone (peak B) → β″ (peak C) → β′ (peak D) → dissolution of β′ (peak E) → β (peak F) → dissolution of β (peak G).

Hardness measurements

It is known that Al-Mg-Si alloys can be strengthened by the precipitation of several metastable phases which are produced by artificial aging [12]. During the aging treatment the supersaturation of solute atoms in aluminum matrix is gradually reduced. The strength is increased because a high density of fine coherent or semi-coherent precipitates nucleates and grows [5]. Figure 3 presents the effect of artificial aging time at 150°C on Vickers hardness of the alloy. The hardness of the alloy was substantially influenced by the aging times. The hardness starts to increase and reaches the maximum value at 30 min of aging at 150°C, which corresponds to the β″ phase formation. It is well known that the β″ phase greatly contributes to bake hardening in this alloy system [18]. According to Liao et al [19], the reason behind the impact of β″ on mechanical properties is not clear, which needs further investigation. After that, the hardness starts to decrease slightly with the appearance of the β′ and β precipitates as reported by Fang et al [5].

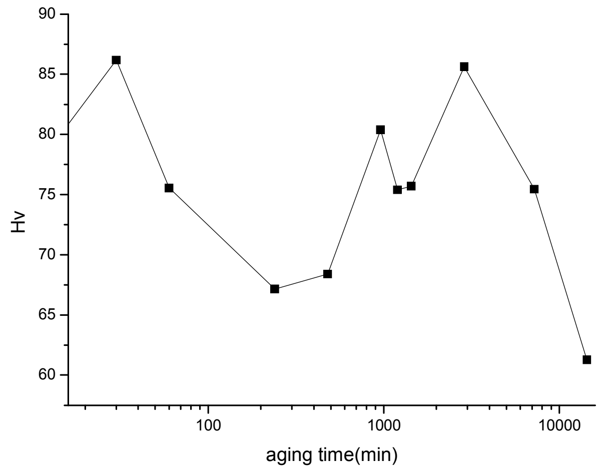

Fig. 3 Hardness variation versus aging time at 150°C of quenched Al-Mg-Si alloy.

The comparison between hardness and DSC curves is illustrated in figure 4 which displays the DSC curve obtained at heating rate of 20°C/min and the corresponding hardness curve of the as-quenches alloy. It is clear that alloy hardness follows roughly the same evolution of DSC curve and reflects the phase transformations during non-isothermal heat treatment of the quenched alloy.

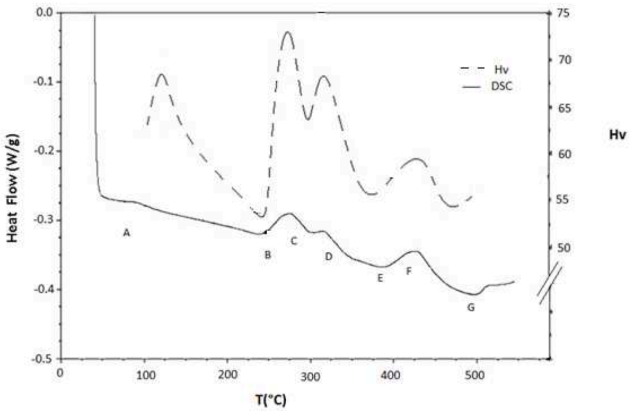

Fig. 4 DSC curve (heating rate 20°C/min) and the corresponding hardness plot for as-quenched Al-Mg-Si alloy.

On the other hand, figure 5 illustrates the negative effect of natural aging on the hardness of the alloy because this pre-natural aged alloy has a lower hardness than the non-natural aged alloy. Consequently, the NA has an adverse effect on hardness evolution during AA, the so-called 'negative' effect [7].

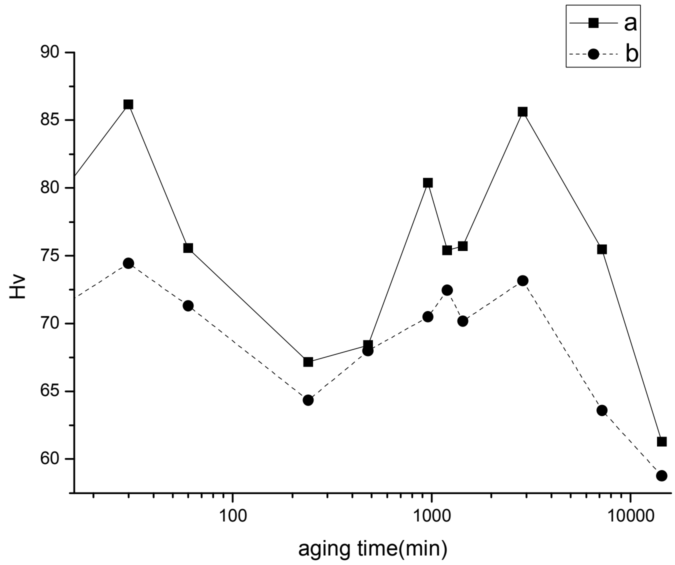

Fig. 5 Hardness variation versus aging time at 150°C of a) quenched Al-Mg-Si alloy and b) natural aged (27 months) Al-Mg-Si alloy.

Conclusions

In this study the precipitation sequence of Al-Mg-Si alloy was investigated. DSC tests show the effect of the natural aging on the precipitation sequence. For non natural aged, (quenched alloy), the precipitation sequence can be summarized as :(SSSS) → G.P. zone→ β″→ β′→β, however, for natural aged alloy, it can be expressed as: (SSSS) → β″→ β′→ β (Mg_2Si) which indicates that natural aging has an effect on the subsequent precipitation processes. The maximum hardness is obtained after aging at 150°C for 30 min. There was a close correlation between the DSC curves and hardness measurements. It was shown that the precipitation temperature shift to a higher temperature when heating rate increases. These findings will contribute to the comprehension of the precipitation sequence in Al-Mg-Si alloy.

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