

Effect of Amount of Pre-strain on Bake-hardening Response of an Al-Mg-Si Alloy

Tetsuya Masuda^{1,2}, Yasuo Takaki² and Shoichi Hirose³

¹ Graduate student, Department of mechanical engineering and materials science, Yokohama National University, 79-5 Tokiwadai, Hodogaya, Yokohama, Kanagawa 240-8501, JAPAN

² Material & process research section, Aluminum sheets and coils research department, Kobe steel, Ltd., 15 Kinugaoka, Moka, Tochigi 321-4367, JAPAN

³ Department of mechanical engineering and materials science, Yokohama National University, 79-5 Tokiwadai, Hodogaya, Yokohama, Kanagawa 240-8501, JAPAN

The effect of amount of pre-strain on the bake-hardening response of an Al-0.6mass%Mg-1.0 mass%Si alloy was investigated by means of Vickers hardness test and transmission electron microscopy (TEM). The bake-hardening response at 443K was improved by pre-straining to 3%, but reduced with increasing amount of pre-strain larger than 5%. TEM observation confirmed that the evolution of bake-hardening response with pre-straining is related to the number density of fine precipitates in the matrix. This suggests that the improvement and degradation of bake-hardening can be explained by the contradicting effects of dislocations induced by pre-straining.

Keywords: Al-Mg-Si alloy, pre-strain, bake-hardening response, β'' phase, dislocation

1. Introduction

Heat treatable Al-Mg-Si alloys are mainly used for automotive body panels because of their good formability, corrosion resistance, surface quality and precipitation strengthening after paint-bake treatment at 443-453K (i.e. bake-hardening response) during car manufacturing processes. It is well known that the β'' phase greatly contributes to the bake-hardening response and the alloys containing excess of Si over Al-Mg₂Si quasi-binary composition exhibit more pronounced bake-hardening[1]. Pre-straining has a substantial effect on precipitation kinetics and processes for Al-Mg-Si alloys. A large amount of pre-strain lowers the bake-hardening due to the unfavorable heterogeneous precipitation on dislocations induced by pre-straining[2]. On the other hand, the authors recently reported that a small amount of pre-strain below 3%, combined with pre-aging process, enhances the bake-hardening response due to the accelerated precipitation of the β'' phase in the matrix[3]. However, the mechanism of such effects of pre-strain on the bake-hardening response has not been fully clarified.

In this work, the effect of amount of pre-strain by 0.5 to 30% on bake-hardening behavior of an Al-0.6mass%Mg-1.0mass%Si alloy was investigated in attempts to clarify the mechanism of enhancement or degradation of bake-hardening by pre-straining.

2. Experimental

The chemical composition of the alloy utilized in this work is listed in Table 1. The alloy ingot was homogenized and hot- and cold-rolled through the standard manufacturing processes to a plate with final thickness of 1.0mm. The specimens were solution heat-treated at 823K for 60s, and then water quenched. Shortly after water quenched, pre-strain was introduced with a tensile testing machine (by 0.5, 3, 5 and 8%) or with a cold-rolling mill (by 10 and 30%). The corresponding equivalent strain by which the two different deformation modes can be compared is 5.0×10^{-3} , 3.0×10^{-2} , 4.9×10^{-2} , 7.7×10^{-2} , 1.2×10^{-1} or 4.1×10^{-1} , respectively. Some specimens were processed without pre-straining for comparison. The hardness was measured by a Vickers hardness tester with 19.6N load and dwell time

of 15s to evaluate the bake-hardening response during final aging at 443K. The specimens were held at room temperature for approximately 300s between solution heat treatment and final aging. For the specimens final aged at 443K for 3.6ks, transmission electron microscopy (TEM) observation was performed under accelerating voltage of 200kV.

Table 1 Chemical composition of the alloy utilized in this work [in mass%].

Si	Fe	Mn	Mg	Al
1.02	0.17	0.07	0.57	Bal.

3. Results

3.1 Bake-hardening response

The hardness evolution during final aging at 443K is shown in Fig.1(a) for the specimens with different amounts of pre-strain. The hardness before final aging; HV_{Before} , is increased with increasing amount of pre-strain from HV46(0% pre-strain) to HV78(30% pre-strain). The time to peak hardness becomes shorter with increasing amount of pre-strain. To compare the bake-hardening response, the increment in hardness by final aging up to 3.6ks (indicated by broken line in Fig.1(a)); ΔHV ($=HV - HV_{\text{Before}}$), is plotted in Fig.1(b). ΔHV increases with 0.5% pre-strain, and then decreases with increasing amount of pre-strain. Namely, it was found that the bake-hardening response of the alloy is enhanced by <3% pre-strain, but reduced by >5% pre-strain.

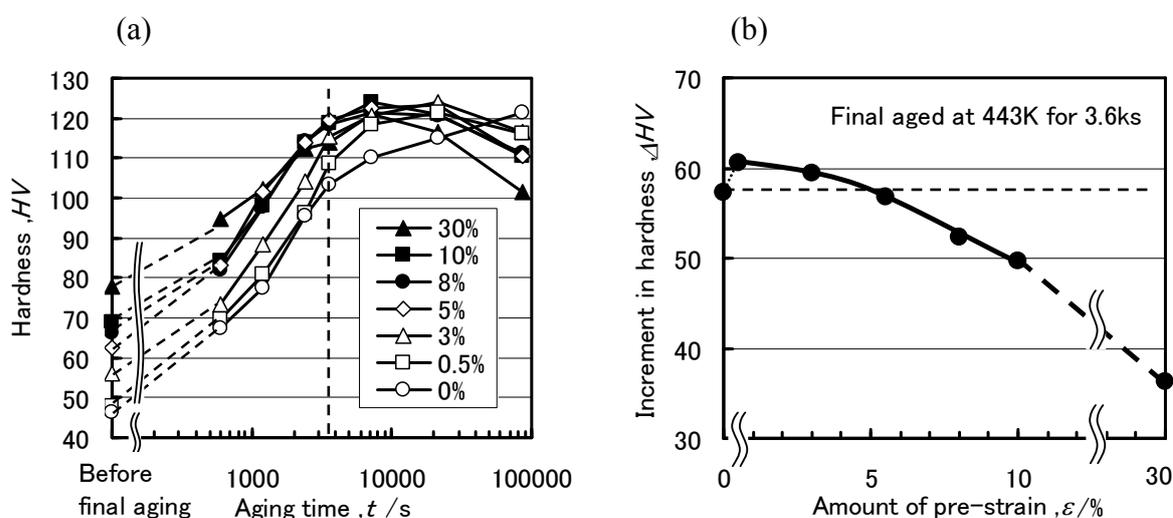


Fig.1 (a)Hardness evolution during final aging at 443K and (b)increment in hardness; ΔHV , after final aged for 3.6ks of the specimens with 0 to 30% pre-strain.

3.2 TEM microstructure

Fig.2 shows TEM bright- and dark-field images together with selected area diffraction patterns (SADPs) of the specimens without pre-strain, with 3 and 30% pre-strain, subjected to final aging at 443K for 3.6ks. It is found from bright-field images that the dislocation density becomes higher with increasing amount of pre-strain. In dark-field images, the β'' precipitates in the matrix, which give rise to streaks along $[010]_{\text{Al}}$ and $[100]_{\text{Al}}$ in SADPs, decrease in number density with increasing amount of pre-strain due to the formation of large precipitates along dislocation lines. The corresponding size distribution of precipitates is shown in Fig.3. It is revealed that while large precipitates than 10nm in maximum length monotonically increase in number density with increasing amount of pre-strain (Fig.3(a)), the number density of fine precipitates with <10nm once increases with 3% pre-strain, then decreases with larger amount of pre-strain than 5% (Fig.3(b)).

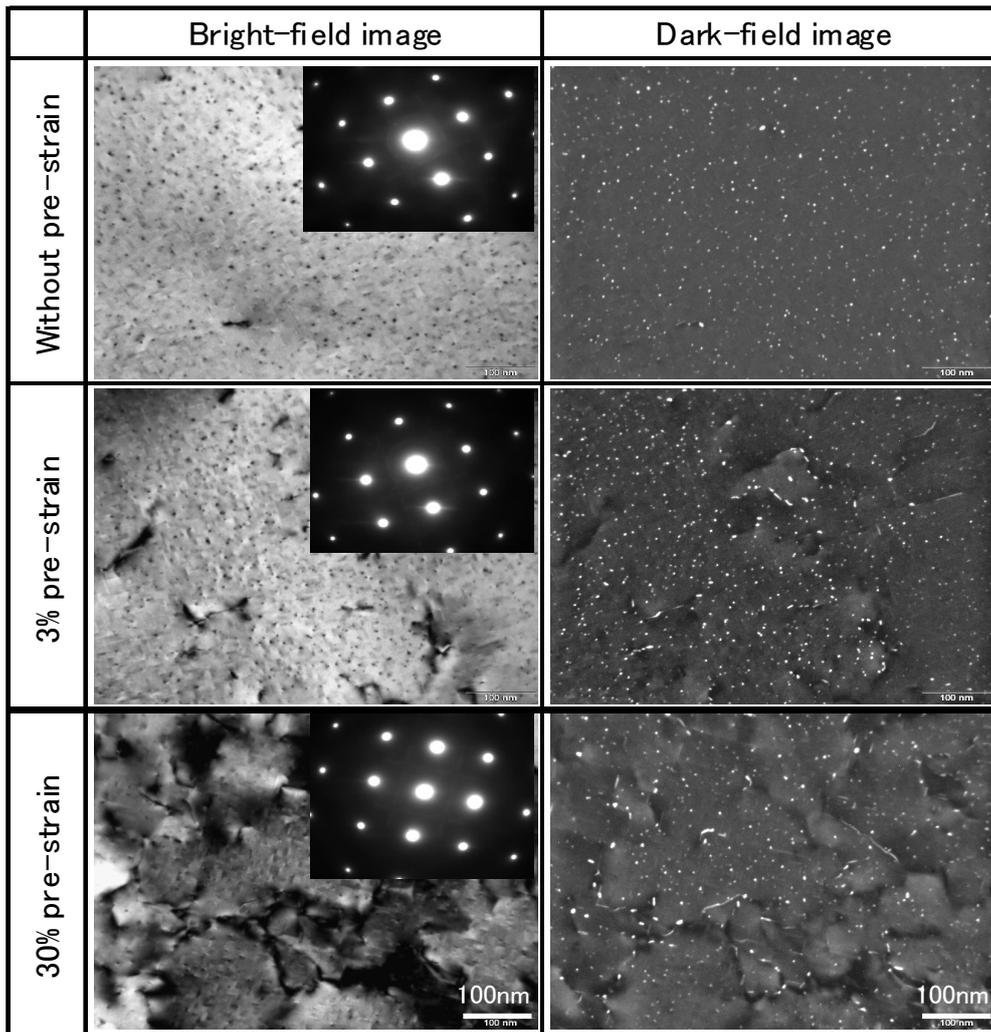


Fig.2 TEM bright- and dark-field images and selected area diffraction patterns of the specimens without pre-strain, with 3 and 30% pre-strain, subjected to final aging at 443K for 3.6ks.

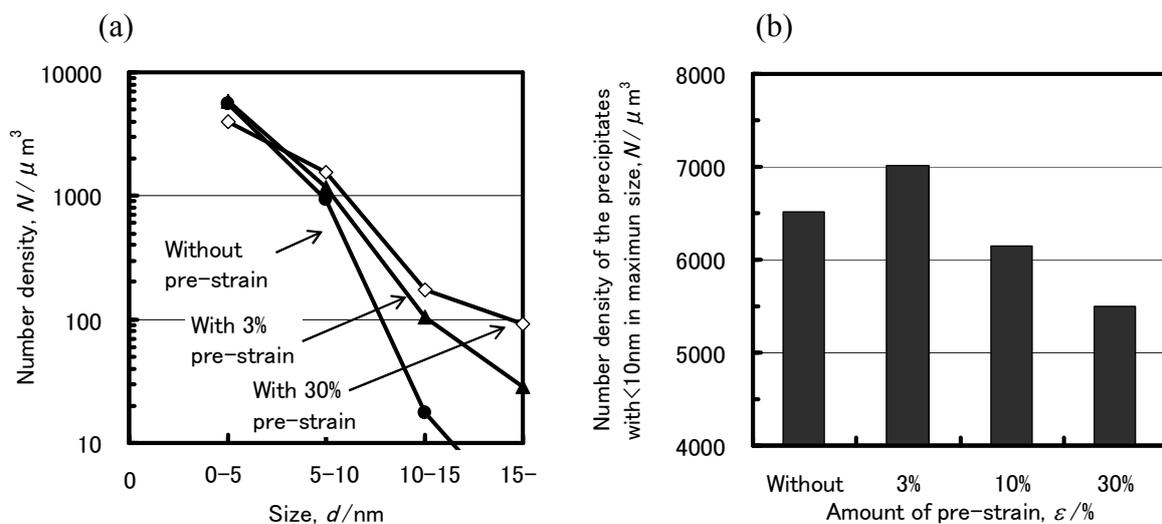


Fig.3 (a)Size distribution of all the observed precipitates and (b)number density of smaller precipitates than 10nm in maximum length for the specimens without pre-strain, with 3 to 30% pre-strain, subjected to final aging at 443K for 3.6ks.

4. Discussion

4.1 Evolution of bake-hardening response with amount of pre-strain

As shown in Figs.1, 2 and 3, the effect of pre-straining on the bake-hardening response strongly depends on amount of pre-strain. Fig.4 shows the relationship between the number density of smaller precipitates than 10nm; N (Fig.3(b)) and the increment in hardness; ΔHV (Fig.1(b)) in the specimens with different amounts of pre-strain. It is suggested that bake-hardening response at 443K is mainly determined by the number density of fine precipitates, since ΔHV well agrees with N irrespective of amount of pre-strain. Pre-strain by 3% increases the number density of smaller precipitates than 10nm, and enhances the bake-hardening response. The authors previously reported that smaller amount of pre-straining than 3% suppresses the age-hardenability of the present Al-Mg-Si alloy at lower temperatures below 373K, but improves the bake-hardening response at 443K[3]. This can be explained by the decreased concentration of quenched-in excess vacancies with pre-straining, resulting in the suppressed clustering of solute atoms in the matrix. On the other hand, pre-strain larger than 5% decreases the number density of smaller precipitates than 10nm, and lowers the bake-hardening response due to the preferential heterogeneous formation of larger precipitates on dislocation lines. Softening of the alloy specimen by recrystallization and grain growth is thought to be negligible at 443K, since recrystallization is expected to occur only above 573K when 20% pre-strain is applied to this alloy[4].

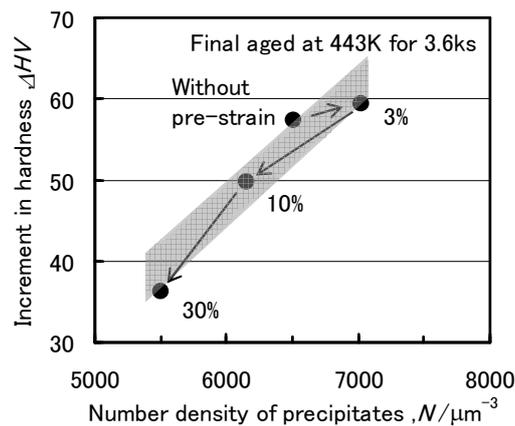


Fig.4 Relationship between number density of precipitates with <10nm N and increment in hardness ΔHV for the specimens with different amounts of pre-strain.

4.2 Modeling of precipitation in the matrix

According to the classical nucleation theory[5], nucleation rate J is given by

$$J = N_s \cdot \beta \cdot Z \cdot \exp\left(-\frac{\Delta G}{kT}\right), \quad (1)$$

where N_s is the number of potential nucleation sites, $\beta = A \cdot D \cdot c$ is frequency factor, Z is Zeldovich non-equilibrium factor, ΔG is activation energy for nucleation, k is Boltzman constant, T is aging temperature, A is a constant, D is diffusivity and c is solute concentration in the matrix, respectively. Since it is believed that the site saturation does not occur in the early stages of phase decomposition, the number density of precipitates in the matrix N_p is thought to be related to J .

In this work, to clarify the effect of amount of pre-strain, the ratio of number densities of precipitates in the matrix for the specimens with and without pre-strain N_p/N_p' was defined as the product of c/c' and N_s/N_s' ; i.e

$$\frac{N_p}{N_p'} = \frac{J}{J'} = \frac{c}{c'} \cdot \frac{N_s}{N_s'}, \quad (2)$$

where N_p' , J' , c' , N_s' are the values for the specimen without pre-strain.

(a) Solute concentration in the matrix c

Assuming that dislocations induced by pre-straining act as sink sites for quenched-in excess vacancies, the estimated lifetime τ of quenched-in excess vacancies to annihilation at dislocations pronouncedly decreases with amount of pre-strain as shown in Fig.5[3]. Since it is thought that solute concentration in the matrix c increases with decreasing τ due to the suppressed formation of clusters during natural aging, c can be defined as

$$c = c_0 - (c_0 - c') \cdot \frac{\tau}{\tau'}, \quad (3)$$

where c_0 is the initial solute concentration and τ' is lifetime of quenched-in excess vacancies for the specimen without pre-straining. In this work, c_0 and c' were adapted as 1.58at% and 1.34at% by taking into account concentrations of Si and Mg in the alloy. Broken lines in Fig.6 show pre-strain dependence of the estimated ratio of solute concentrations c/c' . It is noted that c/c' increases with small amount of pre-strain and saturates at ~ 1.18 above 0.5% pre-strain.

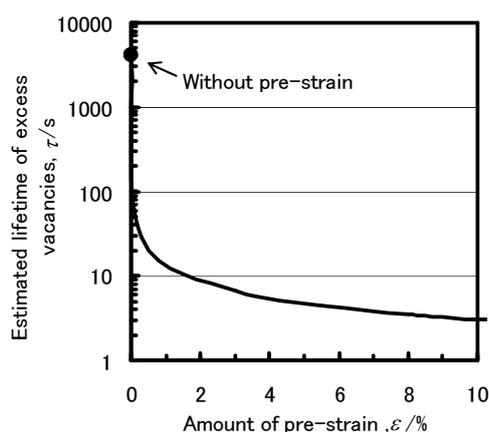


Fig.5 Pre-strain dependence of estimated lifetime τ of quenched-in excess vacancies[3].

(b) Number of potential nucleation sites N_s

In general, precipitates are expected to form preferentially on dislocation lines due to the decreased activation energies for nucleation and growth. It is considered that potential nucleation sites in the matrix decreases with increasing amount of precipitation on dislocations as a result of solute atoms consumption. The potential nucleation sites in the matrix N_s can be therefore assumed as

$$N_s \propto 1 - \pi r^2 \rho, \quad (4)$$

where r is the radius of cylindrical zones affected by precipitation on dislocation lines and $\rho = X\varepsilon^n/Y$ is the dislocation density calculated from reported values $X=0.185\text{n}\Omega\text{m}$, $n=0.648$ (for an Al-0.47 mass%Mg alloy[6]), $Y=3\times 10^{-25}\Omega\text{m}^3$ (for high-purity aluminum[7]) and true strain ε . In this work, r was assumed to be 20nm from TEM observation, and dislocation densities of the specimens without pre-strain, with 3, 10 and 30% pre-strain were estimated as 1×10^{11} [8], 6×10^{13} , 1×10^{14} and $3\times 10^{14}\text{m/m}^3$, respectively. Dotted lines in Fig.6 show pre-strain dependence of the estimated ratio of potential

nucleation sites in the matrix N_s/N_s' . It is noted that N_s/N_s' monotonously decreases with increasing amount of pre-strain.

4.3 Number density of precipitates in the matrix N_p/N_p'

The evolution of N_p/N_p' estimated through Eq.(2) is shown by straight lines in Fig.6. The trend of N_p/N_p' change is similar to the change of number densities of smaller precipitates than 10nm in Fig.3(b). Therefore, the evolution of bake-hardening response with pre-straining in Fig.1(b) can be explained by the contradicting effects of dislocations induced by pre-straining; i.e. the enhanced precipitation in the matrix due to the highly supersaturated solute concentrations (<3% pre-strain) and the suppressed precipitation in the matrix due to the preferential formation of large precipitates on dislocation lines (>5% pre-strain).

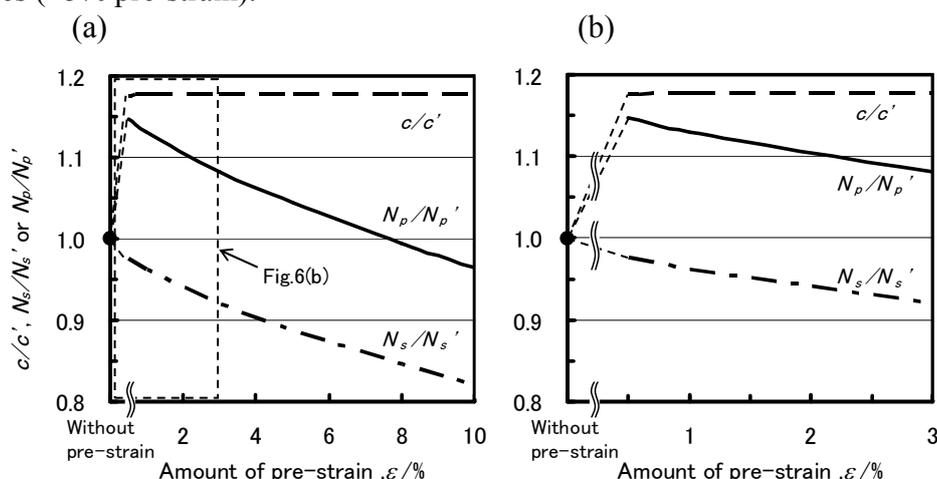


Fig.6 Pre-strain dependence of number densities of precipitates in the matrix N_p/N_p' , solute concentrations in the matrix c/c' and numbers of potential nucleation sites N_s/N_s' .

5. Conclusions

The effect of pre-strain on the bake-hardening response of an Al-0.6mass%Mg -1.0mass%Si alloy was investigated. The bake-hardening response strongly depends on amount of pre-strain. Smaller amount of pre-straining below 3% accelerated bake-hardening during final aging at 443K, whereas larger amount of pre-straining reduced the bake-hardening response. This can be explained by the contradicting effects of dislocations induced by pre-straining; i.e. the enhanced precipitation of fine β'' in the matrix due to the highly supersaturated solute concentrations and the suppressed precipitation in the matrix due to the preferential formation of large precipitates on dislocation lines.

References

- [1] T. Inaba, K. Tokuda, H. Yamashita, Y. Takebayashi, T. Minoura and S. Sasabe: KOBELCO TECHNOLOGY REVIEW 26 (2005) 55-62.
- [2] K. Matsuda, M. Terasaki, S. Tada and S. Ikeno: JJILM 45 (1995) 95-100.
- [3] T. Masuda, Y. Takaki, T. Sakurai and S. Hirose: Mater. Trans. 52 (2010) 325-332.
- [4] D. C. Long, Y. Ohomori and K. Nakai: Mater. Trans. 41 (2000) 690-695.
- [5] J. Yang and M. Enomoto: ISIJ int. 45 (2005) 1335-1344.
- [6] S. Komatsu, M. Ikeda, T. Miyashita, H. Yukimachi and T. Ikeda: Proceedings of ICAA-6 (1998) 991-996.
- [7] J. G. Rider and C. T. B. Foxon: Philos. Mag. 13 (1966) 286-303.
- [8] W. D., Jr. Callister: *Materials Science and Engineering, An Introduction*, (John Wiley & Sons, Hoboken, New Jersey, 2007) p.166.