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EFFECT OF PRE-AGING ON ARTIFICIAL AGE-HARDENING BEHAVIOR OF Al-Mg-Si ALLOYS WITH Mg + Si = 1.5 MASS%

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ABSTRACT

In this study, hardness test, differential scanning calorimetry (DSC) and transmission electron microscopy were carried out to investigate the age-hardening behavior of Al-Mg-Si alloy with a Mg+Si concentration of 1.5 mass%, and a Mg/Si ratio of 1.0. Pre-aging treatment was conducted for the solution-treated and then water-quenched specimen at 100, 80, 60, 40, 20 (natural aging), 0 and -20 ˚C for 20 min or 5 h, followed by artificial aging at 170 ˚C for 20 min. It was found that when pre-aging time is prolonged from 20 min to 5 h, the bake-hardening response is deteriorated irrespective of the pre-aging temperature. In particular, the specimens subjected to pre-aging at 20-40 ˚C for 5 h hardly increased the hardness with the subsequent artificial aging. From the results of DSC measurement, it was suggested that as pre-aging temperature is lowered, the amount of solute clusters formed during pre-aging, i.e. Mg-Si clusters and Si-rich clusters, is decreased, but the ratio of Si-rich clusters is increased, resulting in the deteriorated bake-hardening response.

KEYWORDS

Al-Mg-Si alloy, Bake-hardening response, Pre-aging, Precipitation, Solute clusters

INTRODUCTION

In recent years, Al-Mg-Si alloys have been widely used for automotive body panels due to their excellent characteristics such as high strength-to-weight ratio and good formability. Their strength is increased not only by artificial aging during paint-bake treatment; i.e. “bake-hardening”, but also when kept at room temperature (RT); i.e. “natural aging”, in association with the suppressed bake-hardening response. Such a “negative effect of two-step aging” has been a serious problem because Al-Mg-Si alloy coils are unavoidably maintained at RT in the manufacturing process of body panels; e.g. shipping and storage. However, it was reported that pre-aging above 70°C before the two-step aging can restore the bake-hardening response, and such an improvement is related to the formation of solute clusters (Sato, 2006; Abid et al., 2010; Takaki et al., 2014; Zandbergen et al., 2015). From the comprehensive analysis by three-dimensional atom probe (Aruga et al., 2014 and 2015), it is believed that Mg-Si clusters formed during pre-aging transform into the strengthening β" phase, whereas Si-rich clusters formed during pre-aging act as a detrimental precursor of β" phase. In this study, it was aimed to investigate the effect of pre-aging conditions, especially pre-aging temperature and time, on the formation of solute clusters during pre-aging, in order to elucidate the mechanism of the age-hardening behavior of an Al-Mg-Si alloy.

EXPERIMENTAL

Al-Mg-Si alloy sheet with a Mg+Si concentration of 1.5 mass% and a Mg/Si ratio of 1.0 was utilized. The chemical composition of the alloy is listed in Table 1 with its abbreviation for mass% of Mg and Si (i.e. 7M8S indicates ~0.7 mass%Mg and ~0.8 mass%Si). All specimens were subjected to solution heat treatment at 560°C for 30 min and then water-quenched into RT. Pre-aging treatment at 100, 80, 60, 40, 20, 0 and –20°C for 0 min (i.e. directly artificial-aged), 20 min or 5 h was carried out, followed by artificial aging at 170°C for 20 min without natural aging. The applied aging conditions were designated as artificial-aged ‘A_z’, pre-aged ‘$P_{x°Cy}$’ and pre-aged + artificial-aged ‘$P_{x°Cy}A_z$’, respectively (The subscripts x, y and z represent pre-aging temperature, pre-aging time and artificial aging time). Vickers hardness of each specimen was measured by a Matsuzawa MMT-X1 tester with a load of 4.9 N and a dwell time of 15 s. Differential scanning calorimetry (DSC) was carried out with a constant heating rate of 10°C/min.

Table 1. Chemical composition of the investigated alloy

Alloy	Elements (mass%)			Mg+Si (mass%)	Mg/Si
	Mg	Si	Al		
7M8S	0.73	0.81	Bal.	1.54	1.04

RESULTS AND DISCUSSION

Figure 1a shows the attained Vickers hardness of $P_{x°C5h}A_{20min}$ specimens. The result of A_{20min} specimen is also shown for demonstrating their negative effect of two-step aging. HV(AQ), ΔHV(P) and ΔHV(A) represent the hardness in the as-quenched condition, the increments of hardness during pre-aging and artificial aging (i.e. bake-hardening response), respectively. It is obvious that the hardness of $P_{0\text{-}100°C5h}A_{20min}$ specimens is smaller than that of A_{20min} specimen and the smallest hardness is obtained when pre-aged at 20°C. On the other hand, $P_{-20°C5h}A_{20min}$ specimen exhibits the same attained hardness as A_{20min} specimen, suggesting that the storage in refrigerator does not affect the age-hardening response of this alloy. From Figure 1b where ΔHV(P) and ΔHV(A) are compared, it was found that as pre-aging temperature is lowered, ΔHV(P) is decreased in both of two temperature ranges; i.e. higher temperature range from 100 to 60°C and lower temperature range from 40 to –20°C, whereas ΔHV(A) is increased only in lower temperature range (*cf.* In higher temperature range, higher ΔHV(A), in comparison with $P_{20°C5h}A_{20min}$ (natural aged) specimen, was obtained irrespective of pre-aging temperature).

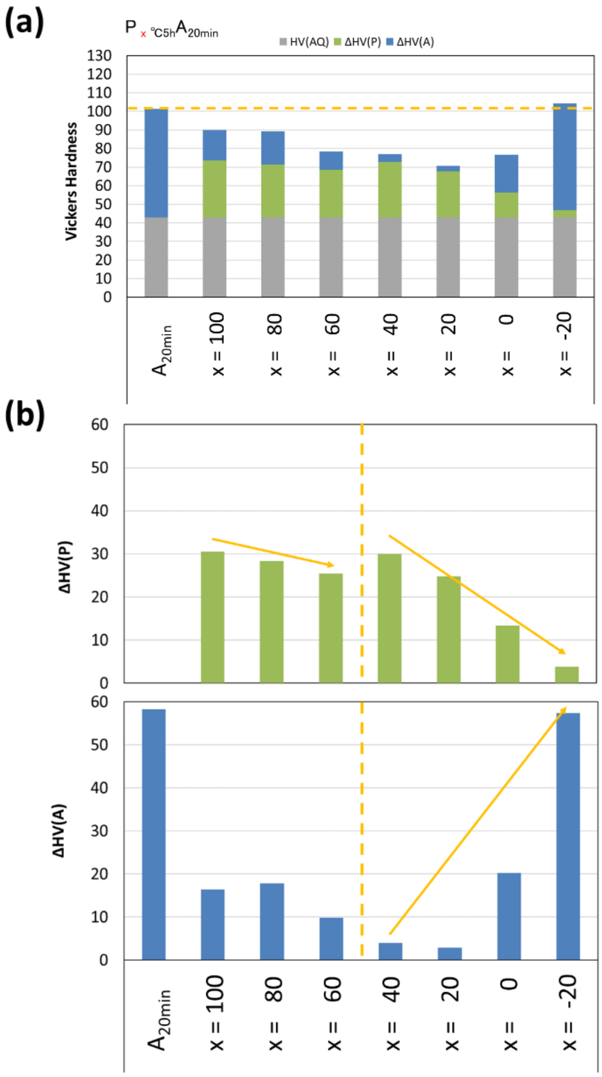

Figure 1. (a) Attained Vickers hardness of A_{20min} and $P_{x°C5h}A_{20min}$ (x=100 to –20) specimens with composition of 7M8S. The contribution of ΔHV(P) and ΔHV(A) is compared in (b)

Figure 2 represents the attained Vickers hardness, ΔHV(P) and ΔHV(A) of A_{20min} and $P_{x°Cy}A_{20min}$ specimens (x = 100, 60 and 40, y = 20 min and 5 h). It is obvious that when pre-aging time is prolonged, the attained hardness is decreased, ΔHV(P) is increased, and ΔHV(A) is decreased.

Figure 3 shows DSC curves of $P_{x°Cy}$ specimens (x = 100, 60 and 40, y = 20 min and 5 h). According to previous studies (Edwards et al., 1998; Zhen et al., 1997; Gupta et al., 2001, Serizawa et al., 2008), exothermic peaks (i), (iii) and (iv) and endothermic peak (ii) can be assigned to the formation of Mg-Si clusters, β” phase and β’ phase, and the dissolution of Si-rich clusters, respectively. Therefore, it is likely that the area of peak (i) decreases if the amount of solute clusters formed during pre-aging is increased (in other words, the residual solute concentration in the matrix after pre-aging is decreased), whereas the area of peak (ii) increases if the amount of Si-rich clusters formed during pre-aging is increased. In this study, to quantify the area of those peaks, a baseline was rationally assumed for each DSC curve, and an integrated value of heat flow between the peak-start and peak-end temperatures was calculated (Although the value may vary to some extent depending on how the baseline is drawn, the authors believe that the order of the magnitude does not change under the investigated pre-aging conditions). It was confirmed from Table 2 that as pre-aging temperature is lowered, the area of peaks (i) and (ii) increases either in $P_{x°C20min}$ specimen or in $P_{x°C5h}$ specimen. This suggests that the total amount of solute clusters formed during pre-aging is decreased, but the amount of Si-rich clusters, and thus the ratio of Si-rich clusters among those clusters are increased with lowering pre-aging temperature (In fact, no peaks (i) and (ii) in $P_{100°C\ y}$ specimen confirm that only Mg-Si clusters are formed during pre-aging at 100°C). On the other hand, the amount of solute clusters is increased with increasing pre-aging time because the area of peak (i) decreases in Table 2, and ΔHV(P) is increased in Figure 2b. In $P_{60°C\ y}$ and $P_{40°C\ y}$ specimens, furthermore, the area of peak (ii) increases with increasing pre-aging time, suggesting that the amount of Si-rich clusters is increased as pre-aging time is prolonged (The ratio of Si-rich clusters appears to be the highest when pre-aged at 40°C because of the most decreased ΔHV(A) in Figure 2b). This deduces again that Si-rich clusters formed during pre-aging inhibit the formation of β" phase in the subsequent artificial aging.

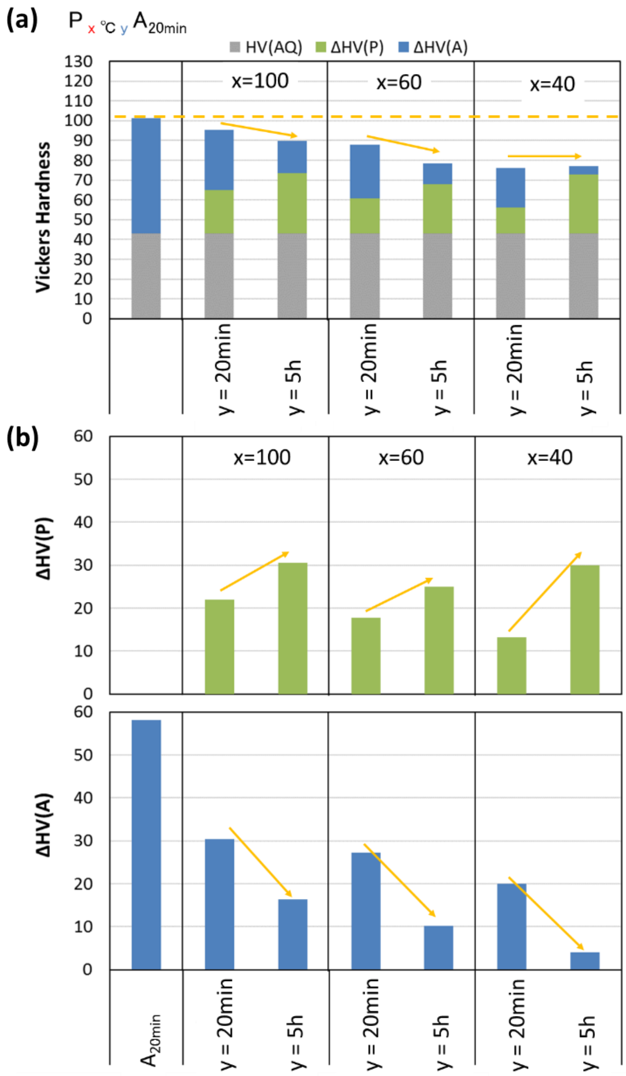

Figure 2. (a) Attained Vickers hardness of A_{20min} and $P_{x°Cy}A_{20min}$ (x=100, 60 and 40, y=20min and 5h) specimens with composition of 7M8S. The contribution of ΔHV(P) and ΔHV(A) is compared in (b)

Table 2. Area of peaks evaluated by DSC measurement

	$P_{100°C\ 20min}$	$P_{100°C\ 5h}$	$P_{60°C\ 20min}$	$P_{60°C\ 5h}$	$P_{40°C\ 20min}$	$P_{40°C\ 5h}$
Area of peak (i) (J/g)	0	0	6.31	0	30.1	10.5
Area of peak (ii) (J/g)	0	0	7.21	8.94	13.2	25.1

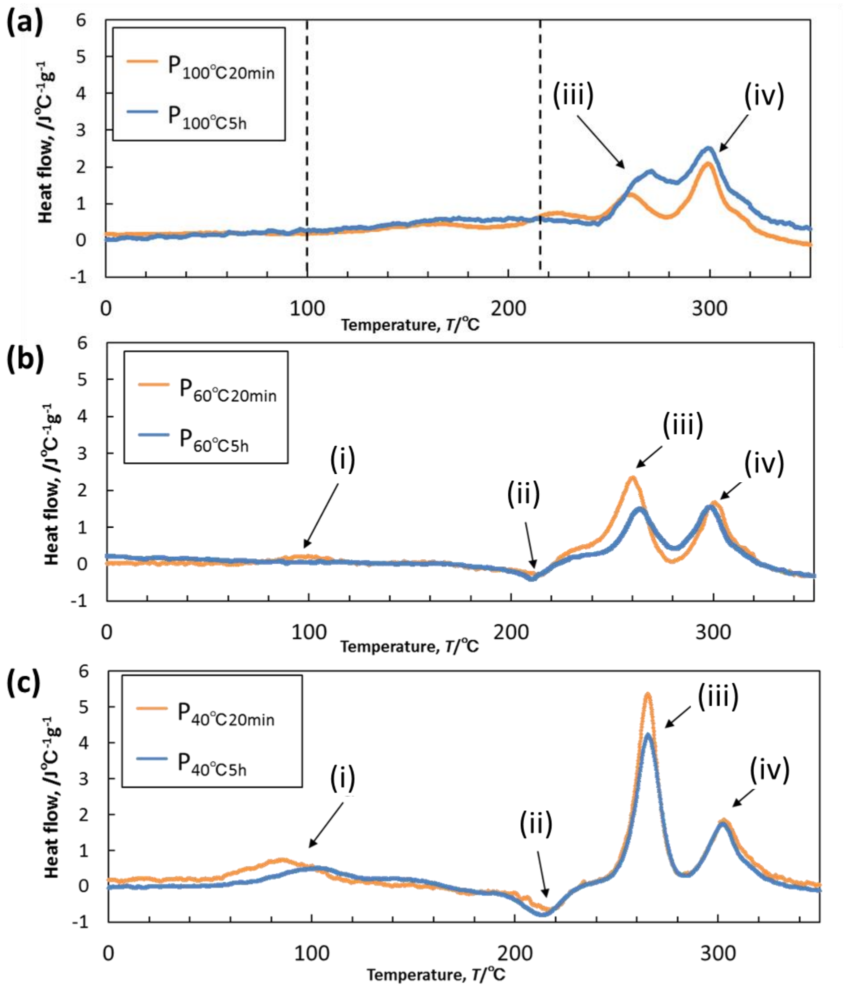

Figure 3. DSC curves of (a) $P_{100°C\ y}$, (b) $P_{60°C\ y}$ and (c) $P_{40°C\ y}$ (y=20min and 5h) specimens with composition of 7M8S

CONCLUSIONS

1. When pre-aged at 0 to 100°C, an Al-0.73mass%Mg-0.81mass%Si alloy subjected to artificial aging at 170°C for 20 min exhibits the attained hardness smaller than that of directly aged specimen (i.e. negative effect of two-step aging). The smallest hardness was obtained when pre-aged at 20°C.
2. When pre-aging time is prolonged from 20 min to 5 h, the increment of hardness during pre-aging ΔHV(P) is increased in the specimens pre-aged at 0 to 100°C, but their attained hardness and increment of hardness during artificial aging (i.e. bake-hardening response) ΔHV(A) are both decreased.
3. DSC measurement suggests that only Mg-Si clusters are formed during pre-aging at 100°C, and as

pre-aging temperature is lowered, the total amount of solute clusters is decreased, but the ratio of Si-rich clusters among those clusters is increased.

4. It is considered that the degree of ΔHV(A) in pre-aged specimens is affected not only by the type and amount of solute clusters (Mg-Si clusters and Si-rich clusters) formed during pre-aging, but also by the amount of residual solute concentration in the matrix after pre-aging.

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