Influence of Natural Aging on Artificial Age Hardening of an Aluminum Cast Alloy

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ABSTRACT

The natural age hardening and its influence on age hardening of Al11Si0.3Mg cast alloy were investigated by hardness measurement. Differential scanning calorimetry (DSC) and TEM analysis are used to analyze the age precipitation behaviors. The artificial age hardening response is significantly influenced by natural aging time before artificial aging. There is a harmful effect for natural aging 5-30 h on artificial age hardening response. TEM and DSC analysis results show that the precipitation of β″ and/or β′ phases during artificial aging is influenced obviously by natural aging time before heating. The relationship of artificial age hardening response with the precipitation of β″ and/or β′ phases has been discussed.

INTRODUCTION

Al-Si-Mg cast alloys are widely used in automotive industries for structural components as the results of their excellent castability, good corrosion resistance, low specific gravity, and good mechanical properties. The desired mechanical properties are achieved by T6 treatment, which follows the casting process[1]. T6 treatment is a process in which cast sample are solution treated and quenched followed by artificial aging. Fine distribution hardening phases which interact with dislocation to strengthen the alloy have precipitated by T6 treatment[2]. For Al-Si-Mg alloys, Mg and Si atoms over saturated in α (Al) form Mg/Si co-clusters and GP zones in the early stage of aging, and then coherent β″ and/or β′ phases precipitated with the duration of aging to produce the maximum strengthening of the material[3,4]. Heat treatment parameters, such as solution temperature and time, quenching rate, artificial aging temperature and time, could have a great influence on mechanical properties [5-8]. After solution treatment and quenching, alloys are in a non-equilibrium microstructure for α(Al) over saturated with Si/Mg atoms and vacancies. Holding quenched sample at room temperature, Mg/Si co-clusters and/or

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GP zones may precipitate [3]. There are some reports about the effect of natural aging time between quenching and artificial aging on artificial aging hardening response of aluminum alloys[9-11], but lack of systematic investigations of natural aging time on artificial age hardening Al-Si-Mg cast alloys.

This paper has studied the natural age hardening of Al11Si0.3Mg cast alloy and its effect on artificial age hardening response by hardness measurement, DSC and TEM analysis were used to investigate the precipitation behaviors related to different age hardening stages.

EXPERIMENTS

High purity Al99.7, Mg99.95, Si99 was used in preparation of experiment Al11Si0.3Mg alloy. The alloy was prepared in an electric resistance furnace. The melt was modified by Al-5Sr master alloy at 730℃ holding for about 30 min to ensure complete homogenization and modification, and then poured into the permanent mold. Samples were cast with mold temperature 100℃.

An electric furnace with a temperature control of ±3 ℃ was used for solution treatment and artificial aging. Cast samples of Al11Si0.3Mg alloy are solution treated 4 h at 545℃ followed by water quenched (called quenched sample). Quenched samples are aged at 150℃ for 20 h after different natural aging time (called T6 treatment). Hardness testing was carried out using a HB hardness tester. Each hardness value was the average of at least three measurements.

DSC analysis was performed using a differential scanning calorimeter (NETZSCH DSC 404). Non-metallic crucibles (Al$_2$O$_3$) were used. First, both of the reference crucible and sample crucible were empty, the cell equilibrated at 25℃ and then heated to 500℃ with a heating rate of 10℃ min$^{-1}$ under an argon atmosphere with a flow rate of 80 ml min$^{-1}$, DSC base curve was obtained. Second, a super purity aluminum specimen was placed in the reference crucible, a sample of equal mass was placed in the sample crucible, the obtained DSC base curve was selected as the corrective curve, upper operation was repeated consequentially, and DSC curve associated with transformation reactions of the experiment sample was obtained. Samples for DSC analysis are deposited in a -7℃ refrigerator.

TEM samples were cut to 0.5 mm thick by a line-cutting machine. Thin samples were mechanically polished to 0.1 mm and then thin foils transparent for electronic beam were obtained using an ion polish machine. Microstructures were observed using a H-800 TEM operated at 150 kV.

RESULTS AND DISCUSSION

Aging hardening and precipitation. Quenched samples of Al11Si0.3Mg alloy were held at room temperature for natural aging different times before artificial aging at 150℃ for 20 h. Fig.1 shows the natural age hardening behavior of the quenched sample (545℃ 4h + water quenched) and the artificial age hardening response with different natural aging time (545℃ 4h + water quenched) + nature aging different time + 150℃ 20h). After solution treatment and quenching, the structure of quenched sample is in non-equilibrium that α (Al) is over saturated with Si and Mg atoms and vacancies. Si/Mg/vacancy clusters precipitate gradually during natural aging that should be as the nuclei of GP zones hardening of α (Al) [2]. It can be seen from Fig.1 that hardness increases with natural aging time until
up to 100 h and is stable near peak hardness after natural aging more than 100 h for quenched sample. The slow natural age hardening speed can be explained by the small diffusion speed of Si and Mg atoms at room temperature. Artificial age hardening response varies with the natural aging time.

Figure 1. Hardness vs natural aging time.

There is a harmful effect of natural aging 5-30 h on age hardening response. This harmful effect of natural aging may be related to the microstructure formed during this stage. Heating the quenched sample immediately for artificial aging, the over saturated vacancies accelerate the diffusing of Si and Mg atoms to form GP zones that should be big enough as the nuclei of $\beta''$ phases during aging. The over saturated vacancies will accumulate and disappear gradually with natural aging. For the quenched sample natural aged more than 48 h, enough volume of GP zones have precipitated that should act as the nuclei of $\beta''$ phases during artificial aging. Moreover, nature aging quenched sample less than 48h, clusters of Si and Mg atoms should be the main precipitates. The size of Si and Mg clusters is not big enough as the nuclei of $\beta'$ phases and will dissolve during heating.

TEM microstructure for Al11Si0.3Mg alloy quenched samples natural aging 12 h and 48 h are shown in Fig.2(a) and Fig.2(b) respectively. The contrast effect shown in Fig.2 (b) for natural aging 48 h sample reflects the crystal distortion due to the precipitation of GP zones that hardening the alloy as shown in Fig.1. Comparing Fig.2(a) and Fig.2(b), little of precipitates appear for natural aging 12 h sample that consistent with the little natural aging as shown in Fig.1.

Figure 2. TEM micrograph for Al11Si0.3Mg quenched samples natural aging 12 h (a) and 48 h (b).
Fig. 3 shows TEM micrographs for T6 sample of Al11Si0.3Mg alloy natural aging 12 h and 48 h before artificial aging. Both coherent β” phases precipitated homogeneously and semicoherent β’ phases precipitated along with dislocations are detected by analyzing the selected area diffraction patterns for two samples. It can be seen that there are more coherent β” phases for natural aging 48 h sample followed by artificial aging. Comparing Fig. 2 (a) and Fig. 3 (a), little GP zones as the β” nuclei result in lack of β” precipitates and more β’ phases precipitated along with dislocations during artificial aging for natural aging 12 h sample. Moreover, GP zones precipitated after natural aging 48 h should be as the β” nuclei resulting in the high density β” precipitate by comparing Fig. 2 (b) and Fig. 3 (b). The lack of β” precipitates may be the causes of harmful effect for natural aging 5-30 h on age hardening response.

Precipitation analysis by DSC. For AlSiMg alloys, precipitates during aging are precursor phases of β(Mg2Si) [3]. According to the generally accepted sequential representations for precipitation in age hardenable alloys, the following stages GP zones→β”→β’→β(Mg2Si) or some of them may be associated with the aging curves [3].

DSC analysis was carried out on quenched samples natural aged 12 h and 48 as shown in Fig. 2, in order to investigate the precipitation behavior during artificial aging.

![Figure 3. TEM micrograph for Al11Si0.3Mg T6 samples natural aging 12 h (a) and 48 h (b) before artificial aging.](image)

![Figure 4. DSC curves at a heating rate of 10 °C/min.](image)
Based on the previous DSC investigations of Al-Mg-Si alloys [12], exothermic peak centered at around 100℃ is caused by GP zones precipitation; exothermic peaks centered at around 250℃ are caused by the precipitation of β″ and/or β′ phases. The age hardening response of the AlSiMg cast alloy depends on the size and amount of β″ and/or β′ precipitates.

It can be seen from Fig.4 that β″ and/or β′ precipitation peaks were detected in DSC curve for three samples and this precipitation peaks shift higher temperature for natural aging 12 h sample. The precipitation peak corresponding to GP zones didn’t appear. For quenched sample natural aged for 12 h, an endothermic peak at about 100℃ appears that should be caused by the dissolution of clusters formed during natural aging, which suggests that clusters should precipitate during natural aging 12 h and will dissolve in the heating process. For quenched sample natural aged for 48 h, endothermic peak doesn’t appear. This means that the GP zones precipitated after natural aging 48 h as shown in Fig.2(1) is big enough as the nuclei of β″ phases in the heating process.

The β″ and/or β′ precipitation peaks shift higher temperature for natural aging 12 h sample shown in Fig.4 means that β″ precipitation dynamic increases because of the less homogeneous nuclei and the disappear of over saturated vacancies. More β′ phases precipitate along with dislocations as shown in Fig.3(a) is consistent with this phenomena.

CONCLUSIONS

(1) Natural aging quenched samples of Al11Si0.3Mg cast alloy at room temperature, hardness increases obviously after natural aging more than 20 h and is stable near peak hardness after natural aging more than 100 h. Artificial age hardening response varies with the holding time at room temperature before heating. Artificial aging immediately or after natural aging more than 48 h, artificial age hardness response is higher enough. There is a harmful effect for natural aging 5-30 h on age hardening response.

(2) For quenched sample natural aging 48 h, GP zones have precipitated that should act as the nuclei of β″ phases during artificial aging. For quenched sample natural aging 12 h, clusters of Si and Mg atoms should be the main precipitates that will dissolve during heating and resulting in the lack of β″ precipitates.

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